According to the Decision of the NAQAER No. 19 from 06.12.2019, JES is classified as B+ journal

Main subjects areas of the Journal of Engineering Science:

A. Industrial Engineering
- Mechanical Engineering and Technologies
- Applied Engineering Sciences and Management
- Materials Science and New Technologies
- Electrical Engineering and Power Electronics
- Energy systems
- Light Industry, New Technologies and Design
- Industrial and Applied Mathematics
- Vehicle and Transport Engineering

B. Electronics and Computer Science
- Electronics and Communication
- Microelectronics and Nanotechnologies
- Biomedical Engineering
- Computers and Information Technology
- Automation

C. Architecture, Civil and Environmental Engineering
- Architecture, Urbanism and Cadaster
- Civil Engineering and Management
- Energy Efficiency and New Building Materials
- Environmental Engineering

D. Food Engineering
- Food Technologies and Food Processes
- Food Industry and Management
- Biotechnologies, Food Chemistry and Food Safety
- Equipment for Food Industries

The structure of the journal corresponds to the classification of scientific publications: *Engineering, Multidisciplinary.*

How to publish a paper:

1. Send the manuscript and information about the author to the **Editorial Board address:** jes@meridian.utm.md
2. Manuscripts are accepted only in English, by e-mail, in template file (www.jes.utm.md)
3. After a review, you will be notified of the editorial board's decision.
4. After the Journal has been published, we will send it to you immediately by mail.

CONTENT

D. Food Engineering

CONȚINUT

OPEN ACCESS

CC BY

CHARACTERISTICS OF REFUSALS IN LOW-VOLTAGE DISTRIBUTION SYSTEMS

Victor Popescu [1], ORCID: 0000-0002-4634-2255,
Mihai Tîrşu[1], ORCID: 0000-0002-1193-6774,
Ilie Nuca [1], ORCID: 0000-0001-7750-4846,
Vasile Leu[1], ORCID: 0000-0002-1318-6663,
Vadim Cazac [1*], ORCID: 0000-0002-2887-7520,
Tatiana Balan [2], ORCID: 0000-0002-8897-105X

1Technical University of Moldova, 168 Stefan cel Mare Blvd., Chisinau, Republic of Moldova
2State University of Moldova, 60 Alexei Mateevici str., Chisinau, Republic of Moldova
*Corresponding author: Vadim Cazac, vadim.cazac@ie.utm.md

Abstract. This paper focuses on the determination of the characteristics of refusals in low voltage electrical distribution systems. In order to solve this problem, the phenomena of occurrence of refusals in low-voltage electrical distribution systems, conditioned by different influencing factors, processed and mathematically modelled. The main results of the research consist in establishing the static characteristics of the refusals, the mathematical models that determine the occurrence of the cause phenomena and the parameters that characterize the distributions of the refusals, depending on the occurrence per system, depending on the occurrence per season, depending of interruptions duration and the number of affected consumers. The significance of the results obtained consists in solving a number of problems faced by the operating services of electrical distribution systems, concerning the assurance of reliability of operation, providing the possibility of forecasting and preventing the occurrence of the phenomena of occurrence of refusals, which enable the justified planning of prophylactic measures and ensuring the reliability level.

Keywords: *electrical distribution systems, refusal characteristics, influence factors, mathematical models.*

Abstract. Articolul se concentrează pe determinarea caracteristicilor refuzurilor în sistemele electrice de distribuție de joasă tensiune. Pentru a rezolva această problemă, au fost prelucrate și modelate matematic fenomenele de apariție a refuzurilor în sistemele de distribuție a energiei electrice de joasă tensiune, condiționate de diferiți factori de influență. Principalele rezultate ale cercetării constau în stabilirea caracteristicilor statice ale refuzurilor, a modelelor matematice care determină apariția fenomenelor cauze și a parametrilor care caracterizează distribuțiile refuzurilor, în funcție de apariția pe sistem, în funcție de apariția pe anotimp, în funcție de durata întreruperilor și de numărul de consumatori afectați. Semnificația rezultatelor obținute constă în rezolvarea unei serii de probleme cu care se confruntă serviciile de operare a sistemelor electrice de distribuție,

privind asigurarea fiabilităţii funcţionării, oferirea posibilităţii de prognozare şi prevenire a apariţiei fenomenelor de apariţie a refuzurilor, ceea ce permite planificarea justificată a măsurilor preventive şi asigurarea nivelului de fiabilitate.

Cuvinte cheie: *sisteme electrice de distribuție, caracteristici de refuz, factori de influență, modele matematice.*

1. Introduction
At this stage, an important number of refusals are occurring in low-voltage electricity distribution systems from the Republic of Moldova, which are severely affecting consumers and causing substantial economic damage [1, 2]. Establishing the factors causing these refusals and determining the reasons for their occurrence, allows the development of a mechanism to prevent and ensure continuity of supply to consumers, with a reduction in economic damage [3, 4].

The static characteristics of refusals, the causes of their occurrence and their impact on the reliability of distribution systems, are not currently studied to the level stipulated by current documents on reliability indicators [5, 6].
Although multiple researches have been done by researchers in the field so far, but the data in the literature on the theoretical and practical basis of the reliability of low voltage distribution systems are quite modest and require further study, especially the static characteristics of refusals, the causes of their apparition and their effect on reliability, as they are not currently known at the level stipulated by the current documents on the reliability of these systems [7-11].

Reliability of low-voltage distribution systems can only be ensured based on an in-depth knowledge of the phenomena accompanying this process, which permit technically and economically planning of the measures and activities of power system operation services to ensure the standard reliability indicators [12, 13].

Accordingly, the focus of the research is on the characteristics of refusals in low voltage electricity distribution systems caused by various random factors for the 5-year period (2016-2021) [1, 2].

The general objective of the work is to develop mathematical models which determine the laws for distributing refusals in these systems, conditioned by different influencing factors [14, 15].

The results obtained and the mathematical models developed allow forecasting refusals with 95% reliability and their parameters, which provides the possibility of justified planning of measures to prevent cause factors and the mechanisms development to ensure the reliability of low-voltage distribution systems, reducing operating costs and economic damage caused by these refusals [16].

2. Materials and Methods
To solve the problems formulated for research have been: graph theory; probability theory; methods of statistical analysis and experimental data processing; linear and nonlinear equations theory; mathematical modeling; calculation techniques with the software „Easy Fit 5.5", „Stat Graphics", „Curve Expert" [15, 16].
The following steps were taken to carry out the research:
- Recording of refusals in low-voltage electricity distribution systems (10 systems and 57 subsystems) for a period of 5 years (2016-2021).

- Classification of refusals according to their occurrence and influencing factors.
- Development of the analysis and systematization concept of refusals according to season and geographical location of systems.
- Determination of laws and the parameters which characterize the distributions of faults for all influencing factors.
- Developing the mathematical models that identify the laws of faults occurrence.
- Argumentation of the procedure for forecasting random refusals in distribution systems and the parameters that characterize these refusals.

3. Results

The study found that the reliability of low-voltage distribution systems, directly affected by a number of random factors that determine the continuity of the electricity supply to consumers. Considerations relating to the aspect of ensuring the continuity and quality of power supply for consumers also identified, by determining the reasons for the occurrence of refusals caused by random factors and establishing the mechanism for reducing the level of influence of these factors. It is established that the level of reliability of electrical distribution systems can only be assured on the basis of an understanding of all phenomena accompanying the operating processes of these systems and through technical and economic justified planning of necessary measures to ensure the standard reliability indicators. It was found necessary to develop the procedure for forecasting the intensity of faults conditioned by different factors and to estimate the weight of the impact of random influencing factors on reliability indicators, with consideration for the number of interruptions, their duration and the number of consumers affected, which allows justified planning of technico-economic activities in order to ensure reliability of low voltage distribution systems. It was found necessary to develop the procedure for forecasting the intensity of refusals conditioned by various factors and to estimate the weight of the impact of random influencing factors on reliability indicators, with consideration for the number of interruptions, their duration and the number of consumers affected, which allows a justified planning for technico-economic activities to ensure the reliability of low voltage distribution systems.

Methodology for reliability analysis of distribution systems using graph theory and diacoptics has been proposed, which allow analytical reliability calculations to be performed for systems with any structure, according to which complex systems have been examined on subsystems, sections, graphs and nodes.

The solution for outage classification and processing is presented, which allows to characterize specific refusals depending on each influencing factor (for each system according to season) and the concept for assessing and forecasting the reliability level of distribution systems, which systemize the consecutiveness of operations done in the process of reliability assessment. Processing the characteristic information on interruptions in the systems reviewed, using a standard procedure of analysis and calculation, the unique concept of a generalized methodological approach for the assessment of reliability indicators was proposed. Using the specific unit of line length notion, which allows to determine and compare the actual level of reliability for all power lines, disregarding of their total length.

The methodological and theoretical-scientific support for assessing the reliability of electrical distribution systems was provided by graph and matrix theory; probability theory; methods of statistical experimental data analysis and processing of faults in distribution systems; linear and non-linear of equations theory; math modelling.

Using the classification method and the developed concept of refusal processing, the frequency of occurrence of defects resulting from each random factor was determined for each system by season.

In Table 1 are presents the processed results of random faults that took place seasonally in the investigated systems by different groups of factors, reported on a specific unit of line length.

Table 1

The faults generated by different groups of factors

Groups of factors	Seasonal number of faults per specific unit of line length				
Name	Winter	Spring	Summer	Autumn	Annual
Animal and bird action	0.17	0.13	0.33	0.35	0.98
Climatic conditions	6.33	5.77	3.88	5.17	21.15
Equipment defects	5.28	4.52	4.42	4.77	18.99
Acts of vandalism	0.34	0.39	0.49	0.36	1.58
Unidentified factors	5.94	4.56	5.00	5.61	21.11
Damage caused by vegetation	0.83	0.36	0.65	0.55	2.39
The action of mechanisms	0.08	0.26	0.28	0.15	0.77
Operational errors	0.03	0.01	0.03	0.02	0.09
TOTAL	**19**	**16**	**15**	**17**	**67**

All this has made it possible to make simple the calculation and to establish rejection distributions for all the factors, according to the frequency of occurrence per system and season, which allows to determine structures and measures to improve reliability in power distribution systems. In the process of assessing the reliability of the investigated systems the following was done: using the proposed methodology, primary information on refusals that occurred in low voltage distribution systems (10 systems and 57 subsystems) for the 5-year operating period (years 2016-2021) was processed; based on the developed classification concept, all random refusals were selected, which allowed structuring and classifying the information sets according to 8 groups influencing factors by geographical location and season; the concept of homogenizations of primary information was proposed, which ensures the possibility of comparing the intensity of refusals conditioned by different influencing factors for any distribution system, regardless of the summary length of the lines, allowing the determination of numerical values of reliability indicators based on a standard analysis and calculation procedure.

It has been established only to predict influence of random factors on the reliability of power systems, it is necessary to determine the laws of refusals distribution generated by factors of these distributions.

Based on findings, for the following indices where examined experimental and theoretical distributions: frequency of occurrence of faults by system and season, duration of faults and number of consumers which was disconnected.

Table 2 shows examples of mathematical models and their parameters, which establish the distribution laws of faults as a function of duration, for the groups of factors examined.

Mathematical models determining the distribution laws of refusals

Groups of factors	Type Function	The mathematical model	Mathematical model parameters			
			a	b	c	d
Animal and bird action	Weibull	$y = a - be^{-cx^d}$	1612.01	1682.03	0.24	-0.57
Damage caused by vegetation	Log-Logistic 3-Param.	$y = a/[1 + (x/b)^c]$	3803.98	463.97	3.29	-
The action of mechanisms	Weibull	$y = a - be^{-cx^d}$	274.98	270.99	4.51	-2.39
Climatic conditions	Lognormal 3- Param.	$y = e^{a+b/x+c\ln(x)}$	11.57	-3.40	-2.27	-
Unidentified factors	Weibull	$y = a - be^{-cx^d}$	1166.98	1169.01	2.19	-1.79
Equipment defects	Lognormal 3- Param.	$y = e^{a+b/x+c\ln(x)}$	11.69	-4.01	-2.21	-
Acts of vandalism	Weibull	$y = a - be^{-cx^d}$	538.95	535.99	3.48	-2.11
Operational errors	Exponential	$y = ae^{b/x}$	1.61	4.52	-	-

The mathematical models developed confirm that the 8 groups influencing factors, in terms of the duration of interruptions caused, behave differently. It can be seen that 4 of them (vandalism acts, action of animals, action of various mechanisms, unidentified factors) are characterized by a Weibull distribution, 2 factors (faults in equipment, weather condition) have a Lognormal distribution (3-parameters), one group of factors (operating errors) has an Exponential distribution and one group of factors (damage caused by vegetation) has an Log-Logistic distribution (3-parameters).

At the same time, at each stage of the research carried out, the theoretical models made in this study were compared with the obtained experimental results. Thus, for each group of factors, comparing the experimental distributions and the elaborated theoretical models, which determine the distributions of the refusals and the static characteristics, based on the application of the similarity criteria, it was found that the degree of overlap is about 95 %.

So, for example, arbitrarily selecting a group of factors among those examined, as an example, in figure 1, is graphically presented the experimental distribution of refusals according to duration, caused by climatic conditions, and in figure 2, the theoretical model elaborated is graphically presented, which determines the law of distribution of refusals according to duration, for this group of factors - climatic conditions, and the compatibility for this model, the similarity criteria indicate an insignificant deviation of about 5 %.
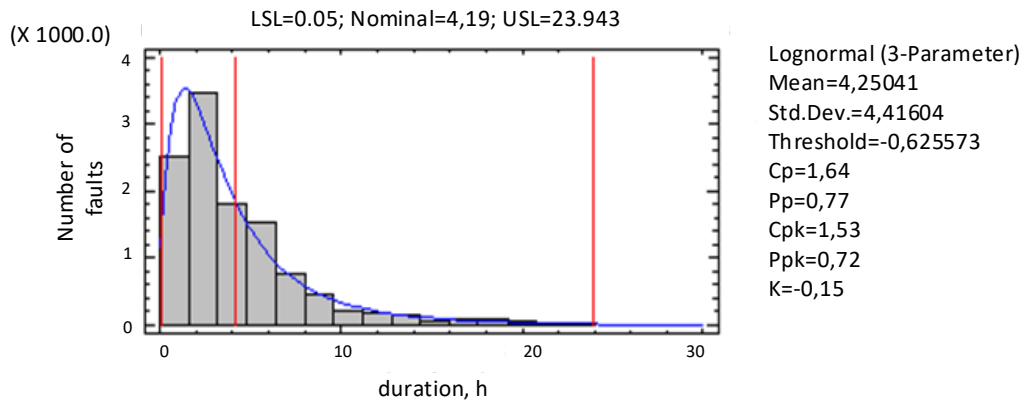
Figure 1. Graphic presentation of the experimental distribution of refusals according to duration, caused by climatic conditions.

It was found that, according to the number of affected consumers, 6 groups of factors were described using Weibull distribution law (vandalism acts, action of animals and birds, action of different mechanisms, damage caused by vegetation, equipment faults, operating errors), one of them (weather conditions) was described using Lognormal model (3-Parameters) and (unidentified factors) with the Log-Logistic model (3-Parameters).

On the basis of the mathematical models developed, the main parameters of the replications, which characterize the interruptions caused by the 8 groups of influencing factors examined, were established according to the duration of disconnections and number of consumers which were affected (total number of refusals caused, the average number of disconnections per season, average duration of disconnections, average number of disconnected consumers, root mean square deviation, coefficient of variation, minimal and maximal duration of disconnections, minimal and maximal number of disconnected consumers, crosstalk, marginal values of the confidence interval, skewness and excess coefficients).
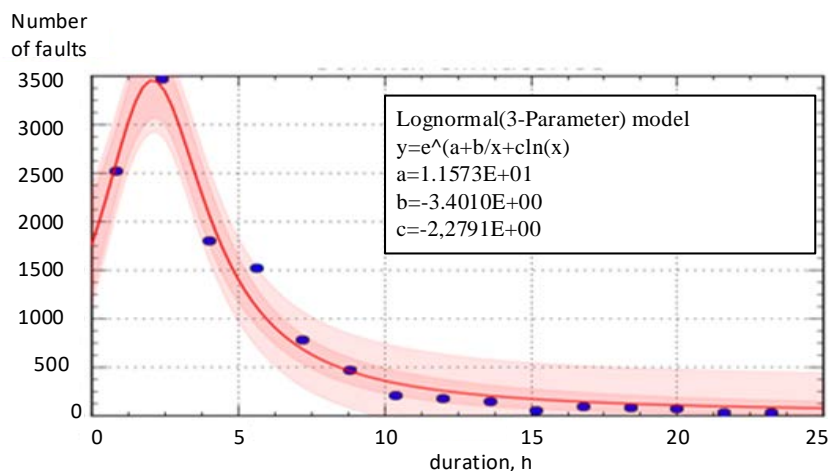
Figure 2. Graphic presentation of the developed mathematical model, which determines the distribution of refusals according to duration, caused by climatic conditions.

4. Discussion

Based on the above, the necessary criteria and parameter values were determined to characterize the influence of random factors on the reliability of power distribution systems and to forecast with 95 % reliability the following indices: expected number of interruptions on the system, depending on geographical location and season; duration of these interruptions; number of consumers likely to affect [1, 3].

Analyzing the distribution of random refusals and the parameters examined over the given time interval, it can be seen that the significant number of refusals that occurred due to unforeseen random factors influenced the level of reliability of these systems over the entire time period examined (2016-2021).

Because of the analysis of the reliability indicators, during the period investigated in the examined systems, there were 915463 refusals, which affected the process of energy supply and influenced the indicators of continuity and quality. Because of this, their impact on system reliability and quality only mitigated by forecasting these events in order to develop the mechanism for justified planning of operational works [4-6].

As a result, according to the developed method the reliability prediction of the investigated systems performed, taking into account the influence of random factors. The influence of the factors forecast based on the following parameters: expected number of refusals per system (per year per specific unit length); expected number of refusals per season; expected duration of refusals; expected number of consumers affected. These parameters establish the degree of operational reliability of the equipment installed in the power distribution systems and the results obtained from their forecasting allow technically and economically justified planning of activities to ensure the quality and continuity of the power supply for consumers, in compliance with the standard reliability indicators.

5. Conclusions

The mathematical modelling allowed establishing the theoretical distributions characterizing refusals according to duration and number of consumers affected, with the highlighting of four types of theoretical models such as Weibull, Lognormal, Log-Logistic and Exponential and covering all the characteristic experimental distributions for the 8 groups of influence factors included in the list of classification of interruptions.

The processing of the experimental data groupings offers the possibility to determine the values of all coefficients in the theoretical distributions established for describing the statistical behavior of the groups of random factors and to obtain the concrete relationships for calculating the distributions and the number of conditional breaks of all influencing factors accepted as significant in this research.

Thus, mathematical models determining the laws of occurrence of random interruptions developed, which allow forecasting with 95% reliability of all indices characterizing the level of operational reliability of low-voltage distribution systems, and this provides the possibility of specifying measures to prevent refusals, reducing operating costs and economic damage.

Conflicts of Interest: The authors declare no conflict of interest.

References
1. Popescu V. Method for determining the factors affecting the process of functioning of distribution systems. *Intellectus* 2020, 3-4, pp. 137-140 [in Russian].

2. Popescu V. The evolution of disconnections from the distribution electrical systems from Republic of Moldova. *Journal of Sustainable Energy* 2019, 10 (2), pp. 75-78.
3. Popescu, V. Analysis of short-circuit currents in distributive electric networks. *Intellectus* 2015, 2, pp. 113-115.
4. Pacureanu, I.R.; Juravle, D.; Albut-Diana, D. The Reliability of Protection Implemented in Low Distribution Network From Brasov City. *Analele Universităţii din Oradea, Fascicula de Energetică* 2009, 15, pp. 45-51.
5. Popescu, V.; Rotari, V. Appreciation of the level of reliability of electrical systems. *Ştiinţa agricolă* 2015, 1, pp. 83-86. [in Romanian].
6. Popescu, V.; Gaina, A.; Bodarev, G. Reliability analysis of power distribution systems the voltage of which is 6 and 10 KV. *Intellectus* 2012, 1, pp. 94-103.
7. Popescu, V. The study of the transient processes accompanied by the voltaic arc and their influence on the reliability of the distribution systems. *Analele universităţii din Oradea, Fascicula de Energetică* 2007, 13, pp. 60-63 [in Romanian].
8. Popescu, V.; Racul, A.; Burbulea, I. Reliability analysis of power distribution systems. *Journal of Sustainable Energy* 2012, 3 (3), pp. 156-158.
9. Erhan, F.; Popescu, V. Characteristics of faults in systems for distribution of electricity. *Buletinul AGIR* 2013, 4 (8), pp. 237-241.
10. Popescu, V.; Popa, A.; Bantas, R. Reliability analysis of systems for distribution of electricity. *ACTA Electrotehnica* 2013, 5 (54), pp. 387-389.
11. Popescu, V.; Racul, A.; Bodarev, G. Reliability index evaluation of electrical distribution systems. *International Journal of Innovative Research in Science, Engineering and Technology* 2015, 4 (6), pp. 4229-4232.
12. Voronca, S.L. Risk Analysis and Risk Management. Development of Risk Indicators in Electricity Transmission Companies. *Journal of Sustainable Energy* 2010, 1, pp. 25-31.
13. Hazi, Gh.; Hazi, A. Conditions for Efficient use of the 1000 V Energy Distribution. *Annales of the University of Craiova* 2007, 31, pp. 25-31.
14. Erchan, F.; Mocanu, A. The Problem of the Reliability of the Electric Power Equipment. *Analele Universităţii din Oradea, Fascicula de Energetică* 2007, 13 p.
15. Secui, D.C. The Sensitivity of the Electrical Substations Reliability Indices at the Variation of the Circuit-Breakers Stucking Probability. *Annals of the Oradea University. Fascicle of Energy Engineering* 2008, 14, pp. 51-57.
16. Ardeleanu, M.E. Fault Localization in Cables and Accessories by Off-Line Methods. *Annales of the University of Craiova* 2007, 13, pp. 25-31.

Submission of manuscripts: jes@meridian.utm.md

OPEN ACCESS

RELIABILITY OF SERIAL-PARALLEL NETWORKS VS RELIABILITY OF PARALLEL-SERIAL NETWORKS WITH CONSTANT NUMBERS OF SUB-NETWORKS AND UNITS

Veronica Andrievschi-Bagrin *, ORCID: 0000-0001-8364-9873,
Alexei Leahu, ORCID: 0000-0002-1670-0111

Technical University of Moldova, 168 Stefan cel Mare Blvd., Chisinau, Republic of Moldova
*Corresponding author: Veronica Andrievschi-Bagrin, *veronica.bagrin@ati.utm.md*

Abstract. In this paper it is performed, based on dynamic models, a comparative analysis of the reliability of two types of networks: serial-parallel and parallel-serial networks when the numbers of subnets and units in each subnet are predefined, constant numbers, but also when the lifetimes of the units are independent random variables. The equations for calculating the reliability of the related networks have been deduced. These functions are deduced for the dynamic model which is less studied and it is prooved to be relevant to the static model too. All equations are demonstrated and graphically illustrated in some examples. A few examples are analyzed graphically for different values of the number of units in the subnet and the number of subnets. This paper contains four different network topology models which are also analyzed by equations and graphically. The mathematical model described and the deduced equations will serve as a basis for the subsequent analysis of the dynamic networks of various topologies and various types of random variables that describe the lifetimes of the units of the analyzed system.

Keywords: *cumulative distribution function, distributions, global maximum, lifetime, survival functions.*

Rezumat. Pe baza modelelor dinamice în lucrare se realizează o analiză comparativă a fiabilității a două tipuri de rețele: rețele serial-paralele și paralel-seriale, când sunt predefinite numerele de subrețele și unități din fiecare subrețea, numere constante, dar și când duratele de viață ale unităților sunt variabile aleatorii independente. Au fost deduse ecuațiile pentru calcularea fiabilității rețelelor aferente. Aceste funcții sunt deduse pentru modelul dinamic, care este mai puțin studiat și care a fost demonstrat anterior drept relevant pentru modelul static. Toate ecuațiile sunt demonstrate și ilustrate grafic în câteva exemple. Unele exemple sunt analizate grafic pentru diferite valori ale numărului de unități din subrețea și ale numărului de subrețele. Această lucrare conține patru modele diferite de topologie de rețea care sunt, de asemenea, analizate prin ecuații și grafic. Modelul matematic descris și ecuațiile deduse vor servi ca bază pentru analiza ulterioară a rețelelor dinamice de

diverse topologii și diferite tipuri de variabile aleatorii, care descriu durata de viață ale unităților sistemului analizat.

Cuvinte cheie: *funcție de distribuție cumulativă, distribuții, maxim global, durată de viață, funcții de supraviețuire.*

1. Introduction

First of all, let us observes that many mathematical models in Network's Reliability deal with serial and parallel Networks as a subsystem of Network with more complex structure/topology considered in many works as [1-5]. At the same time, it can happen that due to the different topologies / structures of two networks made up of some and the same elements, they have different reliability [6,7]. If for elementary networks, such as serial and parallel networks, it is obvious that the last are, under the stated conditions, more reliable, but in the case of more complex structures, the answer is not so obvious. Let us take, for beginning, two standard network types: serial-parallel named scheme (*A*) and parallel-serial named scheme (*B*), according to the figure 1.
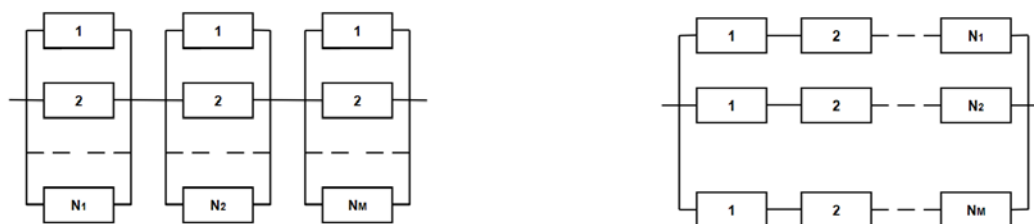
A - Serial-Parallel Network scheme   **B** - Parallel-Serial Network scheme
Figure 1. Serial-parallel and parallel-serial schemes.

Reliability of serial-parallel networks vs parallel networks it was well studied in the Chapter 3 of the book [8], but their conclusion was founded on the base of the static models. Next, we will adopt dynamic mathematical models for the networks represented above. More exactly, we suppose the following: the lifetime of each network's unit is described as a nonnegative r.v. whose cumulative distribution function (c.d.f.) is known and lifetimes of such units are independent random variables (i.r.v.). Total number of units N_k in the k-th sub-network is a constant (natural) number, where k = *1, 2, ..., M* and number *M* of all sub-networks is also a constant (natural) number. At the same time, each unit becomes irreplaceable at the time of its fall. On the other hand, reliability of the network coincides with the survival function, i.e., with the probability that the network will survive a longer time than *x*, which coincides, in fact, with the tail of c.d.f. for the lifetime of the entire network which we find in other specialist books [9-15].

In the following we are interested in:
- obtaining equations for calculation of the reliability of networks of type *A* and *B* under different conditions;
- to perform, on the basis of this equations, a comparative analysis of the networks from the point of view of their reliability in some particular cases.

2. Reliability of serial-parallel and parallel-serial networks where numbers of sub-networks and units in each sub-network are integers.

So, as a starting point in the study of reliability of more complicated mathematical models of networks of type **A** or **B** let us take the case when the numbers N_k, k = 1, 2, ..., *M* and *M* are integers. We consider that the lifetimes of all units compounding the Network are

non-negative, independent random variables (i.r.v.) X_{kj} with c.d.f. $F_{kj}(x)$ for j - th unit of the k - th sub-network, $j = \overline{1, N_k}$, $k = \overline{1, M}$. Then we observe that, for each type of Network, the lifetime of sub-networks is i.i.d.r.v. $Y_k = min (X_{kl}, X_{k2}, ..., X_{kNk})$, in the case of parallel-serial Networks, or $Y_k = max (X_{kl}, X_{k2}, ..., X_{kNk})$, in the case of serial-parallel Networks, $k = \overline{1, M}$. So, the lifetime of entire Network is the r.v. $U = min (Y_l, Y_2, ..., Y_M)$, in the case of serial-parallel Networks or is the r.v.

$V = max (Y_l, Y_2, ..., Y_M)$, in the case of parallel-serial Networks. But c.d.f of the r.v. $min (X_{kl}, X_{k2}, ..., X_{kNk})$ coincides with $1 - \prod_{j=1}^{N_k}(1 - F_{kj}(x))$ and c.d.f. of the r.v. $max (X_{kl}, X_{k2}, ..., X_{kNk})$ coincides with $\prod_{j=1}^{N_k} F_{kj}(x)$, k=1, 2, ..., M. In the above described conditions, we have that c.d.f.:

$$F_U(x) = P(U \leq x) = 1 - \prod_{k=1}^{M}\left[1 - \prod_{j=1}^{N_k} F_{kj}(x)\right] \tag{1}$$

$$F_V(x) = P(V \leq x) = \prod_{k=1}^{M}\left[1 - \prod_{j=1}^{N_k}(1 - F_{kj}(x))\right] \tag{2}$$

On the base of this equations we may: a) calculate survival / reliability function of serial-parallel and parallel-serial networks with integer number of subnetworks and integer number of units in each subnetwork and b) formulate the algorithm by which we can determine which Network is more reliable, **A** or **B**? Indeed:

a) Using equations (l) - (2) and description of survival / reliability function, we find that survival / reliability functions of serial-parallel and parallel-serial networks may be calculate, respectively, by the following equations

$$S_{S-P}(X) = P(U > x) = 1 - F_U(x) = \prod_{k=1}^{M}\left[1 - \prod_{j=1}^{N_k} F_{kj}(x)\right] \tag{3}$$

$$S_{P-S}(X) = P(V > x) = 1 - F_V(x) = 1 - \prod_{k=1}^{M}\left[1 - \prod_{j=1}^{N_k}(1 - F_{kj}(x))\right] \tag{4}$$

b) Formally, to determine which kind of the Network is more reliable, we must compare survival / reliability functions $S_{U_M}(x)$, $S_{V_M}(x)$ for concrete lifetime distributions $F_{kj}(x)$, $j = \overline{1, N_k}$, $k = \overline{1, M}$.

As example, let us take the particular case when lifetimes of all units are independent, identically distributed random variables (i.i.d.r.v.) X_{kj}, i.e., c.d.f. $F_{kj}(x) = F(x)$, $j = \overline{1, N_k}$, $k = \overline{1, M}$. Then, from Eq. (3) and Eq. (4), we deduce that the corresponding survival/reliability functions of serial-parallel A and parallel-serial B networks are

$$S_{S-P}(x) = P(U > x) = 1 - F_U(x) = \prod_{k=1}^{M}[1 - (F(x))^{N_k}] \tag{5}$$

$$S_{P-S}(x) = P(V > x) = 1 - F_V(x) = 1 - \prod_{k=1}^{M}[1 - (1 - F(x))^{N_k}] \tag{6}$$

In fact, in this particular case, Eq. (5) and Eq. (6) show us that the property of one network to be more reliable than the other does not depend of the lifetime distribution function. This is a consequence of the following

Proposition 1. *If lifetimes of all units are i.i.d.r.v., then survival / reliability functions* $S_{S-P}(x) \geq S_{P-S}(x)$ *or* $S_{S-P}(x) \leq S_{P-S}(x)$ *if and only if, respectively functions* $g_{S-P}(M, N_1, N_2, ..., N_M; q) \geq g_{P-S}(M, N_1, N_2, ..., N_M; q)$ *or* $g_{S-P}(M, N_1, N_2, ..., N_M; q) \leq g_{P-S}(M, N_1, N_2, ..., N_M; q)$ *for every* $q \in [0, 1]$,
where

$$g_{S-P}(M, N_1, N_2, ..., N_M; q) = \prod_{k=1}^{M}[1 - q^{N_k}] \text{ and}$$

$$g_{P-S}(M, N_1, N_2, \ldots, N_M; q) = 1 - \prod_{k=1}^{M}[1 - (1 - q)^{N_k}].$$

Proof. Before comparing the c.d.f. $F_U(x)$ and $F_V(x)$, let us take into account their characteristic properties. So, we observe that both functions are monotonous non-decreasing functions and $0 \leq F_U(x) \leq 1, 0 \leq F_V(x) \leq 1$, for every $x \in (-\infty, +\infty)$. If we denote by $q = F(x)$ for fixed x, then, according to the Eq. (5) and Eq. (6), the problem of the comparison of this c.d.f. will be equivalent to comparison of the functions

$$\prod_{k=1}^{M}[1 - q^{N_k}] \text{ and } 1 - \prod_{k=1}^{M}[1 - (1 - q)^{N_k}], for \ every \ q \in [0,1].$$

So, the problem of comparison of the survival / reliability functions $S_{S-P}(x), S_{P-S}(x)$ for every $x \in (-\infty, +\infty)$ will be equivalent to the comparison of the functions $g_{S-P}(M, N_1, N_2, \ldots, N_M; q), g_{P-S}(M, N_1, N_2, \ldots, N_M; q)$.

Remark 1. According to the proof, we observe, in fact, that the functions $g_{S-P}(M, N_1, N_2, \ldots, N_M; q), g_{P-S}(M, N_1, N_2, \ldots, N_M; q)$ represents, respectively, survival / reliability functions of above mentioned Networks when lifetime distribution $F(x)$ coincides with Uniform distribution on the interval [0, 1].

Before we give some examples, we mention that in case $M = 1$, i.e., network A becomes a serial type network and network B becomes a parallel type, the result is obvious. Specifically, parallel networks are always more reliable than serial networks. Therefore, in the following examples we will consider that the number M of subnets is greater than one.

Example 1. *a)* Let us consider serial-parallel and parallel-serial networks with number of subnetworks $M = 3$ and numbers $N_1 = 4$, $N_2 = 2$, $N_3 = 2$ of units in corresponding subnetworks. Then the following graphical representation of

$$g_{S-P}(3, 4, 2, 2; q) = (1 - q^4)(1 - q^2)^2 \ and$$

$$g_{P-S}(3, 4, 2, 2; q) = 1 - (1 - (1 - q)^4)(1 - (1 - q)^2)^2$$

show us that $g_{S-P}(3, 4, 2, 2; q) \geq g_{P-S}(3, 4, 2, 2; q)$ **not** for every $q \in [0,1]$, reprezented in figure 2.
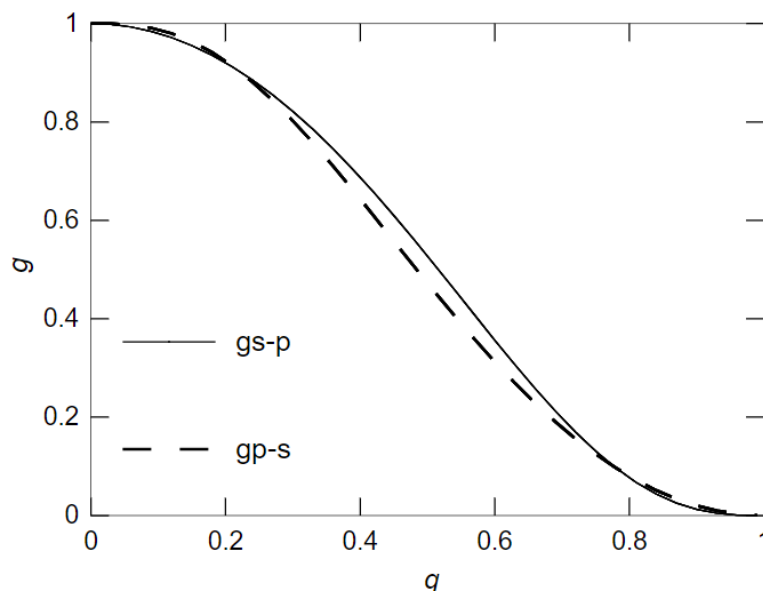
Figure 2. Grafics for g_{S-P} and g_{P-S} presented in the example 1*a*.

This means that the reliability of both networks cannot be unequivocally compared. More than that

b) Considering serial-parallel and parallel-serial networks with number of subnetworks *M=3* and numbers *N₁=2, N₂=2, N₃=2* of units in corresponding subnetworks, we show graphically in figure 3 that

$$g_{S-P}(3,2,2,2;q) = (1-q^2)^3 \leq g_{P-S}(3,2,2,2;q) = [1-(1-(1-q)^2)^3]$$
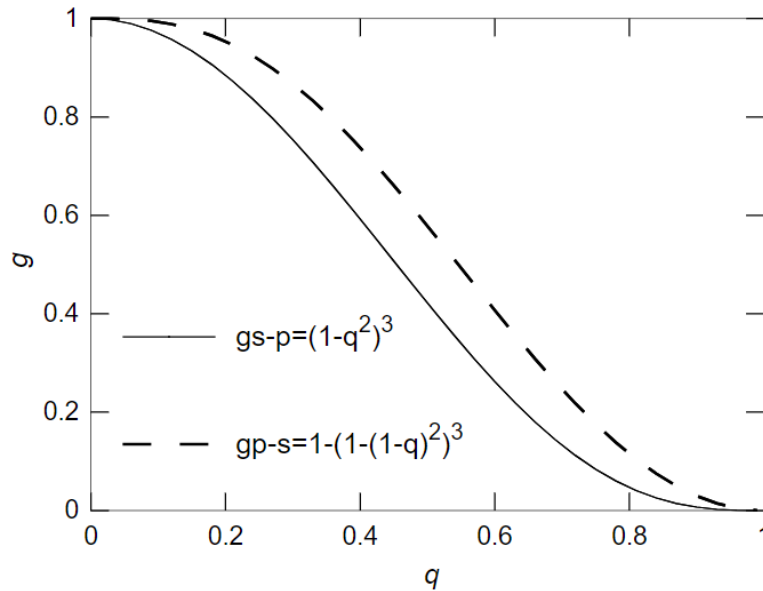
Figure 3. Grafics for g_{S-P} and g_{P-S} presented in the example 1*b*.

So, in this case, parallel-serial Network is more reliable than serial-parallel Network.

       Remark 2. According to the Proposition 1 and Remark 1 we note that the conclusions regarding the reliability of the type **A** network vs the reliability of the type **B** network in dynamic case remain valid in static case, i.e., when all units have one and the same probability of fall equal to q, where 0 <q <1. Moreover, both examples *a)* and *b)* above refer to the case where $min(N_1, N_2, ..., N_M) < M$, for which shows that the reliability of the Network **A** vs Network **B** it cannot be uniquely characterized.

3. Results

Now, we can prove the following

       Proposition 2. *For M > 1, if the lifetimes of the network units are i.i.d.r.v. and* $min(N_1, N_2, ..., N_M) \geq M$, *then the survival / reliability functions* $S_{S-P}(x) \geq S_{P-S}(x)$, *i.e., networks of type A are more reliable than networks of type B, regardless of the lifetime distribution of their units.*

Proof. Indeed, according to the Proposition 1, it is sufficient to prove that

$$\prod_{k=1}^{M}[1-q^{N_k}] \geq 1 - \prod_{k=1}^{M}[1-(1-q)^{N_k}],$$

i.e., that

$$g(M, N_1, N_2, ..., N_M; q) = \prod_{k=1}^{M}[1-q^{N_k}] + \prod_{k=1}^{M}[1-(1-q)^{N_k}] \geq 1$$

for every $q \in [0,1]$, as soon as $min(N_1, N_2, ..., N_M) \geq M$. The fact that $q \in [0,1]$, implies inequality

$$g(M, N_1, N_2, ..., N_M; q) \geq h(M; q) = [1 - q^M]^M + [1 - (1 - q)^M]^M.$$

The following graphical representation of $h(M; q)$, as a function of q for $M \in \{2, 3, 4\}$ represented in figure 4 , suggest as that $h(M; q) \geq 1$ with the local minimal value 1 in the points $q = 0$ and $q = 1$.
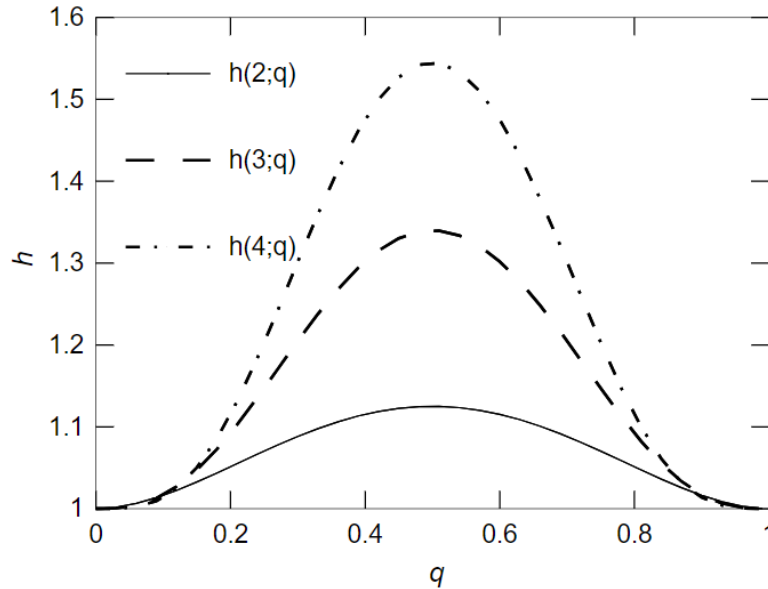
Figure 4. Grafics for $h(M; q)$ presented in the proposition 2.

Let us study the properties of $h(M; q)$, as a function of q for fixed $M > 1$. His derivative with respect to variable q is equal to

$$\frac{dh(M; q)}{dq} = M^2[(1 - (1 - q)^M)^{M-1}(1 - q)^{M-1} - (1 - q^M)^{M-1}q^{M-1}]$$

and equation $\frac{dh(M;q)}{dq} = 0$ have a single solution q = 1/2 in the interval (0, 1).

Furthermore, $\frac{dh(M;q)}{dq} > 0$ *for* $0 < q < \frac{1}{2}$, *and* $\frac{dh(M;q)}{dq} < 0$ *for* $\frac{1}{2} < q < 1$. This means that for each integer $M > 1$ the point q = 1/2 is a point of global maximum of $h(M; q)$ and this function is a concave of on the interval [0, 1] (see the previous graphical representation). So, $h(M; q) \geq 1$, because $h(M; 0) = h(M; 1) = 1$ are the points of minimum of this concave with the global maximum in the point q = 1/2. This implies that $g(M, N_1, N_2, ..., N_M; q) \geq h(M; q) \geq 1$, which means in this case that serial-parallel network is more reliable than parallel- serial network.

Example 2. a) Let us consider serial-parallel and parallel-serial networks with number of subnetworks M = 2 and numbers $N_l = 2$, $N_2 = 2$ of units in corresponding subnetworks. Then the following graphical representation in figure 5.

$$g_{S-P}(2, 2, 2; q) = (1 - q^2)^2 \text{ and } g_{P-S}(2, 2, 2; q) = 1 - (1 - (1 - q)^2)^2,$$

confirm that $g_{S-P}(2, 2, 2; q) \geq g_{P-S}(2, 2, 2; q)$ for every $q \in [0,1]$.
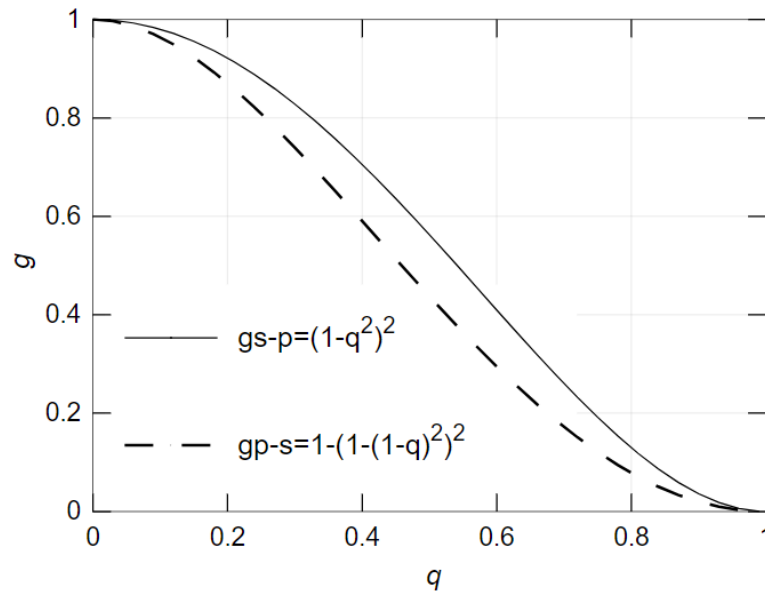
Figure 5. Grafics for g_{S-P} and g_{P-S} presented in the example 2a.

b) Now, let us consider serial-parallel and parallel-serial networks with number of subnetworks $M = 2$ and numbers $N_1 = 5$, $N_2 = 8$ of units in corresponding subnetworks. Then the following graphical representation of

$$g_{S-P}(2, 5, 8; q) = (1 - q^5)(1 - q^8) \text{ and } g_{P-S}(2, 5, 8; q) = 1 - (1 - (1 - q)^5)(1 - (1 - q)^8),$$

confirm too that $g_{S-P}(2, 5, 8; q) \geq g_{P-S}(2, 5, 8; q)$ for every $q \in [0, 1]$ reprezented in figure 6.
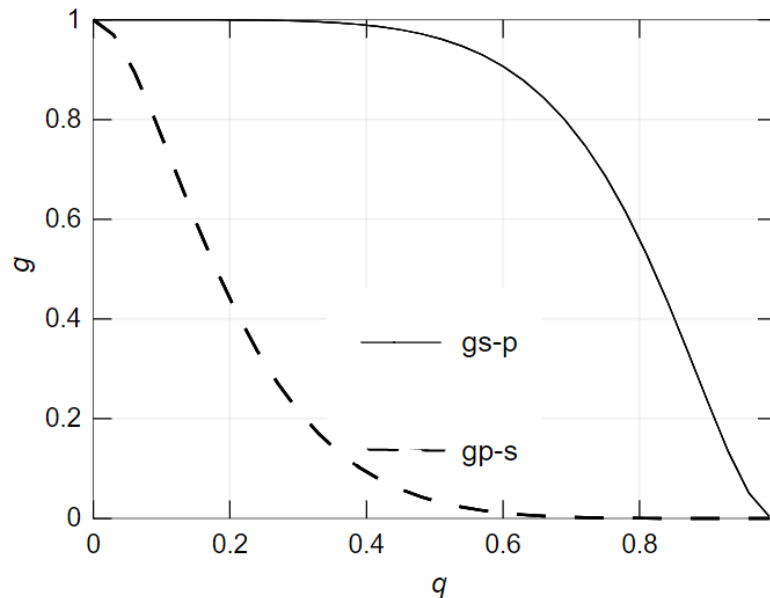
Figure 6. Grafics for g_{S-P} and g_{P-S} presented in the example 2b.

4. Discussion: comparative analysis with other models

The calculation Eq. (5) and Eq. (6) for the reliability of type *A* and *B* networks allow us to solve, in a dynamic case, the problem of reliability of the models proposed and researched in the paper [8], but in a static case. These are the following models:

C. **Serial-parallel.** The number of subnets is equal to N> 1 and the number of units in each subnet is equal to M> 1, the subnets being connected in serial and the units in parallel;

D. Parallel-serial. The number of subnets is equal to M> 1 and the number of units in each subnet is equal to N> 1, the subnets being connected in parallel and the units in serial. In both models **C** and **D** it is assumed that the lifetimes of the units are i.i.d.r.v. with c.d.f. *F(x)*. Then, from Eq. (5) and Eq. (6), we deduce that the corresponding survival/reliability functions of serial-parallel **C** and parallel-serial **D** networks are

$$S_{S-P}(x) = [1 - (F(x))^M]^N \qquad (7)$$

$$S_{P-S}(x) = 1 - \left[1 - (1 - F(x))^N\right]^M \qquad (8)$$

Using the same techniques of mathematical analysis of the survival/reliability function (7)-(8) we can prove

Proposition 3. *For every M > 1 and N>1, if the lifetimes of the network units are i.i.d.r.v., then the survival / reliability functions $S_{S-P}(x) \geq S_{P-S}(x)$, i.e., networks of type **C** are more reliable than networks of type **D**, regardless of the lifetime distribution of their units.*

In fact, this sentence extends the authors' conclusions [8] to the **C** and **D** dynamic models. In particular, example 2 a) above confirms these conclusions. The example below illustrates that these conclusions are also valid in M> N or M <N cases.

Example 3.   a) Let us consider serial-parallel and parallel-serial networks **C** and **D** when *N = 3* and *M=2*. Then the following graphical representation of

$$g_{S-P}(3,2,2,2;q) = (1 - q^2)^3 \; and \; g_{P-S}(2,3,3\,;q) = 1 - (1 - (1 - q)^3)^2,$$

confirm that $g_{S-P}(3,2,2,2;q) \geq g_{P-S}(2,3,3;q)$ for every $q \in [0,1]$.
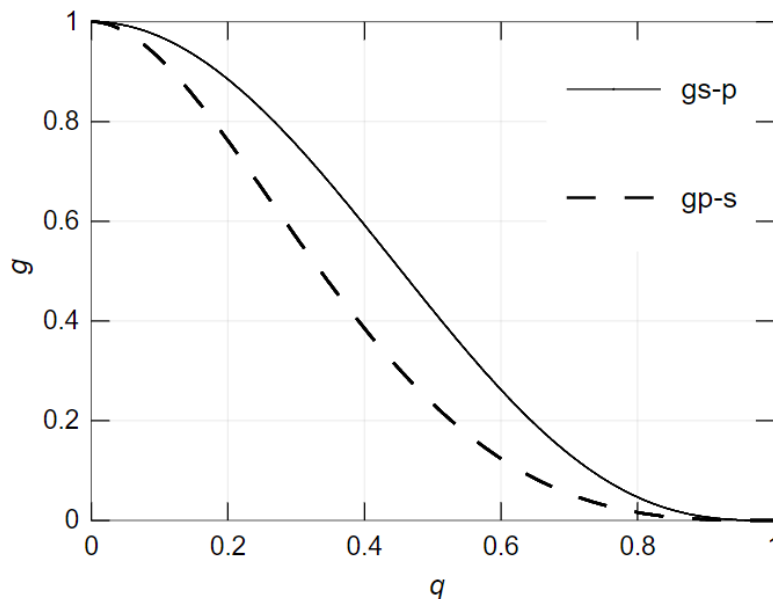
Figure 7. Grafics for g_{S-P} and g_{P-S} presented in the example 3a.

b) Conversely, let us consider serial-parallel and parallel-serial networks **C** and **D** when *N = 2* and *M=3*. Then the following graphical representation of

$$g_{S-P}(2,3,3;q) = (1 - q^3)^2 \; and \; g_{P-S}(3,2,2,2\,;q) = 1 - (1 - (1 - q)^2)^3,$$

confirm that $g_{S-P}(2,3,3;q) \geq g_{P-S}(3,2,2,2\,;q)$ for every $q \in [0,1]$.
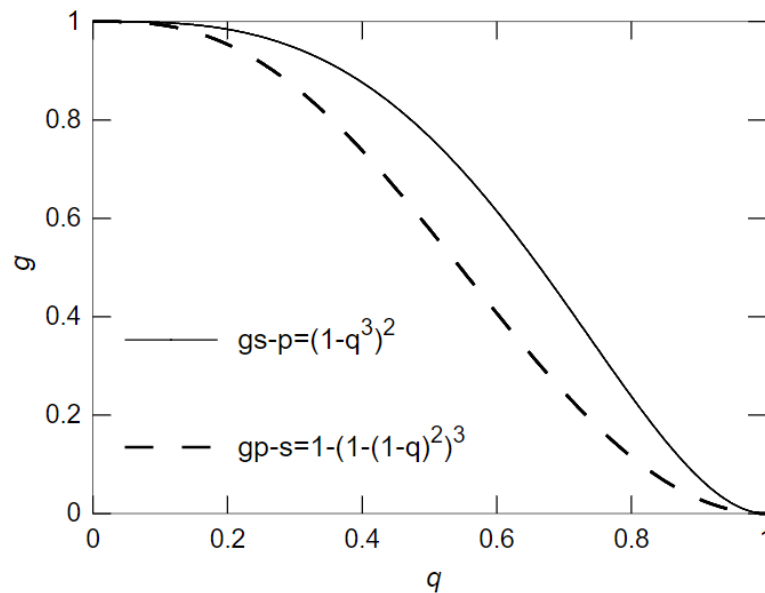
Figure 8. Grafics for g_{S-P} and g_{P-S} presented in the example 3b.

5. Conclusions

Equations for calculating their reliability was deduced for the serial-parallel and parallel-serial networks when numbers of the units and sub-networks are constant numbers, considering that the lifetimes of the units are independent random variables.

In the particular case, when the lifetimes of the units are i.i.d.r.v. we have proved (see Proposition 1), that the property of one network to be more reliable than the other does not depend of the lifetime distribution function.

The examples provided and Proposition 2 show us that, in this particular case, the serial-parallel networks of type **A** are always more reliable than the parallel-serial networks of type **B** as soon as the number *M* of the subnetworks is greater than 1 and the subnetwork with the smallest number of units exceeds this number *M*.

Our deductions based on dynamic models generalize, in fact, through Proposition 3, the conclusions of the other authors conclusions based on static models.

Conflicts of Interest: The authors declare no conflict of interest.

References
1. Louzada, F.; Bereta, M.P.E.; Franco, M.A.P. On the Distribution of the Minimum or Maximum of a Random Number of i.i.d. Lifetime Random Variables. *SCIRT* 2012, 3, pp. 350-353.
2. Adamidis, K.; Loukas, S. A lifetime distribution with decreasing failure rate. Statistics & Probability Letters. *SCIRT* 1998, 1 (39), pp. 35-42.
3. Kus, C. A new lifetime distribution. Computational Statistics and Data Analysis. *IDEAS* 2007, 51 (9), pp. 4497-4509.
4. Ushakov, I. Is reliability theory still alive? *RTA* 2007, 2 (1), pp. 6-19.
5. Leahu, A.; Munteanu, B. GH.; Cataranciuc, S. On the lifetime as the maximum or minimum of the sample with power serial distributed size. *Romai J* 2013, 9 (2), pp. 119-128.
6. Birolini, A. *Reliability Engineering: Theory and Practice.* Springer, Heidelberg, Germany, 2013, pp. 2-13.
7. Sheldon, M.R. *Introduction to Probability Models.* 10th ed., Elsevier, Oxford, UK, 2010, pp. 579-622.
8. Kapur, K.C.; Lamberson, L.R. *Reliability in engineering design.* Wiley, India, 2009, pp. 75-94.

9. Barlow, R. E.; Proschan, F. *Mathematical Theory of Reliability*. Wiley, New York, USA, 1965, pp. 5-9.

10. Gnedenko, B.V. *Mathematical aspects of reliability theory*. Publ. House Radio i Sviaz: Moscow, 1983, pp.79-133.

11. Ushakov, I.A. *Probabilistic Reliability Models*. John Wiley & Sons, New York, USA, 2012, pp. 1-18.

12. Platon, S.; Leahu, A. *Reliability of triage stations - modeling and estimation*. Printech, Bucureşti, Romania, 2001, pp.11-32 [in Romanian].

13. Rykov, V.V.; Balakrishnan N.; Nikulin M.S. *Mathematical and Statistical Models and Methods in Reliability*. Birkhäuser, Boston, MA, 2010, pp.123-128.

14. Munteanu B. Gh.; Leahu, A.; Cataranciuc, S. On The Limit Theorem For The Life Time Distribution Connected With Some Reliability Systems And Their Validation By Means Of The Monte Carlo Method. *American Institute of Physics Conference Proceedings*, USA, 2013, 1557, pp. 582-588.

15. Terruggia, R. Reliability analysis of probabilistic networks. Doctoral thesis, University of Turin, Italy, 2010, pp. 22-31.

Citation: Andrievschi-Bagrin, V.; Leahu, A. Reliability of serial-parallel networks vs reliability of parallel-serial networks with constant numbers of sub-networks and units. *Journal of Engineering Science* 2022, 29 (4), pp. 17-26. https://doi.org/10.52326/jes.utm.2022.29(4).02.

Publisher's Note: JES stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.

Submission of manuscripts: jes@meridian.utm.md

OPEN ACCESS

METHODOLOGICAL AND INSTRUMENTAL SUPPORT TO DEVELOPMENT OF ELECTRIC TRACTION SYSTEMS WITH ASYNCHRONOUS MOTOR

Alexandru Tarlajanu *, ORCID: 0000-0001-9310-1270

Technical University of Moldova, 168 Stefan cel Mare Blvd., Chisinau, Republic of Moldova
*Corresponding author: Alexandru Tarlajanu, *alexandru.tarlajanu@ie.utm.md*

Abstract. The present work aims to analyze from a methodological and instrumental point of view the development of asynchronous motor propulsion systems. For this purpose, PWM and SVM modulation methods, FOC and DTC vector control techniques, as well as their advantages and disadvantages are described. The theoretical bases and functional schemes of FOC and DTC control are given, including the asynchronous motor mathematical model, similar to the direct current motor model. Special attention is paid to creating of a common meaning for describing the components operation equations of the traction system, equations that can be used for system modeling and simulation. The paper also refers to a series of modern voltage converters with vector control, which are produced by several companies in different countries and are intended for the control of three-phase asynchronous motors. The results of the study create a basis for research and development of traction systems with multiphase asynchronous motors for urban electric passenger vehicles.

Keywords*: DTC control technique, FOC control technique, PWM modulation, SVM modulation, vector control.*

Rezumat. Această lucrare îşi propune să evidenţieze sprijinul metodologic şi instrumental pentru dezvoltarea sistemelor de propulsie cu motoare asincrone. În acest scop, sunt descrise metode de modulare PWM şi SVM, tehnici de control vectorial FOC şi DTC, precum şi avantajele şi dezavantajele acestora. Sunt prezentate bazele teoretice şi schemele funcţionale ale controlului FOC şi DTC, inclusiv modelul matematic al motorului asincron, similar modelului motorului cu curent continuu. O atenţie deosebită este acordată creării unui sens comun pentru descrierea ecuaţiilor de funcţionare a componentelor sistemului de tracţiune, ecuaţii care pot fi utilizate pentru modelarea şi simularea sistemului. Lucrarea se referă şi la o serie de convertoare moderne de tensiune cu control vectorial, care sunt produse de mai multe companii din diferite ţări şi sunt destinate controlului motoarelor asincrone trifazate. Rezultatele studiului stau la baza cercetării şi dezvoltării sistemelor de tracţiune cu motoare asincrone polifazate pentru vehiculele de pasageri electrice urbane.

Cuvinte cheie: *tehnica de control DTC, tehnica de control FOC, modulaţie PWM, modulaţie SVM, control vectorial.*

1. Introduction

Passenger transport has been and remains a challenge for the administration of large and medium-sized cities. An adequate response to this challenge involves the creation of a balanced and efficient urban transport system, using different types of transport, including subway, tram, trolleybus, bus, minibus, etc. At the same time, taking into account international and European regulations on the reduction of energy consumption and emissions, at present, the development of urban transport systems focuses on the predominant use of electric transport, including trolleybuses, which are more efficient and environmentally friendly [1]. For example, the efficiency of electric transport is 95-98%, and that of the internal combustion engine 20-30% [2].

In the evolution of electric traction systems, traction systems with three-phase asynchronous motors show a number of advantages compared to DC motors systems, including reduced size, weight and cost at equal powers, higher power and speed at the same size, high reliability, better operating characteristics, etc. For example, the efficiency of asynchronous traction motors is 3-5% higher that of the DC motor [3].

This paper aims to highlight and systematize the methods and instruments for electric traction systems realization and to create a basis for research and development of the traction systems with multiphase (more than three) motors for urban passenger electric vehicles. The expected objectives include: identification of the study object; description of the control principle and operating equations of PWM modulator; presentation of theoretical bases and block diagrams of vector control; description of FOC control algorithm of the asynchronous motor; presentation of DTC technique and of SVM modulation principle.

2. Traction system of urban electric passenger vehicles

It is known that the traditional traction system of urban electric passenger vehicles based on three-phase asynchronous motors consists of a three-phase converter, an asynchronous traction motor, a feedback loop and a data bus (figure 1).
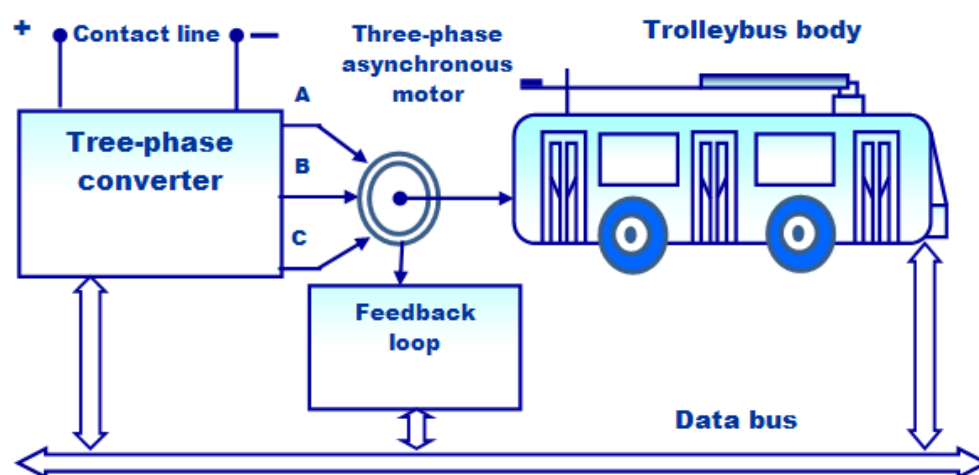
Figure 1. Electric traction system.

The three-phase converter converts DC energy from the contact line into a three-phase system with variable voltage and frequency that ensures the regulation of the asynchronous motor speed. It can be a current inverter or a voltage inverter. The current inverter has a simpler control system, but only changes the frequency of the output voltage and a voltage converter is required to regulate the inverter current. Meanwhile, the voltage inverter with

modulated signals changes both the frequency and the voltage of the three-phase output system. It does not require a voltage converter; instead, it has a sophisticated control system.

Due to widespread use of the results of technical-scientific progress and modern achievements in the field of electro mechanics, power electronics and information technologies, the traction system converter becomes multifunctional with communication capabilities, protection and diagnosis of equipment, automatic adjustment of traction and braking regimes, intelligent optimization, traffic monitoring, etc. [1, 3, 4].

In general, the voltage inverter consists of two distinctive components, namely, the control circuit and the power circuit (figure 2). The power circuit (figure 3) transforms the direct current energy into three-phase alternating current, using the control principle with width-modulated pulses. It can be made as a bridge inverter or a bridge arm inverter. The bridge arm inverter has several advantages (simpler design and operating principle, twice the number of power transistors, reduced overall dimensions, etc.) compared to the bridge inverter and is used more frequently in practice. The control circuit generates the control signals for the power circuit and ensures the complete control of the traction system.
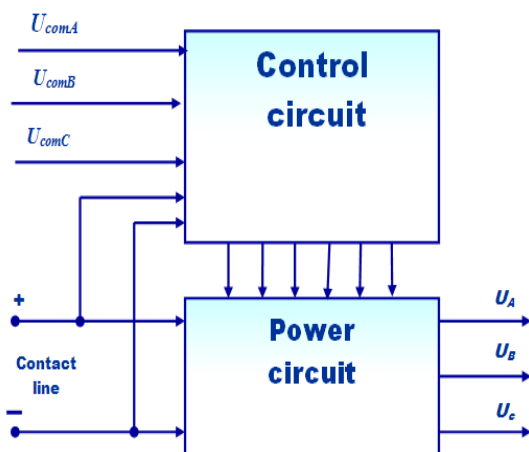
Figure 2. Voltage invertor.
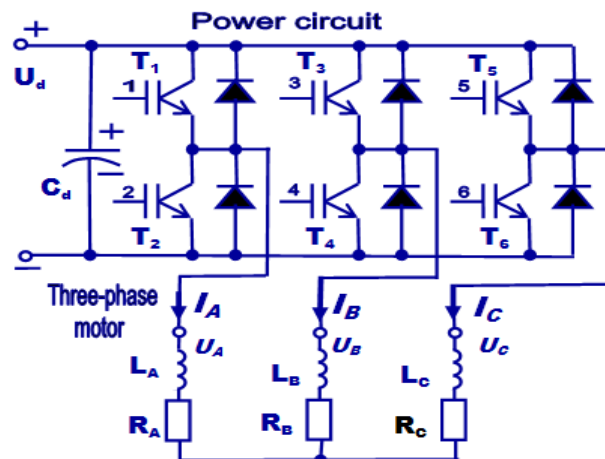
Figure 3. Power circuit.

For the transformation of direct current energy into three-phase alternating current, it is necessary that:

• the pairs of transistors T_1T_2, T_3T_4, T_5T_6 of the three bridge arms, work in series, with the blocking /conduction time, determined by the pairs of the respective control signals;

• to ensure a pause (a "dead time") when switching the bridge arm transistors from the driving mode to the blocking mode, in order to avoid short-circuit cases;

• to ensure a 120° electrical phase shift between the control signal pairs of the force circuit arms.

As the load of the inverter is inductive, transistors T_1-T_6 must be supplemented with discharge diodes [5].

The switching elements of the power circuit are built with Insulated Gate Bipolar Transistors (IGBT), with IGBT intellectual power modules or with Metal – Oxide – Semiconductor Field Effect Transistors (MOSFET), completed with discharge/recovery diodes.

As mentioned, the inverter control circuit ensures the switching regime of the power circuit transistors, according to the selected modulation principle. There are several operating principles of the control circuit, but, at the moment, the practical traction systems of urban passenger electric vehicles mainly use PWM modulation.

3. Width-modulated pulse control principle

PWM voltage inverter is widely described in the literature, here there will be specified some general aspects, necessary to describe its operating equations. The principle of PWM modulation is given in figure.4.
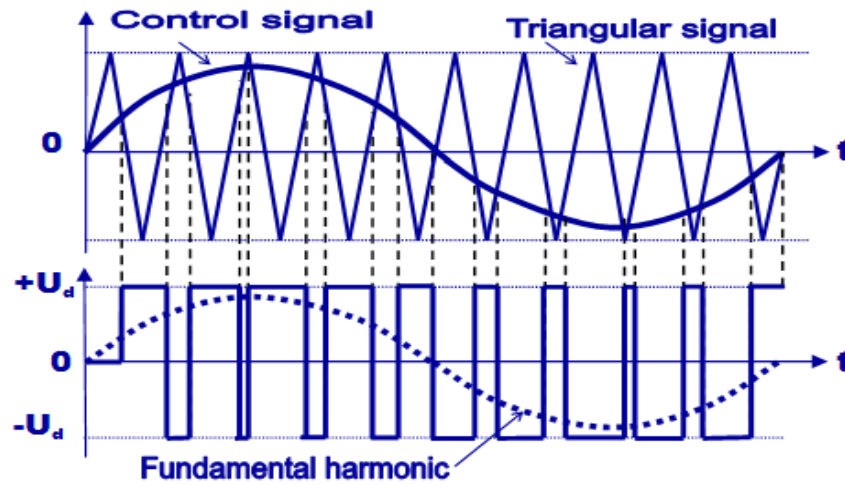
Figure 4. Principle of PWM modulation.

The PWM modulation technique consists in comparing a sinusoidal modulator control signal $U_{com}(t)$ with an alternating periodic triangular signal U_{tr}, as a carrier signal.

$$U_{com}(t) = U_{com} \sin \omega t = U_{com} \sin 2\pi f t \tag{1}$$

The intersection points of the two signals determine the switching frequency of the bridge arm transistors. By means of frequency f and the amplitude U_{com} of the control signal, the frequency and amplitude of the bridge arm output signal are varied. The output voltage (fundamental harmonic) of the PWM inverter is an approximation of a series of width-modulated pulses with variable filling factor, so that the average value on each switching period of the bridge arm transistors corresponds to an inverter output voltage sinusoid.

The following operating equations can be used to model and simulate the three-phase voltage inverter [5]. The inverter control signals U_{comA}, U_{comB}, U_{comC} are described by the equations system:

$$\left\{ \begin{array}{l} U_{comA}(t) = U_{com} \sin \omega t \\ U_{comB}(t) = U_{com} \sin(\omega t - 2\pi/3) \\ U_{comC}(t) = U_{com} \sin \omega t + 2\pi/3) \end{array} \right. \tag{2}$$

In these formulas, $\omega = 2\pi f$, and f is the frequency of the fundamental harmonics of the voltages U_A, U_B, U_C at the inverter output.

Line voltages U_{AB}, U_{BC}, U_{CA} and phase voltages U_A, U_B, U_C (figure 3) are described by the output voltages of the inverter arms U_{AN}, U_{BN}, U_{CN} with respect to the negative bar of the contact line.

$$\left\{ \begin{array}{l} U_{AN}(t) = \dfrac{U_d}{2} + \dfrac{U_d}{2} m_a \sin \omega t \\ U_{BN}(t) = \dfrac{U_d}{2} + \dfrac{U_d}{2} m_a \sin(\omega t - 2\pi/3) \\ U_{CN}(t) = \dfrac{U_d}{2} + \dfrac{U_d}{2} m_a \sin(\omega t + 2\pi/3), \end{array} \right. \tag{3}$$

$$\left\{\begin{array}{l} U_{AB}(t) = U_{AN}(t) - U_{BN}(t) \\ U_{BC}(t) = U_{BN}(t) - U_{CN}(t) \\ U_{CA}(t) = U_{CN}(t) - U_{AN}(t) \end{array}\right. \tag{4}$$

Phase voltages U_A, U_B, U_C and the currents I_A, I_B, I_C at the output of the inverter arms (figure 3) are determined with the systems of equations:

$$\left[\begin{array}{l} U_A = \frac{2}{3} U_{AN} - \frac{1}{3}(U_{BN} + U_{CN}) \\ U_B = \frac{2}{3} U_{BN} - \frac{1}{3}(U_{AN} + U_{CN}) \\ U_C = \frac{2}{3} U_{CN} - \frac{1}{3}(U_{AN} + U_{BN}); \end{array}\right. \tag{5}$$

$$\left[\begin{array}{l} U_A(t) = R_A I_A(t) + L_A \frac{dI_A(t)}{dt} + E_A(t) \\ U_B(t) = R_B I_B(t) + L_B \frac{dI_B(t)}{dt} + E_B(t) \\ U_C(t) = R_C I_C(t) + L_C \frac{dI_C(t)}{dt} + E_C(t). \end{array}\right. \tag{6}$$

But PWM modulation is not the only function of the control circuit; it also performs a series of asynchronous motor control techniques, including vector control, automatic motor speed adjustment, the traction system status monitoring and information etc. For this purpose, the electric traction system (figure 1) contains a feedback loop with sensors and transducers (current, voltage, speed), as well as an interface bus of the system components.

4. Theoretical bases of vector control

Vector control is an efficient technique for aligning the dynamic characteristics of the asynchronous motor with those of the DC motor. This refers, first of all, to the regulation in a wide range and with a high precision of the three-phase asynchronous motor rotation speed which depends on the load torque. Being of major importance for the performance of the traction system, this control technique is extensively described in current profile publications [6-10] and is based on the integration of asynchronous motor mathematical model with the voltage inverter model. The vector control, initially developed by Blaschke, provides the synthesis of a complex current of two components, one of them generates the flux, and the other - the electromagnetic torque of the motor.

According to [7], vector control is based on two fundamental principles:

1) Transformation of three-phase system (axes A, B, C) into an orthogonal system (figure 5), attached to the stator, with a direct current (X axis) and a square current (Y axis), which generates, respectively, the flow and electromagnetic torque of the motor (Clarke transformation).

2) The transition from a stationary reference system to a dynamic system (figure 6) that rotates synchronously with the electromagnetic torque of the rotor, by transforming a sinusoidal quantity of the stationary reference system into a constant quantity of the dynamic system that rotates with the frequency of the sinusoidal quantity (Park transformation).

The Clarke transformation is performed with the following equations [8, 9].

$$\begin{bmatrix} I_{XS}(t) \\ Ii_{YS}(t) \end{bmatrix} = \begin{bmatrix} 1 & \cos(\varphi) & \cos(2\varphi) \\ 0 & \sin(\varphi) & \sin(2\varphi) \end{bmatrix} \cdot \begin{bmatrix} I_{As}(t) \\ I_{Bs}(t) \\ I_{Cs}(t) \end{bmatrix}, \tag{7}$$

where: $I_{As}(t), I_{Bs}(t), I_{Cs}(t)$ are the three-phase system stator currents; $I_{Xs}(t), I_{Ys}(t)$ - stator current $I_s(t)$ components in the X-Y system; $\varphi = 2\pi/3$.
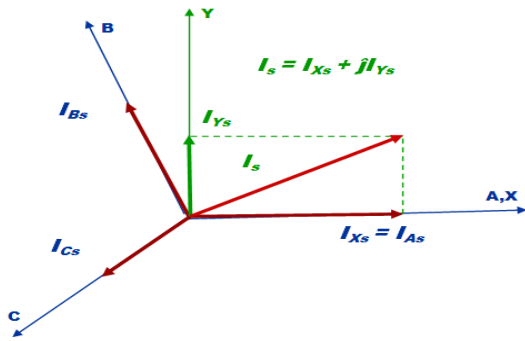
Figure 5. Clarke transformation.   **Figure 6.** Park transformation.
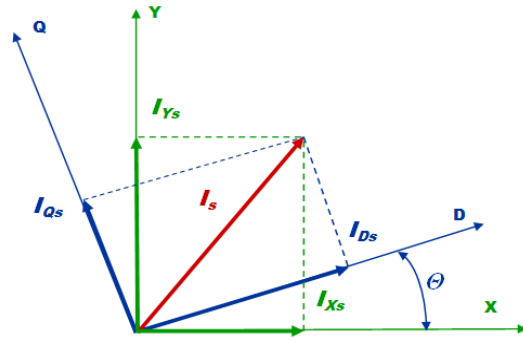
Transition from a stationary reference system to a dynamic system and the relations between the two systems quantities are described a:

$$\begin{bmatrix} I_{Ds}(t) \\ I_{Qs}(t) \end{bmatrix} = \begin{bmatrix} \cos(\Theta) & -\sin(\Theta) \\ \sin(\Theta) & \cos(\Theta) \end{bmatrix} \cdot \begin{bmatrix} I_{Xs}(t) \\ I_{Ys}(t) \end{bmatrix}, \tag{8}$$

where: $\Theta = \omega t$ is the vector I_s angle of rotation, and ω - the motor rotation speed or its mechanical angular velocity.

The mathematical model of the asynchronous motor (Figure 3) in the stationary X-Y system, attached to the stator, is described by the following equations [6]:

• Stator voltage differential equations:

$$\begin{cases} U_{Xs} = R_s I_{Xs} + \dfrac{d}{dt}\psi_{Xs}; \\ U_{Ys} = R_s I_{Ys} + \dfrac{d}{dt}\psi_{Ys}, \end{cases} \tag{9}$$

where: R_s is the stator phase resistance, and ψ_{Xs}, ψ_{Ys}, - the stator flux components.

• Differential equations of rotor voltage:

$$\begin{cases} U_{Xr} = 0 = R_r I_{Xr} + \dfrac{d}{dt}\psi_{Xr} + \omega\psi_{Yr}; \\ U_{Yr} = 0 = R_r I_{Yr} + \dfrac{d}{dt}\psi_{Yr} - \omega\psi_{Xr}, \end{cases} \tag{10}$$

where: R_r is the rotor phase resistance.

• The stator and rotor flux equations, expressed by the spatial vectors of their currents:

$$\begin{cases} \psi_{Xs} = L_s I_{Xs} + L_m I_{Xr}, \ \psi_{Ys} = L_s I_{Ys} + L_m I_{Yr}; \\ \psi_{Xr} = L_r I_{Xr} + L_m I_{Xs}, \ \psi_{Yr} = L_r I_{Yr} + L_m I_{Ys}, \end{cases} \tag{11}$$

where: L_s and L_r are, respectively, the stator and the rotor phase inductance.

L_m - the mutual stator-rotor inductance.

• Torque equation, expressed by the spatial vectors of flux and currents:

$$m_e = \frac{3}{2}P_p(\psi_{Xs}I_{Ys} + \psi_{Ys}I_{Xs}), \tag{12}$$

where: P_p is the number of poles per phase.

When switching from the stationary *X-Y* reference system to the *D-Q* system, the mathematical model of the motor is described by the following equations [6]:

$$
\begin{cases}
U_{Ds} = R_s I_{Ds} + \dfrac{d}{dt}\psi_{Ds} - \omega_s\psi_{Qs}; \\[4pt]
U_{Qs} = R_s I_{Qs} + \dfrac{d}{dt}\psi_{Qs} + \omega_s\psi_{Ds}; \\[4pt]
U_{Dr} = 0 = R_r I_{Dr} + \dfrac{d}{dt}\psi_{Dr} - (\omega_s - \omega)\psi_{Dr}; \\[4pt]
U_{Qr} = 0 = R_r I_{Qr} + \dfrac{d}{dt}\psi_{Qr} - (\omega_s - \omega)\psi_{Qr}; \\[4pt]
\psi_{Ds} = L_s I_{Ds} + L_m I_{Dr}\,, \;\; \psi_{Qs} = L_s I_{Qs} + L_m I_{Qr}; \\[4pt]
\psi_{Dr} = L_r I_{Dr} + L_m I_{Ds}\,, \;\; \psi_{Qr} = L_r I_{Qr} + L_m I_{Qs}; \\[4pt]
m_e = \dfrac{3}{2}P_p(\psi_{Ds} I_{Qs} + \psi_{Qs} I_{Ds}),
\end{cases}
\tag{13}
$$

where: ω_s is the synchronous angular speed of the motor.

The operating equations "Eq. (1) - Eq. (13)" are the modeling and simulation basis of vector control systems with asynchronous motor. To implement vector control, the FOC and DTC techniques are frequently used.

5. Field-oriented control of the asynchronous motor

The field-oriented control, as one of the vector control approaches, provides the decomposition of the stator current into the components generating flux and torque with their separate control. The vector control diagram of the asynchronous motor with PVM modulation is given in figure 7.
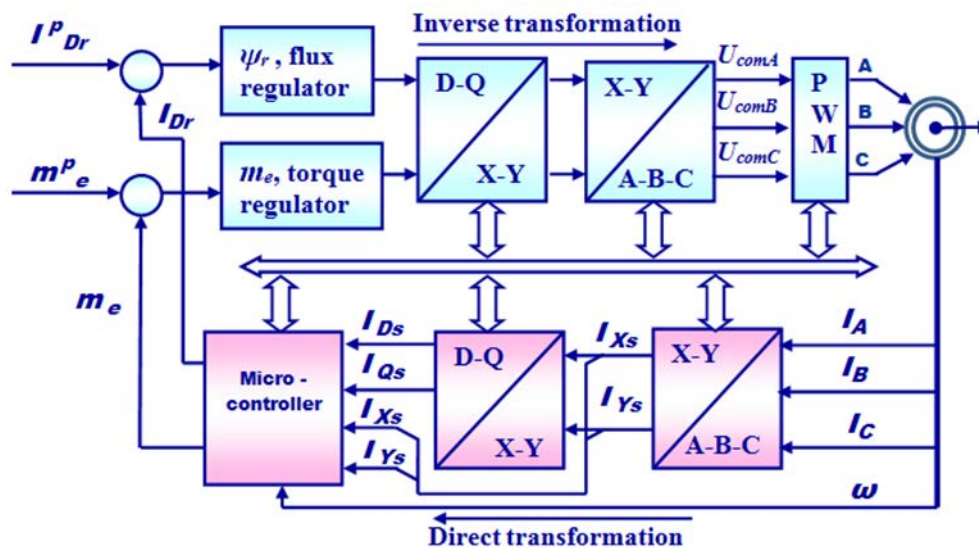
Figure 7. Field-oriented control scheme.

The microcontroller: assists the direct/inverse transformations of the reference systems and of the respective signals; ensures the feedback loop (signals I_{Dr}, m_e) of the flux and torque regulators; leads the PVM modulation process; ensures the analog/digital and digital/analog transformations etc. Flux and torque regulators can be PID-type standard regulators or can be replaced with microcontroller special software applications, if the response time of the system allows it. I_{Dr}^p, m_e^p signals determine the prescribed or reference

values of the motor flux and torque, while I_{Dr} and m_e are the calculated values that characterize the motor current state.

According to [6], inverse transformation of the reference system *D-Q* into *A-B-C* (Fig. 7) and generation of control signals for the power circuit transistors (Fig. 3) can also be achieved by the Space Vector Modulation (SVM) technique. This combination is known as SVM-PWM control technique.

6. Direct torque control technique

As mentioned, FOC technique controls the asynchronous motor speed and torque indirect, by decomposing the stator current into the flux and torque generating currents with their separate control. That requires multiple calculations and transformations and, together with PVM modulation, increases the reaction time of the traction system, which diminishes the control process quality indices and limits the FOC application.

To improve the performance of the control system, a Direct Torque Control (DTC) technique is used (fig. 8). DTC is similar to FOC technique, except that it is based on the flux and torque decomposition with their independent control [8].
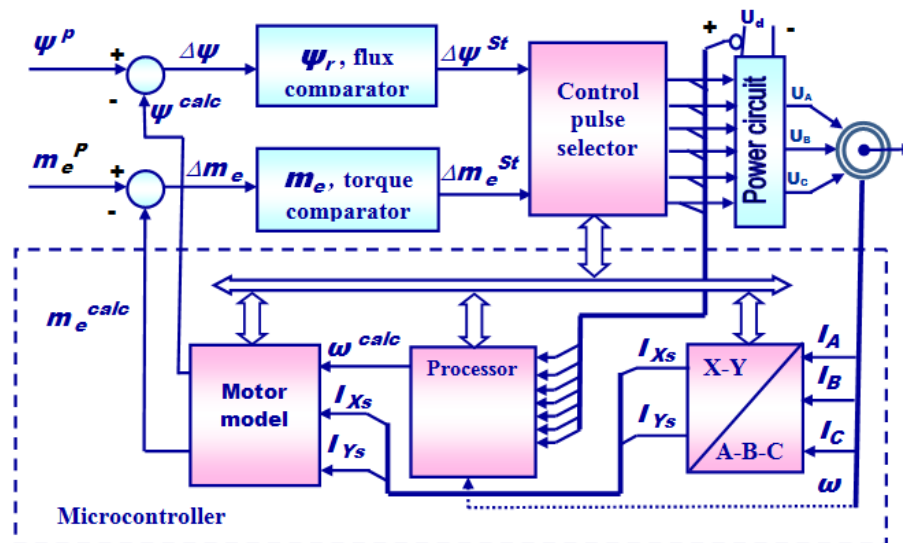
Figure 8. Direct torque control scheme.

In principle, the DTC technique ensures direct flux and torque calculation without the use of speed sensors or rotor shaft positioning. This technique is based on performant signal processing technologies and the creation of advanced mathematical models of the asynchronous motor, which allow the high-precision simulation of its operating characteristics. As the created model exactly reflects the characteristics of the physical motor, the calculation of the flux and the current torque is performed in real time, with high precision and speed, which contributes to improving the performance of the control system [8].

The flux and torque comparators determine the current state of the $\Delta\psi$ flow and Δm_e torque error, compared to their prescribed values ψ^P, m_e^P and a hysteresis band. The flux comparator is bi-positional and determines the state of increase $\Delta\psi^i$ or decrease $\Delta\psi^d$ of the error, and the torque comparator is three-positional and determines the states of increase Δm_e^i, decrease Δm_e^d and equality $\Delta m_e=$ of the torque error. The outputs of the comparators are connected to the control pulse selector which performs the SVM modulation technique. For this purpose, eight state variants (table 1) of the power circuit (fig. 3) are identified and,

respectively, eight stator voltage vectors, depending on the switching combinations of transistors T_1-T_6 [12, 13].

The inverter output voltage vector or stator voltage vector is determined by the equation

$$U_s = \frac{2}{3}U_d\left(U_A + e^{\frac{j2\pi}{2}}U_B + e^{\frac{j4\pi}{2}}U_C\right) \tag{14}$$

Vectors V_0 and V_7 are zero and when applied the motor flux and torque become zero [12].

It is important that selection of the stator voltage vector depends not only on the outputs of the flux and torque comparators, but also on the current positioning of the stator flux vector.

Table 1

Power circuit states

Inverter state	Voltage vector	State code	Inverter arm		
			U_A	U_B	U_C
State 0	V_0	000	T_2	T_4	T_6
State 1	V_1	100	T_1	T_4	T_6
State 2	V_2	110	T_1	T_3	T_6
State 3	V_3	010	T_2	T_3	T_6
State 4	V_4	011	T_2	T_3	T_5
State 5	V_5	001	T_2	T_4	T_5
State 6	V_6	101	T_1	T_4	T_5
State 7	V_7	111	T_1	T_3	T_5

This is why the trajectory of the stator circular flux vector is divided into six symmetrical sectors (figure 9), and the microprocessor determines the current stator voltage vector based on the data in table 2 [10-12]. Fig.9 shows the selection variants of the voltage vector, when the stator flux vector is in sector 1.

Table 2

Voltage vector selection

Error		Sector					
Flux	Torque	S_1	S_2	S_3	S_4	S_5	S_6
	Δm_e^i	V_2	V_3	V_4	V_5	V_6	V_1
$\Delta\psi^i$	$\Delta m_e^=$	V_0	V_7	V_0	V_7	V_0	V_7
	Δm_e^d	V_6	V_1	V_2	V_3	V_4	V_5
	Δm_e^i	V_3	V_4	V_5	V_6	V_1	V_2
$\Delta\psi^d$	$\Delta m_e^=$	V_7	V_0	V_7	V_0	V_7	V_0
	Δm_e^d	V_5	V_6	V_1	V_2	V_3	V_4

Based on the above, the FOC and DTC control techniques, as well as the PWM and SVM modulation methods have both advantages and disadvantages, and the selection of an optimal option depends on the pursued objectives. This must be done in the research and development process.
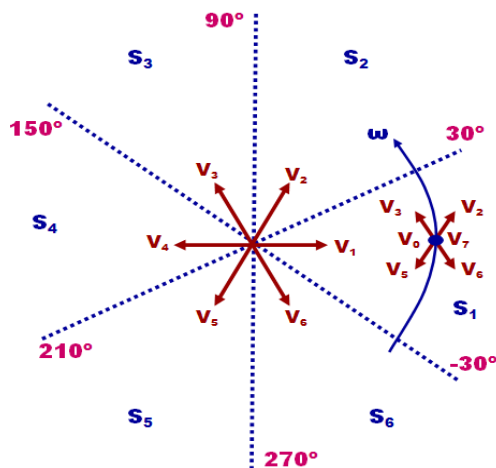
Figure 9. Stator flux sectors.

It should be mentioned that according to [14-18], the theoretical approaches and techniques described in this paper, with some modifications and completions, can be successfully used in the research and development of control systems with poly-phase asynchronous motors, including six-phase motors.

7. Conclusions

The current development trend of traction systems for urban electric passenger transport is manifested by the replacement of direct current motors with asynchronous motors. This involves the use of the voltage inverter and asynchronous motor control techniques, similar and as efficient as those of the DC motor. More frequently, the voltage inverter is made on the PVM and SVM principles, using the controlled power transistors. At the moment, vector control is the optimal control solution for the asynchronous motor. For this purpose, FOC and PWM control techniques are used. In this paper, continuity and a common sense is ensured in the description of operating equations of the components of traction systems with asynchronous motors, which can be used for research and development of control systems with poly-phase motors, including six-phase motors.

The paper was presented at the works of the conference SIELMEN 2021.

Conflicts of Interest: The authors declare no conflict of interest.

References
1. Ehsani, M; Gao, Y.; Gay, S.E.; Emadi, A. *Modern electric, hybrid electric, and fuel cell vehicles: fundamentals, theory, and design*. CRC Press LLC, Boca Raton, Florida, SUA, 2004, 387 p.
2. Electrical Buses: Development and Implementation in Chisinau Municipality, Moldova. Available online: https://www.researchgate.net/publication/337502147 (accessed on 10 August 2022).
3. Urban electric vehicles traction: achievements and trends. Available online: https://www.google.com/search?q=Urban+electric+vehicles+traction (accessed on 18 August 2022).
4. Tracțiune electrică. Available online: https://docplayer.gr/45606721 (accessed on 14 September 2022).

5.   Invertorul PVM trifazat de tensiune. Available online: http://www.euedia.tuiasi.ro/lab_ep/ep_files/Lucrarea_26.pdf (accessed on 26 October 2022).

6. 3-Phase AC Induction Vector Control Drive with Single Shunt Current Sensing. Available online: https://www.nxp.com/docs/en/reference-manual/DRM092.pdf (accessed on 30 August 2022).

7.   Induction motor control by vector control method. Available online: http://irjes.com/Conference/Raee/D1722.pdf (accessed on 16 September 2022).

8.   Induction motor modeling for vector control purposes. Available online: https://www.researchgate.net/publication/269517505 (accessed on 26 September 2022).

9.   Clarke's and Park's Transformations. Available online: http://www.siegereng.com.br/img/artigo16/Part%204%20Clark&Park.pdf (accessed on 28 September 2022).

10. What is direct torque control (DTC) for AC induction motors? Available online: https://www.motioncontroltips.com/what-is-direct-torque-control-dtc-for-ac-induction-motors/ (accessed on 18 September 2022).

11. Direct Torque Control Strategies of Induction machine: Comparative studies. Available online:https://www.intechopen.com/books/direct-torque-control-strategies (accessed on 06 October 2022).

12. Design of Direct Torque Controller of Induction Motor (DTC). Available online: https://www.researchgate.net/publication/286703597 (accessed on 20 September 2022).

13. Implementation of the vector control system for traction asynchronous motors. Available online: https://elth.ucv.ro/fisiere/anale/2011/27.pdf (accessed on 28 August 2022).

14. Realization of an Asynchronous Six-Phase Induction Motor Drive Test-Rig. Available online: https://www.researchgate.net/publication/318442386_Realization_of_an_asynchronous_six-phase_induction_motor_drive_test-rig (accessed on 26 September 2022).

15. Behavior of a six phase induction motor fed by multilevel inverter. Available online: https://www.researchgate.net/publication/297732620_Behavior_of_a_six_phase_induction_motor_fed_by_multilevel_inverter (accessed on 12 October 2022).

16. Direct torque and flux control of asymmetrical six-phase induction motor with zero sequence components elimination. Available online: http://revue.elth.pub.ro/upload/88839007_NAbjadi_RRST_1_2017_pp_36-41.pdf (accessed on 16 October 2022).

17. Independent Field-Oriented Control of Two Split-Phase Induction Motors From a Single Six-Phase Inverter. Available online: https://ieeexplore.ieee.org/document/1512470 (accessed on 26 October 2022).

18. Development of Vector Control Structures for Traction Systems with Six-Phase Asynchronous Motors. Available online: https://ieeexplore.ieee.org/document/9600403 (accessed on 26 October 2022).

Submission of manuscripts: jes@meridian.utm.md

OPEN ACCESS

DETERMINATION OF SOME SOLUTIONS OF THE STATIONARY 2D NAVIER-STOKES EQUATIONS

Iurie Baltag *, ORCID: 0000-0003-3426-1287

Technical University of Moldova, 168 Stefan cel Mare Blvd., Chisinau, Republic of Moldova
*Corresponding author: Iurie Baltag, *iurie.baltag@mate.utm.md*

Abstract. In this paper, various solutions of the stationary Navier-Stokes equations, which describe the planar flow of an incompressible liquid (or gas), are determined, i.e., solutions containing the components of the velocity of flow - the functions *u*, *v* and the created pressure - *P*. The paper contains three proven theorems, as well as various examples and particular examined cases. Applying Theorem 1, we can find various solutions, where the velocity components represent the imaginary and real parts of a differentiable function of a complex variable. Theorem 2 allows us to determine solutions, where the velocity components are expressed by the partial derivatives of the solutions of Laplace's equation of a special form. It is to be mentioned that these theorems give us solutions that do not depend on the viscosity parameter λ. In theorem 3, an original method for obtaining a series of solutions of the Navier-Stokes equations is presented, in which the viscosity coefficient λ participates explicitly; these solutions cannot be obtained by applying Theorems 1 or 2. The paper contains a large number of particular cases examined and examples of exact determined solutions.

Keywords: *stationary two-dimensional Navier-Stokes equations, system of equations with partial derivatives, exact solutions, method of separation of variables, viscosity, pressure, velocity of plane flow of a liquid or gas.*

Rezumat. În această lucrare se determină diverse soluții ale ecuațiilor staționare Navier-Stokes, care descriu curgerea plană a unui lichid (sau gaz) incompresibil, și anume soluții ce conțin componentele vitezei fluxului de curgere - funcțiile *u*, *v* și presiunea creată – *P*. Lucrarea de față conține trei teoreme demonstrate și diverse exemple și cazuri particulare examinate. Aplicând teorema 1, putem afla diverse soluții, în care componentele vitezei reprezintă partea imaginară și cea reală a unei funcții diferențiabile de variabilă complexă. Teorema 2 ne permite să determinăm soluții, în care componentele vitezei sunt exprimate prin derivatele parțiale ale soluțiilor ecuației lui Laplace de o formă specială. Menționăm, că aceste teoreme ne oferă soluții ce nu depind de parametrul vâscozității λ. În teorema 3 este expusă o metodă originală de obținere a unui șir de soluții ale ecuațiilor Navier-Stokes, în care participă în mod explicit coeficientul vâscozității λ; aceste soluții nu pot fi obținute

aplicând teoremele 1 sau 2. Lucrarea conține un număr mare de cazuri particulare examinate și exemple de soluții exacte determinate.

Cuvinte cheie: *ecuații staționare bidimensionale Navier-Stokes, sistem de ecuații cu derivate parțiale, soluții exacte, metoda separării variabilelor, vâscozitate, presiune, viteza fluxului de curgere plană a unui lichid sau gaz.*

1. Introduction

In the present paper, the Navier-Stokes equations are studied in the two-dimensional (2D) case. In this case the Navier-Stokes equations represent a system containing three partial differential equations with three unknown functions.

Until today, the examined problem has not been definitively solved even in the case of stationary equations, that is, equations that describe the processes of the planar flow of a liquid or gas that does not vary in time.

The complexity of the problem lies in the fact that the first two equations in the system are non-linear.

A method is not developed that would allow us to determine all the solutions of this system. Determining the solutions of the system of Navier-Stokes equations is an important mathematical problem and has various applications in fluid and gas mechanics.

The following system of partial differential equations is examined in this paper:

$$\begin{cases} \frac{P_x}{\mu} + uu_x + vu_y = \lambda(u_{xx} + u_{yy}) + F_x \\ \frac{P_y}{\mu} + uv_x + vv_y = \lambda(v_{xx} + v_{yy}) + F_y \\ \qquad u_x + v_y = 0 \end{cases} \tag{1}$$

where: $x, y \in R$; $P = P(x,y)$; $F = F(x;y)$; $u = u(x,y), v = v(x,y)$; $u_x = \dfrac{\partial u}{\partial x}$.

System (1) describes the stationary processes of planar flow of an incompressible liquid or gas. Regarding the derivation of the equations of system (1) and the meaning of the physical processes described by this system, consult the works [1 - 3].

The unknowns of system (1) are the following three functions: *P*, which represents the created pressure; *u* and *v*, which represent the components of the flow velocity of a liquid or gas.

The given external force is *F* and has a potential nature, that is, its components are equal to the partial derivatives of this force - *Fx* and *Fy*. The constants $\lambda > 0$ and $\mu > 0$ are the parameters determined by the viscosity and density of the examined liquid (gas). We mention here, that the viscosity parameter has the form $\lambda = \dfrac{c_0}{R_e}, c_0 > 0,$ where R_e is the Reynolds number.

Some exact solutions of the system (1) are obtained in the papers [4] - [7]. In the paper [8] a series of solutions of the examined system are indicated only for the components of the flow velocity, without determining the pressure.

Suppose that in the plane connected domain *D* functions *P(x, y)*, *u(x, y)*, *v(x, y)* and *F(x, y)* admit partial derivatives continuous up to and including the second order, then the theorems 1-3 stated below is just.

2. Solutions, where the velocity components represent the imaginary part and the real part of a differentiable function of complex variable

Theorem. 1. If $f(z)$ is a function of complex variable $z = x + iy$, differentiable at any interior point $(x; y)$ of domain D, then system (1) admits solutions of the following form in this domain:

$$u = Imf, \quad v = Ref; \quad P = [F - 0{,}5(u^2 + v^2) + C]\mu. \tag{2}$$

where C is an arbitrary constant.

Demonstration T. 1. System (1) is equivalent to the following system:

$$\begin{cases} \dfrac{P_x}{\mu} - F_x + uu_x + vv_x = \lambda\Delta u - v(u_y - v_x) \\ \dfrac{P_y}{\mu} - F_y + uu_y + vv_y = \lambda\Delta v + u(u_y - v_x) \\ u_x + v_y = 0 \end{cases} \tag{3}$$

where $\Delta u = u_{xx} + u_{yy}, \ \Delta v = v_{xx} + v_{yy}$.

Noting that
$$G = \frac{1}{\mu} P - F + 0{,}5(u^2 + v^2) \tag{4}$$

Then from (3) results that
$$\begin{cases} G_x = \lambda\Delta u - v(u_y - v_x) \\ G_y = \lambda\Delta v + u(u_y - v_x) \\ u_x + v_y = 0 \end{cases} \tag{5}$$

Thus, system (1) is equivalent to system (5). Since $Gxy = Gyx$ we derive the first equation from (5) in relation to y, and the second in relation to x and equate the right sides of the obtained equations. As a result, we obtain the following equation for determining the functions u and v:

$$\lambda\Delta(u_y - v_x) - u(u_y - v_x)_x - v(u_y - v_x)_y = 0 \tag{6}$$

Besides this, u and v have to verify also the last equation from the system (5):

$$u_x + v_y = 0. \tag{7}$$

Therefore, the functions u and v can be determined separately, independently of the pressure P, from the system, which consists of equations (6) and (7).

May it be $u = Imf, \ v = Ref, \ f = v(x; y) + iu(x; y),$ were f is a function of complex variable $z = x + iy$, differentiable at any interior point $(x; y)$ of domain D. Then from Cauchy – Riemann conditions [9] we obtain:

$$\begin{cases} v_x = u_y \\ v_y = -u_x \end{cases} \Leftrightarrow \begin{cases} u_y - v_x = 0 \\ u_x + v_y = 0 \end{cases} \tag{8}$$

The second equation in (8) coincides with (7), and from the first it follows that these functions verify equation (6). Since the functions u and v admit continuous derivatives up to the second order inclusive in D, they have continuous mixed derivatives in this domain and from (8) we deduce that they are solutions of Laplace's equation, meaning $\Delta u = u_{xx} + u_{yy} = 0, \Delta v = 0$.

Then from (5) we obtain that $\begin{cases} G_x = 0 \\ G_y = 0 \end{cases} \Rightarrow G(x; y) = C - \text{const.}$

We substitute this result in (4) and express the pressure P. **T. 1.** is proved.

Below we will give 2 examples of determining the solutions of system (1) according to theorem 1.

Example 1. If $f(z) = e^{z^2}$, then we obtain the following solutions of system (1):

$$\begin{cases} u = e^{x^2-y^2} \sin(2xy); v = e^{x^2-y^2} \cos(2xy), \\ P = (F - 0.5e^{2(x^2-y^2)} + C)\mu \end{cases} \quad D = OXY. \tag{9}$$

Example 2. If $f(z) = C_0(z-z_0)^{-1}$, then

$$\begin{cases} u = \frac{C_0(y_0-y)}{(x-x_0)^2+(y-y_0)^2}; \ v = \frac{C_0(x-x_0)}{(x-x_0)^2+(y-y_0)^2}, \\ P = [F - \frac{0.5C_0^2}{(x-x_0)^2+(y-y_0)^2} + C]\mu \end{cases} \quad D = OXY\backslash\{M(x_0; y_0)\}. \tag{10}$$

In solutions (9) and (10) C and C_0 are arbitrary constants.

In [7] the solutions (10) with the constant $C_0 = 4(R_e)^{-1}$ are obtained by a more complicated method. They represent the flow velocities of a liquid and the pressure in the vicinity of the orifice located at the point $M(x_0; y_0)$.

3. Solutions, where the velocity components are solutions of Laplace's equation of a special form

Theorem. 2. If $w(x; y)$ is a harmonic function, i.e., $\Delta w = w_{xx} + w_{yy} = 0$, and has continuous partial derivatives up to the second order in the domain D, then system (1) admits the following solutions in this domain:

$$\begin{cases} u = w_y + C_1 y + C_2 x + C_4; v = -w_x + C_3 x - C_2 y + C_5, \\ P = [F - 0.5(u^2 + v^2) + (C_1 - C_3)[w + 0.5(C_1 y^2 - C_3 x^2) + C_2 xy + C_4 y - C_5 x) + C]\mu \end{cases} \tag{11}$$

where C_k, $k = 1,...,5$ and C are arbitrary constants.

Demonstration T. 2. Suppose that $w(x; y)$ is a harmonic function and has inside the domain D continuous partial derivatives up to and including the second order. And let the functions u and v have the form in (11). Then $u_x + v_y = 0; u_y - v_x = C_1 - C_3$.
From here it follows that these functions verify equations (6) and (7), so they are solutions of system (1). It remains to find out the pressure. In this case we also have, that $\Delta u = 0$, $\Delta v = 0$.
From (5) we obtain:

$$\begin{cases} G_x = (w_x - C_3 x + C_2 y - C_5)(C_1 - C_3) \\ G_y = (w_y + C_1 y + C_2 x + C_4)(C_1 - C_3) \end{cases} \Rightarrow$$
$$G = (C_1 - C_3)[w + 0.5(C_1 y^2 - C_3 x^2) + C_2 xy + C_4 x - C_5 y)] + C.$$

We substitute this result into (4) and obtain the pressure expression from (11). **T. 2.** is demonstrated.

Let's take an example of application of theorem 2.

Example 3. Let $w = ln(x^2 + y^2)$, $C_1 = 2$, $C_3 = 1$, $C_2 = C_4 = C_5 = 0$, then we obtain these solutions:

$$\begin{cases} u = \frac{2y}{x^2+y^2} + 2y; \quad v = x - \frac{2x}{x^2+y^2}, \\ P = [F - \frac{4(2y^2-x^2+1)}{x^2+y^2} + ln(x^2 + y^2) + y^2 - 0{,}5x^2]\mu + C \end{cases} \quad D = OXY\setminus\{O\}. \tag{12}$$

Note. If $C_1 \neq C_3$, then we obtain solutions of the form (11), which cannot be obtained from theorem 1. We mention that in the solutions obtained with the application of theorems 1 and 2 the viscosity parameter λ does not explicitly participate.

4. Solutions, in which the viscosity parameter participates explicitly. Method of separation of variables

Theorem 3. Let the functions $\varphi(x; y)$ and $z(x; y)$ admit in the domain D continuous partial derivatives up to the second order and let $T(\varphi)$ be a function doubly differentiable. Let these functions verify the following equations (13) and (14):

$$\left(\varphi_x^2 + \varphi_y^2\right) \cdot T'' + \Delta\varphi \cdot T' + \Delta z_0 = \varphi \tag{13}$$

$$\varphi_y \cdot z_x - \varphi_x \cdot z_y + \lambda\Delta\varphi = 0 \tag{14}$$

where z_0 is a particular solution of the equation (14).

The solutions for system (1) are determined in the following manner:

First on the determinates the functions u and v out from the following system:

$$\begin{cases} u = \varphi_y \cdot T' + z_{0_y} \\ v = -\varphi_x \cdot T' - z_{0_x} \end{cases} \tag{15}$$

then G from
$$\begin{cases} G_x = \lambda\varphi_y - v \cdot \varphi \\ G_y = -\lambda\varphi_x + u \cdot \varphi \end{cases} \tag{16}$$

and, finally, pressure P from the equality
$$P = \mu[G + F - 0{,}5(u^2 + v^2)]. \tag{17}$$

<u>Demonstration T.3</u> We note $u_y - v_x = \varphi(x; y).$ (18)
Then from (7) and (18) we obtain that $\Delta u = \varphi_y, \; \Delta v = -\varphi_x.$

Replacing in (6), we obtain for φ the following equation:

$$\lambda\Delta\varphi - u\varphi_x - v\varphi_y = 0 \tag{19}$$

In order to make sure that the condition (4) is fulfilled, we introduce the auxiliary function z, which has continuous partial derivatives of the second order in such a way that the equalities are true:

$$u = z_y; \; v = -z_x \tag{20}$$

We substitute expressions (20) in equation (19) and obtain equation (14).

Considering that φ is a given function, we can consider equation (14) in relation to z as a linear equation with partial derivatives of the first order [10]. It is easy to verify that the general solution of this equation has the form

$$z = T(\varphi) + z_0 \tag{21}$$

where $T(\varphi)$ is an arbitrary doubly differentiable function, and z_0 is a particular solution of equation (14). Substituting (21) into equalities (20) and then into (18), we obtain equation (13). Thus, finding the functions u, v and P is reduced to determining the functions T, φ and z from equations (13) and (14). **T. 3.** is demonstrated.

Note. For $\varphi \neq C$ – constant, theorem 3 generates a series of new solutions of the Navier-Stokes equations (1), which differ from those obtained in theorems 1 and 2; in this case at least one of the velocity components u or v will already not be a solution of Laplace's.

Relations (13) and (14) represent two equations with three unknown functions, which allow us to choose one of them in a convenient way for further study, and to determine the other two.

In the following we will study different particular cases, choosing in particular a certain form of the function φ.

Case 1. The function φ is a solution of Laplace's equation, that is $\Delta\varphi = 0$.
In this case, the general solution of equation (14) is $z = T(\varphi)$, $z_0 = 0$, and T is determined from the equation $\left(\varphi_x^2 + \varphi_y^2\right) \cdot T''(\varphi) = \varphi$

This equation can be solved when the expression $\varphi_x^2 + \varphi_y^2$ represents a function that depends on the variable φ or this expression is constant.

We note that in this case only the pressure P depends on the viscosity parameter λ, the functions u and v do not depend on the viscosity.

Example 4. May $\varphi = C \ln(x^2 + y^2)$; $\varphi_x^2 + \varphi_y^2 = \dfrac{4C^2}{x^2+y^2} = \dfrac{4C^2}{e^{\varphi/C}}$; $T''(\varphi) = \dfrac{\varphi \cdot e^{\varphi/C}}{4C^2} \Rightarrow$

$$\Rightarrow T' = \frac{e^{\varphi/C}(\varphi - C)}{4C} + C_1 = \frac{(x^2 + y^2)[\ln(x^2 + y^2) - 1]}{4} + C_1.$$

From (15) we obtain that

$$\begin{cases} u = \varphi_y \cdot T' = Cy \cdot \left[\dfrac{\ln(x^2+y^2)-1}{2} + \dfrac{2C_1}{x^2+y^2}\right], \\ v = -\varphi_x \cdot T' = -Cx \cdot \left[\dfrac{\ln(x^2+y^2)-1}{2} - \dfrac{2C_1}{x^2+y^2}\right]. \end{cases} \tag{22}$$

where $C \neq 0$ and C_1 are arbitrary constants. To determine the pressure according to the formula (17), we first find the function G from the system (16):

$$\begin{cases} G_x = \dfrac{2\lambda Cy}{x^2+y^2} + x \cdot Q; \ \ G_y = -\dfrac{2\lambda Cx}{x^2+y^2} + y \cdot Q; Q = C^2 \ln(x^2 + y^2)\left[\dfrac{\ln(x^2+y^2)-1}{2} + \dfrac{2C_1}{x^2+y^2}\right]. \end{cases}$$
$$\Rightarrow$$
$$\begin{cases} G = -2\lambda C \cdot arctg\left(\dfrac{y}{x}\right) + \dfrac{C^2(x^2+y^2)}{4}\left[\ln^2(x^2 + y^2) - 3\ln(x^2 + y^2) + 3\right] + C_1 \ln(x^2 + y^2) + C_2 \\ P = \mu[G + F - 0.5(u^2 + v^2)] \end{cases}$$

Example 5. $\varphi_x^2 + \varphi_y^2 = C^2 -$ constant. We look for function φ in the following way: .
$\varphi = \alpha(x) + \beta(y) \Rightarrow \varphi_x^2 + \varphi_y^2 = (\alpha')^2 + (\beta')^2 = C^2 \Rightarrow (\alpha')^2 = C^2 - (\beta')^2 = C_1^2$. Then

$$\begin{cases} \alpha'(x) = \pm C_1 \\ \beta'(y) = \pm\sqrt{C^2 - C_1^2} \end{cases} \Rightarrow \begin{cases} \alpha = \pm C_1 x + c_1 \\ \beta = \pm\sqrt{C^2 - C_1^2}\,y + c_2 \end{cases} \Rightarrow \varphi = C_1 x + C_2 y + c, C_1^2 + C_2^2 = C^2.$$

Obviously, $\Delta\varphi = 0$. From $\left(\varphi_x^2 + \varphi_y^2\right) \cdot T'' = \varphi$ we obtain that $T'' = C^{-2}\varphi \Rightarrow T' = \dfrac{\varphi^2}{2C^2} + k$.

For the velocity components we get:
$$\begin{cases} u = \dfrac{C_2\varphi^2}{2C^2} + kC_2 \\ v = -\dfrac{C_1\varphi^2}{2C^2} - kC_1 \end{cases} ; \quad \varphi = C_1x + C_2y + c.$$
(23),

were $C^2 = C_1^2 + C_2^2$; c, C_1, C_2, k – arbitrary constants. From system (16) we find function G:

$$\begin{cases} G_x = \lambda C_2 + \dfrac{C_1\varphi^3}{2C^2} + kC_1\varphi \\ G_y = -\lambda C_1 + \dfrac{C_2\varphi^3}{2C^2} + kC_2\varphi \end{cases} \Rightarrow G = \lambda(C_2x - C_1y) + \dfrac{\varphi^4}{8C^2} + k\dfrac{\varphi^2}{2} + C_3.$$

The Pressure will be equal to $P = \mu[F + \lambda(C_2x - C_1y) + C_4]$, $C_4 = C_3 - \dfrac{(kC)^2}{2}$.
(24)

Case 2. $\varphi = f(y) + g(x)$, where functions f and g are doubly differentiable. Then equation (14) has the following form:

$$f'(y)z_x - g'(x)z_y + \lambda[f''(y) + g''(x)] = 0 \Rightarrow \dfrac{z_x}{g'} - \dfrac{z_y}{f'} + \lambda\left(\dfrac{f''}{f' \cdot g'} + \dfrac{g''}{g' \cdot f'}\right) = 0. \quad (25)$$

We determine functions $f(y)$ and $g(x)$ in the following way: $\begin{cases} f''(y) = C_1f'(y) \\ g''(x) = C_2g'(x) \end{cases} \Rightarrow$

$$\Rightarrow \begin{cases} f'(y) = C_1f(y) + \alpha_1 \\ g'(x) = C_2g(x) + \alpha_2 \end{cases} \Rightarrow \begin{cases} f = k_1e^{C_1y} - \dfrac{\alpha_1}{C_1} \\ g = k_2e^{C_2x} - \dfrac{\alpha_2}{C_2} \end{cases} \Rightarrow \varphi = k_1e^{C_1y} + k_2e^{C_2x} + \alpha \quad (26),$$

where $k_1, k_2, \alpha, C_1, C_2$ are arbitrary constants.

A particular solution of equation (25) is $z_0 = \lambda(C_2y - C_1x)$.

Replacing function z_0 and φ from (26) in (13) we obtain the equation for determining function T:

$$(C_1^2k_1^2e^{2C_1y} + C_2^2k_2^2e^{2C_2x})T'' + (C_1^2k_1e^{C_1y} + C_2^2k_2e^{C_2x})T' = k_1e^{C_1y} + k_2e^{C_2x} + \alpha. \quad (27)$$

Equation (27) can take place when T is linear or one of the functions f or g is constant. May it be

$T = C\varphi \Rightarrow T' = C \Rightarrow T'' = 0.$ Substituting in (27), we get first

$$(C_1^2k_1e^{C_1y} + C_2^2k_2e^{C_2x})C = k_1e^{C_1y} + k_2e^{C_2x} + \alpha \Rightarrow \alpha = 0, \ C_1^2 = C_2^2 = C^{-1}.$$

Replacing in (15) on obtain following solutions of system (1):
$$\begin{cases} u = k_1C_1^{-1}e^{C_1y} + \lambda C_2 \\ v = -k_2C_2^{-1}e^{C_2x} + \lambda C_1 \end{cases}; \quad C_1^2 = C_2^2.$$
(28)

Then we determine the function G from the system (16):
$$\begin{cases} G_x = \lambda\varphi_y - v \cdot \varphi = \lambda k_1C_1e^{C_1y} + (k_2C_2^{-1}e^{C_2x} - \lambda C_1)(k_1e^{C_1y} + k_2e^{C_2x}) \\ G_y = -\lambda\varphi_x + u \cdot \varphi = -\lambda k_2C_2e^{C_2x} + (k_1C_1^{-1}e^{C_1y} + \lambda C_2)(k_1e^{C_1y} + k_2e^{C_2x}) \end{cases} \Rightarrow$$

$$\Rightarrow G = \dfrac{C(k_1e^{C_1y} + k_2e^{C_2x})^2}{2} + \lambda\left(\dfrac{k_1C_2e^{C_1y}}{C_1} - \dfrac{k_2C_1e^{C_2x}}{C_2}\right), C_2 = \pm C_1.$$

Then we find the pressure:

$$P = \mu[F + \lambda k_1 k_2 e^{C_1 y + C_2 x} + C_0].$$ (29)

Let now that $f(y) = m$ - constant. Then $\varphi = m + g(x)$ and from (19) we obtain that:

$$-g'(x)z_y + \lambda g''(x) = 0 \quad \Rightarrow \quad u = z_y = \frac{\lambda g''(x)}{g'(x)}; \; u_y = 0.$$

From (7) we have that

$$u_x + v_y = 0 \Rightarrow v = -u_x \cdot y + \beta(x).$$

From (18) it follows that $u_y - v_x = \varphi \Rightarrow v_x = -\varphi \Rightarrow -u_{xx} \cdot y + \beta'(x) = -m - g(x).$

The last equality can only occur if $u_{xx} = 0, \; \beta'(x) = -m - g(x).$ From this results that

$$(\frac{g''(x)}{g'(x)})'' = 0 \Rightarrow \frac{g''(x)}{g'(x)} = ax + b \Rightarrow g(x) = c \int e^{\left(\frac{ax^2}{2} + bx\right)} dx; \; \beta(x) = -mx - \int g(x)dx.$$

In this way we obtain the following solutions of the velocity components system (1):

$$u = \lambda(ax + b); \; v = -\lambda ay - mx - \int g(x)dx; \; g(x) = \int c\, e^{\left(\frac{ax^2}{2} + bx\right)} dx.$$ (30)

where a, b, m, c are arbitrary constants. The presure in this caze is:

$$P = \mu[F - 0{,}5a\lambda^2(x^2 + ay^2 + bx) - \lambda cy + C_0].$$ (31)

If in (30) we take $a = 0$, then $\varphi = \frac{c}{b}e^{bx} + m$ we obtain the following solutions of system

(1): $u = \lambda b; \; v = c_1 - mx - cb^{-2}e^{bx}; \; P = \mu[F - \lambda(bm + c)y + C_0].$ (32)

If $a = 0$ and $b = 0$, then $g(x) = c_1 + cx$ and we obtain the solutions:

$$u = 0, \; v = -\frac{c}{2\lambda}x^2 + c_1 x + c_2, \; P = \mu[F - \lambda cy + c_0].$$ (33)

Le t $g(x) = m$ - constant. Then $\varphi = m + f(y)$ and we obtain:

$$v = \lambda(ay + b); \; u = \lambda ax + my + \int f(y)dy; \; f(y) = \int c\, e^{\left(\frac{ay^2}{2} + by\right)} dy.$$ (34)

Specifically, if in (33) we take $a = 0$, then $\varphi = \frac{c}{b}e^{by} + m.$ And we obtain solutions:

$$v = \lambda b; \; u = c_1 - my - \frac{c}{b^2}e^{by}; \; P = [F - \lambda(bm + c)x + C_0].$$ (35)

If $a = 0, b = 0$, then $f(y) = cy + c_1$ and we obtain the following solutions:

$$v = 0; \; u = -\frac{c}{2\lambda}y^2 + c_1 y + c_2; \; P = \mu(F - \lambda cx + c_0).$$ (36)

Case 3. $\varphi = f(y) \bullet g(x)$, where functions f and g are doubly differentiable.

In this case equation (14) is: $f'(y)g(x)z_x - g'(x)f(y)z_y + \lambda[g(x)f''(y) + f(y)g''(x)] = 0 \Rightarrow$

$$\frac{gz_x}{g'} - \frac{fz_y}{f'} + \lambda\left(\frac{gf''}{f' \cdot g'} + \frac{fg''}{g' \cdot f'}\right) = 0$$ (37).

We determine f and g in the following way: $\begin{cases} f'(y) = C_1 f(y) \\ g'(x) = C_2 g(x) \end{cases} \Rightarrow \begin{cases} f'' = C_1 f' \\ g'' = C_2 g' \end{cases} \Rightarrow$

$$\begin{cases} f = \alpha_1 e^{C_1 y} \\ g = \alpha_2 e^{C_2 x} \end{cases} \Rightarrow \varphi = \alpha e^{C_1 y + C_2 x}, \alpha = \alpha_1 \alpha_2$$ (38)

Then from (34) we obtain:

$$\frac{z_x}{C_2} - \frac{z_y}{C_1} + \lambda\left(\frac{C_1}{C_2} + \frac{C_2}{C_1}\right) = 0 \Rightarrow C_1 z_x - C_2 z_y = -\lambda(C_1^2 + C_2^2) \tag{39}$$

The general solution of equation (38) is $z = T(\varphi) + z_0$, where $z_0 = \lambda(C_2 y - C_1 x)$.

Substituting (38) in (13), we obtain the equation for $T(\varphi)$:

$$[C_1^2 \alpha^2 e^{2(C_1 y + C_2 x)} + C_2^2 \alpha^2 e^{2(C_2 x + C_1 y)}]T'' + [\alpha(C_1^2 + C_2^2)e^{C_2 x + C_1 y}]T' = \alpha e^{C_1 y + C_2 x}.$$

From here

$$\alpha e^{(C_2 x + C_1 y)} \cdot T'' + T' = \frac{1}{(C_1^2 + C_2^2)} \tag{40}$$

Or

$$\varphi \cdot w' + w = m; \quad m = \frac{1}{(C_1^2 + C_2^2)}, \quad w(\varphi) = T'(\varphi). \tag{41}$$

Equation (40) is a linear ordinary differential equation of order 1 with the unknown function w. The general solution ([11], [12]) of the given equation is: $w = m + \frac{c}{\varphi} = F'$. In this case we obtain the following solutions of system (1):

$$\begin{cases} u = m_1 e^{C_1 y + C_2 x} + cC_1 + \lambda C_2 \\ v = -m_2 e^{C_1 y + C_2 x} - cC_2 + \lambda C_1 \end{cases}; m_n = \frac{\alpha C_n}{C_1^2 + C_2^2}, n = 1, 2. \tag{42}$$

$$G = 0.5\alpha^2 m e^{2(C_1 y + C_2 x)} + \alpha c e^{(C_1 y + C_2 x)} + C_0; \quad P = \mu[F + (\lambda^2 + c^2)(C_1^2 + C_2^2)].$$

Where α, c, C_1, C_2 are arbitrary constants.

Note. In the case of the current given by (41), in the absence of external force, the pressure is constant. Solutions (41) and (23) represent those cases, when there is a linear dependence between the velocity components. Thus, in the case of these solutions we have correspondingly, that

$$v = -\frac{C_2}{C_1}u + \lambda C_1 + \frac{\lambda C_2^2}{C_1}; \quad v = -\frac{C_1}{C_2}u.$$

Case 4. $\varphi = f(t) = f(kx + by)$, where function $f(t)$ is doubly differentiable.
In this case equation (14) has the following form:

$$bz_x - kz_y + \lambda(b^2 + k^2)\frac{f''}{f'} = 0 \tag{43}$$

We will examine the case $\frac{f''}{f'} = C - constant, \ C \neq 0$. Then

$$f'(t) = C_1 e^{Ct} \Rightarrow \varphi = f(t) = \left(\frac{C_1}{C}\right)e^{Ct} + C_2; \ t = kx + by. \tag{44}$$

The general solution for equation (42) will be $z = T(t) + z_0$, where $z_0 = \lambda C(ky - bx)$,
From (6) we obtain the equation for determining the function T:

$$f'T'' + CT' = \frac{f}{f'(k^2 + b^2)}, f' = C(f - C_2). \tag{45}$$

Or

$$(f - C_2) \cdot w' + w = \frac{f}{m(f - C_2)}; \quad m = C^2(k^2 + b^2), \ w(f) = T'(f). \tag{46}$$

We obtained a linear ordinary differential equation of order 1 with the unknown function w.

The general solution for equation (46) is $w = \frac{1}{m(f - C_2)}[f + C_2\ln(f - C_2) + C_0]$.

Hence, considering relations (44) and (45), we obtain the following equality:

$$T' = \frac{e^{-Ct}}{mC_1}[C_1 e^{Ct} + C_2 C^2 t + C_3], C_3 = C_0 + CC_2 \ln\left(\frac{C_1}{C}\right). \tag{47}$$

In this case for the velocity components we have the following solutions:

$$\begin{cases} u = \varphi_y \cdot T' + z_{0_y} = \frac{b}{m}[C_1 e^{Ct} + C_2 C^2 t + C_3] + \lambda k C \\ v = -\varphi_x \cdot T' - z_{0_x} = -\frac{k}{m}[C_1 e^{Ct} + C_2 C^2 t + C_3] + \lambda b C \end{cases} ; \ t = kx + by.$$

(48),

where $m = C^2(k^2 + b^2)$; k, b, C, C_1, C_2, C_3 are arbitrary constants.

Note. If in (48) we take $C_2 = 0$, then we will obtain solutions of the form (42).

Case 5. $\Delta\varphi = C_1\varphi_x + C_2\varphi_y$, with C_1, C_2 − constants.
In this case equation (14) has the following form:

$$\frac{z_x}{\varphi_x} - \frac{z_y}{\varphi_y} + \lambda\left(\frac{C_1}{\varphi_y} + \frac{C_2}{\varphi_x}\right) = 0.$$

(49)

The general solution of this equation will be $z = T(f) + z_0$, where

$$z_0 = \lambda(C_1 y - C_2 x).$$

We will determine the function φ, applying the method of separation of variables ([12], [13]).

If $\varphi = a(x) + b(y)$, then we will obtain solutions of the form (S$_{33}$) − (S$_{35}$) examinate of case 2.

Let $\varphi = a(x)b(y)$. Then from $\Delta\varphi = C_1\varphi_x + C_2\varphi_y \Rightarrow \frac{a''}{a} + \frac{b''}{b} = C_1\frac{a'}{a} + C_2\frac{b'}{b} \Rightarrow$ $\frac{a'' - C_1 a'}{a} = \frac{-b'' + C_2 b'}{b} = C$, where C is an arbitrary constant.

From here, to determine the functions $a(x)$ and $b(y)$ we obtain the following system of ordinary differential equations:

$$\begin{cases} a'' - C_1 a' - Ca = 0, \\ b'' - C_2 b' + Cb = 0. \end{cases}$$

(50)

The general solution of the first equation in (50), depending on the value of $k_1 = C_1^2 + 4C$, will be:

$$1) \ k_1 > 0; \ a(x) = a_1 e^{\alpha_1 x} + a_2 e^{\alpha_2 x}, \quad \alpha_{1,2} = 0{,}5(C_1 \pm \sqrt{k_1});$$
$$2) \ k_1 < 0; \ a(x) = e^{0{,}5 C_1 x}(a_1 \cos(\sqrt{-k_1}x) + a_2 \sin(\sqrt{-k_1}x));$$
$$3) \ k_1 = 0; \ a(x) = e^{0{,}5 C_1 x}(a_1 + xa_2).$$

For the second equation in (50) the form of the general solution will be the same, but depending on the value of $k_2 = C_2^2 - 4C$, replacing in 1), 2), 3) x with y, $\alpha_{1,2}$ with $\beta_{1,2}$ and the arbitrary constants a_1, a_2, with the corresponding arbitrary constants b_1, b_2 ([10], [11]).

From (13) we deduce the equation for determining function T:

$$[(a'b)^2 + \cdot (b'a)^2]F'' + \cdot [a''b + b''a]F' = ab.$$

(51)

We will first consider that $T' = l$ - constant. Then $T'' = 0$ and from (51) we obtain that

$$[\frac{a''}{a} + \frac{b''}{b}]l = 1.$$

(52)

Equality (52) can only occur if both fractions in the square bracket have constant values. We will examine these possibilities.

$\boldsymbol{a)} -C_1^2 < 4C < C_2^2$. Then $k_1 > 0$ şi $k_2 > 0$ and we take $a(x) = a_n e^{\alpha_n x}, b(y) = b_m e^{\beta_m y}$; $n, m = 1, 2,$ where $\alpha_{1,2} = 0{,}5(C_1 \pm \sqrt{k_1})$, $\beta_{1,2} = 0{,}5(C_2 \pm \sqrt{k_2})$,

Then $a'' = \alpha_n^2 a$, $b'' = \beta_m^2 b$ și $l = 1/(\alpha_n^2 + \beta_m^2)$. In this case we obtain the following solutions of system (1):

$$\begin{cases} u = a_n b_m \beta_m e^{\alpha_n x} e^{\beta_m y} \cdot l + \lambda C_1, \quad l = 1/(\alpha_n^2 + \beta_m^2) \\ v = -a_n b_m \alpha_n e^{\alpha_n x} e^{\beta_m y} \cdot l + \lambda C_2 \qquad n, m = 1, 2 \end{cases} \quad (53)$$

In this case the pressure will be $P = \mu[F + \lambda^2(C_1^2 + C_2^2) + C_0]$.

b) $C_2 = 0, C_2^2 < 4C$. Then $k_1 > 0, k_2 < 0$ and $a(x) = a_n e^{\alpha_n x}, \alpha_{1,2} = 0{,}5(C_1 \pm \sqrt{k_1})$,
$b(y) = b_1 \cos\sqrt{C}y + b_2 \sin\sqrt{C}y$; a_n, b_m are arbitrary constants; $n, m = 1, 2$.

Then $a'' = \alpha_n^2 a$, $b'' = -Cb$ și $l = 1/(\alpha_n^2 - C)$. It is easy to verify that if the constants that if the constants C and C_1 are different from zero, then also $\alpha_n^2 - C \ne 0$. In this case we obtain the following solutions:

$$\begin{cases} u = a_n \sqrt{C} e^{\alpha_n x} (b_2 \cos\sqrt{C}y - b_1 \sin\sqrt{C}y)l + \lambda C_1, \quad l = 1/(\alpha_n^2 - C) \\ v = -a_n \alpha_n e^{\alpha_n x} (b_1 \cos\sqrt{C}y + b_2 \sin\sqrt{C}y)l \qquad n = 1, 2 \end{cases} \quad (54)$$

c) $C_1 = 0$ și $4C < -C_1^2$. Then $k_1 < 0$, $k_2 > 0$ and $a(x) = a_1 \cos\sqrt{-C}x + a_2 \sin\sqrt{-C}x$,
$b(y) = b_m e^{\beta_m y}$; $\beta_{1,2} = 0{,}5(C_2 \pm \sqrt{k_2})$; a_n, b_m − arbitrary const.; $n, m = 1, 2$

Then $a'' = Ca$, $b'' = \beta_m^2 b$ and $l = 1/(C + \beta_m^2)$. It is easy to verify that if C and C_2 are different from zero then also $C + \beta_m^2 \ne 0$. In this case we obtain the following solutions:

$$\begin{cases} u = b_m \beta_m e^{\beta_m y} (a_1 \cos\sqrt{-C}x + a_2 \sin\sqrt{-C}x)l, \quad l = 1/(C + \beta_m^2) \\ v = -b_m \sqrt{-C} e^{\beta_m y} (a_2 \cos\sqrt{-C}y - a_1 \sin\sqrt{C}y)l + \lambda C_2; \qquad n = 1, 2 \end{cases} \quad (55)$$

In case the solutions (54) and (55) on determine the function G with the (16) and the pressure P with the formula (17).

Note. The case $k_1 < 0$ and $k_2 < 0$ is impossible because it only takes place if $C_2^2 < 4C < -C_1^2$.
In the case $k_1 = 0$ and $k_2 = 0$ we obtain solutions (53) with $a_n = 0{,}5C_1$, $b_m = 0{,}5C_2$.
If $\alpha_2^2 = \alpha_1^2$ and $\beta_2^2 = \beta_1^2$ then $C_1 = C_2 = 0$. But in this case we have $a'' = Ca, b'' = -Cb$ and the expression from the square brackets of the equation (52) is equal to zero, that is (52) becomes a false identity.

We now return to equation (51) in the case where T' is not constant. In this case we proceed in the same way as in case a) above and take $a(x) = a_n e^{\alpha_n x}, b(y) = b_m e^{\beta_m y}$. We have the following equation for the determination of the function T:

$$a_n b_m e^{\alpha_n x + \beta_m y} \cdot T'' + T' = \frac{1}{(\alpha_n^2 + \beta_m^2)}.$$

This equation has the form of equation (40) studied previously. Solving it in the same way as in the case of equation (40) we will obtain solutions of the form (42), replacing C_1 with α_n, C_2 with β_m and $\alpha = a_n b_m$.

5. Results and Discussion

The present paper contains three proven theorems, as well as various examples and particular examined cases. Applying Theorem 1, we can find various solutions, where the velocity components represent the imaginary and real parts of a differentiable function of a complex variable. Theorem 2 allows us to determine solutions, where the velocity components are expressed by the partial derivatives of the solutions of Laplace's equation of a special form. It is to be mentioned that these theorems give us solutions that do not depend on the viscosity parameter λ.

In the present work, an original method elaborated by the author is presented, with the help can be determined of which solutions of the system (1), in which the viscosity parameter participates explicitly. This method is presented in theorem 3 for obtaining a series of solutions of the Navier-Stokes equations in which the viscosity coefficient λ participates explicitly. The application of this method to obtain different solutions of system (1) is presented in cases 1-5 following the proof of theorem 3.

In the current article a lot of solutions of the Navier-Stokes equations are determined by the system (1), both of general form - solutions (2), (11), as well as the exact solutions - (9), (10), (12), (22), (23), (24), (28), (29), (30)-(36), (42), (48), (52)-(55). Starting with the solution (28) onwards, in all the obtained solutions, their dependence on the viscosity parameter λ is explicitly indicated. We would also like to mention that the main results presented in this paper were reported and discussed in the following international conferences: the symposium UTM 2020, conference MITRE 2021 and CAIM 2022. The obtained results were presented in the following theses of these conferences: [14], [15] and [16].

6. Conclusions

In this paper a lot of solutions of the Navier-Stokes equations are determined both of general form as well as the exact solutions. Different constants participate in the expressions of the obtained solutions, the values of which can be determined based on the initial conditions and the boundary conditions of the examined physical problems.

For example, the solutions (33) or (36) are solutions of the plane flow problem type Poiseuille or Couette ([17]). Thus, for the plane flow of Poiseuille type, we have that for the channel section of diameter *2h*, a component of the velocity is equal to zero, for example $v = 0$, and the boundary conditions are of the form $u(h) = u(-h) = 0$. From here we find that $c_1 = 0$, $c_2 = - (h^2 c)/2\lambda$, and $c > 0$, since the pressure inside the channel decreases. For the Couette type flow we also have $v = 0$ and the following boundary conditions: $u(h) = u_0$, $u(-h) = 0$. Then $c_1 = u_0/2h$, $c_2 = (h^2 c)/2\lambda + u_0/2$ ([17]).

In the research carried out here, the rotor (vortex) of the examined flow is of special importance, namely the expression $\varphi = u_y - v_x$. Thus, if we can experimentally or theoretically determine the rotor of the flow (is it null, or constant, or has a particular shape), then we can apply the results of theorems 1, 2, or 3, demonstrated above, to determine the components of the flow velocity and the pressure created.

Conflicts of Interest: The authors declare no conflict of interest.

References
1. Cernica, I. *Fluid mechanics*. Matrixrom, Bucureşti, 2011, 425 p. [in Romanian].
2. Mazo, A.; Potasev, K. *Hydrodynamics*. CGU, 2013, 128 p. [in Russian].
2. Loitseanschii, L. *Mechanics of fluids and gases*. Nauka, Moscow, 2003, 840 p. [in Russian].
4. Wang, C. Exact solution of the unsteady Navier-Stokes equations. *Appl. Mech. Rev.* 1989, 42, pp. 269-282.
5. Wang, C. Exact solution of the steady-state Navier-Stokes equations. *Annu. Rev. Fluid Mech. Rev.* 1991, 23, pp. 159-177.
6. Polyanin, A.; Zaitsev, V. Exact solutions of the Navier-Stokes equations with generalized separation of variables. *Dokladi RAN* 2001, 380, pp. 491-496. [in Russian]
7. Koptev, A. First integral and ways of further integration of the Navier-Stokes equations. *Izvestia RGU Hertsen, N.* 2012, 147, p. 7-17. [in Russian].

8. Polyanin, A.; Zaitsev, V. *Handbook of nonlinear partial differential equations*. Boca Raton, CRC Press, 2004, 840 p.

9. Enescu, I.; Sava, V. *Special mathematics*. Institutul Politehnic, Iasi, 1981, 391 p. [in Romanian].

10. Stepanov, V. *Differential equations course*. Ed. Lumina, Chisinau, 1970, 488 p. [in Romanian].

11. Kamke, E. *Handbook of Ordinary Differential Equations*. Nauka, Moscow, 1976, 576 p. [in Russian].

12. Aniculăesei, Gh. *Differential and partial differential equations*. Universităţii AIC, Iaşi, 2003, 375 p. [in Romanian].

13. Perjan, A. *Partial differential equations*. USM, Chisinau, 2015, 225 p. [in Romanian].

14. Baltag, Iu. On some determination solutions of the stationary bidimensional Navier-Stokes equations. In *International Conference SIPAMI 2020*, Chisinau, 27 – 28 november 2020, p. 46.

15. Baltag, Iu. Determination on some solutions of the stationary 2D Navier-Stokes equations. In *International Conference MITRE 2021*, Chisinau, 01 – 03 Jule 2021, p. 14.

16. Baltag, Iu. Determination of some solutions of the stationary Navier-Stokes equations. In *International Conference CAIM 2021*, Romania*,* 17 – 18 september 2021, p. 12.

17. Vedeneev, V. *Mathematical theory of stability of plane-parallel fluid flows*. ID Intelect, Moscow*,* 2016, 152 p. [in Russian].

Submission of manuscripts: jes@meridian.utm.md

OPEN ACCESS

DESCRIPTION OF THE STANDARDIZATION PROCEDURE OF STANDARD VOLUME MEASURES AND ANALYSIS OF THE STABILITY OF THEIR METROLOGICAL CHARACTERISTICS

Anna Sabadas [1, 2*], ORCID: 0000-0002-8064-6068,
Alexei Pianih [1, 2], ORCID: 0000-0002-1165-9690

[1]*Stefan cel Mare University of Suceava, 13 University Street, 720229 Suceava, Romania*
[2]*Technical University of Moldova, 168 Stefan cel Mare Blvd., Chisinau, Republic of Moldova*
*Corresponding author: Anna Sabadas, any.sabadas@gmail.com

Abstract. The metrological characteristics of the measuring devices refer to the behavior of the measuring device in relation to the object under measurement, the environment and the human operator. Thus, having the same conditions in the laboratory and a well-trained and competent staff, the stability of the metrological characteristics of the standard volume measures were studied in order to demonstrate their stability over time and provide increased confidence to the subsequent beneficiaries. The measurements were carried out over a period of one year under the same conditions, their results demonstrating that the standard volume measures are able to ensure the traceability of the measurement unit. In this way to ensure the uniformity, accuracy and legality of the measurements performed in different fields - measurement of volume in commercial transactions, fuel supply, wine industry.

Keywords: *standardization, standard measures, volume, metrological characteristics, stability.*

Rezumat. Caracteristicile metrologice ale mijloacelor de măsurare se referă la modul de comportare a aparatului de măsurat în raport cu obiectul supus măsurării, cu mediul ambiant şi cu operatorul uman. Astfel, având aceleaşi condiţii în laborator şi un personal bine instruit şi competent, se studiază stabilitatea caracteristicilor metrologice ale măsurilor de volum etalon pentru a demonstra stabilitatea acestora în timp şi a oferi o încredere sporită beneficiarilor ulteriori. Măsurările propriu-zise au fost efectuate pe perioada unui an în aceleaşi condiţii, rezultatele acestora demonstrând că măsurile de volum etalon sunt capabile să asigure trasabilitatea unităţii de măsură. În acest fel, să se asigure uniformitatea, exactitatea şi legalitatea măsurărilor efectuate în diferite domenii de măsurare la efectuarea măsurărilor volumului cu referire la tranzacţiile comerciale, alimentarea cu combustibili, industria vinicolă.

Cuvinte cheie: *etalonare, măsuri etalon, volum, caracteristici metrologice, stabilitate.*

1. Introduction

The measurement unit for volume is the cubic decimeter (dm^3) or the liter (L), which is defined as the volume occupied by 1 kg of distilled water at 4°C in vacuum at sea level and 4° latitude. A thousandth of a liter is a cubic centimeter (cm^3) or a milliliter (mL) [1].

The purpose of the article is to determine the metrological characteristics of standard volume measures and demonstrate the stability of the measurements over time. Being part of the national standard of the liquid volume unit, their study is very important. The volumetric method and the gravimetric method [2] can be used when calibrating standard volume measures, the latter providing a higher level of accuracy.

The national standard of the liquid volume measurement unit (ETN V) constitutes a basic element of the National System of Standards [4].

ETN V traceability [5] is ensured by the gravimetric method from ETN 09 -15 National Standard of mass. Subsequently, ETN transmits the volume measurement unit to other working standards and measuring instruments in the field according to the traceability scheme, in order to ensure the uniformity, accuracy and legality of the measurements performed throughout the territory of the Republic of Moldova. This is important to ensure correct measurements in different fields: measurement of volume in commercial transactions, fuel supply, wine industry.

2. Materials and Methods

The volume standard measures [6] are a component of the national standard and have the role of volume transfer standard. The standards allow the measurement of the volume of the liquid with an uncertainty of (0.0005 ÷ 0.04) dm³, the measurement accuracy being dependent on the parameters of the environment. To reproduce the volume measurement unit, standard volume measures of 10 dm^3, 50 dm^3, 100 dm^3 are used. Each standard volume measure has a nominal value with an accuracy of 0.02%.

Metal volume measures of the first-order [1] are used to convey the unit of volume to metal volume measures of the second-order and technical volume measures (figure 1). The error of the first-order metal volume measurement at a temperature of 20.0 °C must not exceed 0.02% of the nominal value. First-order metal volume measures are of several types: M1p2; M1p5; M1p10; M1p20; M1p50; M1p100; M1p200; M1p500. In the standardization process, volume measures up to 200 dm^3 are used.

Figure 1. Classification of first-order metal volume measures.
(*done by authors*).

A volume measure (figure 2) consists of:
1. Adjustable legs;
2. Tap;
3. Casing;
4. Sight glass;
5. Maximum level.

The standardization of the volume measures is carried out in the following environmental conditions [3]:

- Ambient temperature: (20.0 ±5.0) °C.
- Air humidity: (30.0 ÷ 80.0) %.
- Atmospheric pressure: (840.0 ÷ 1060.0) hPa.
- The water temperature during standardization must not exceed ± 0.2 °C.
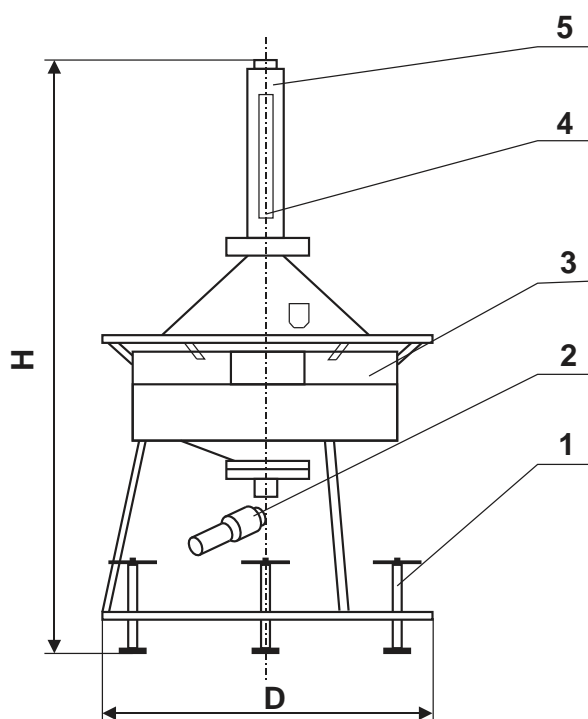
Figure 2. First-order volume measure [1]:
1. adjustable legs; 2. tap; 3. casing; 4. sight glass; 5. maximum level.

In case of non-compliance with the previously indicated environmental conditions, the measurements will be stopped and repeated only after removing the non-conformities.

The standardization operation of the first-order metal volume measure is carried out using measuring means and auxiliary devices, shown in table 1.

Table 1

List of standards, measuring instruments and auxiliary equipment [3].

No.	Name	Type	Technical characteristics
1.	Mass comparator	HRP 200.4 Y.KO	Measurement range: (0.0 ÷200000.0) g
2.	Weighing device with non-automatic operation	APP 25.3Y	Measurement range: (0.0 ÷ 25.0) kg
3.	Digital thermometer with platinum Pt100 resistance	TPM1	Temperature Measurement range: (-25.0 ÷ +250.0) °C

4.	Mechanical chronometer	-	Measurement range: (0.0 ÷ 1800.0) s
5.	Digital thermohygrometer	ПИ-002 1/A	Temperature Measurement range: (5.0 ÷ 98.0) °C Humidity Measurement range: (5.0 ÷ 40.0) %
6.	Pressure, humidity, temperature indicator	PTU 303	Temperature Measurement range: (-20.0 ÷ 80.0) °C Humidity Measurement range: (0.0 ÷ 100.0) % Absolute pressure Measurement range: (500.0 ÷ 1100.0) hPa
7.	Micropipette	-	Measurement range: (2.0 ÷ 10.0) mL

The calibration procedure takes place in several stages:

1. Checking the exterior. Upon receipt of the MM, they undergo a visual examination and the compliance with the following requirements is established:
- Lack of mechanical faults and traces of corrosion;
- The interior of volume measure must be degreased and not contain elements that do not belong to the measure;
- The presence of the identification plate: type, number, nominal value;
- The presence of air bubbles.

2. Preparatory works for calibration. Before carrying out the standardization of the volume measures to be standardized, the following preliminary works must be carried out:
- The volume measure to be standardized needs to be adapted to the environmental conditions in the laboratory and must be placed in the room at least 3 hours before the start of the measurement;
- Check if the environmental conditions meet the requirements specified above;
- Wet the walls, measure the volume to be standardized;
- Check the flatness of the volume measures to be standardized;
- Check the tightness of the standardized volume measures. The volume measure to be standardized is considered watertight if it does not leak water for 3 minutes. If the above condition is not met, the standardization is stopped.
- After the entire volume has drained from the volume measure to be standardized, the tap is kept open for 30 seconds;
- Wet the walls of the transfer vessel;
- The tightness of the transfer vessel is checked. The transfer vessel is considered watertight if there are no water leaks for 3 minutes. If the above condition is not met, the stadardization is stopped.
- After emptying the transfer vessel, it remains face down for 30 seconds.
- Calibration of mass comparator and weighing device with non-automatic operation.

3. Description of the method used. The calibration of the volume measures is carried out by the gravimetric method, according to documents [2]. The gravimetric method consists in the indirect determination of the volume of the working liquid from the volume measure to be standardized, from the mass obtained by weighing the full volume measure and after emptying and converting it into volume at the reference temperature t_o =20.0 °C.

Measurements are performed by two methods: a) direct measurement, b) measurement through the transfer vessel.

Tap water is used for the measurements.

a)   Direct measurement. The volume measure to be standardized is filled with water up to the nominal value. Insert the temperature transducer into the volume measure to be standardized and record the water temperature indications when they are stable and do not vary. The volume measure to be standardized is placed on the plate of the mass comparator, recording the indicated values (I_L). The water in the volume to be standardized is removed and after the entire volume has drained, the tap is kept open for 30 seconds. Then the valve is closed and the indicated values (I_E) are recorded. A minimum of 3 measurements are performed for each volume measurement.

After weighing the empty and full transfer vessel, the total mass is calculated according to the formula:

$$m_{tot} = (I_L) - (I_E), \tag{1}$$

where: I_L − the result of weighing the full vessel.

I_E − the result of weighing the empty vessel.

If direct measurement is not possible, the transfer vessel is used.

b)   Measurement through the transfer vessel. The volume measure to be standardized is filled with water up to the nominal value. Insert the temperature transducer into the volume measure to be standardized and record the water temperature indications when they are stable and do not vary. The moistened transfer vessel is placed on the plate of the mass comparator, and the indicated values (I_E) are recorded. The water in the standardized volume measure is removed in the transfer vessel. The full transfer vessel is placed on the plate of the mass comparator, and the (I_L) values are recorded. A minimum of 3 measurements are performed for each volume measurement.

3. Results and Discussion

The uniformity and accuracy of the measurements in the Republic of Moldova are ensured by a complex of technical-organizational measures, which also includes the activity of transmitting the units of measurement from the national standards to the other standards and from them onwards, to the means of work measurement [7].

In the „Flows and Volumes" laboratory, which operates on the basis of SM EN ISO/IEC 17025 [8], measurements were made with standard volume measures in order to analyze and research the stability of their metrological characteristics. Thus, the measurement data [6] obtained on different days, but under the same conditions, will be presented below.

„The definitions given in the third edition of the International vocabulary of metrology − Basic and general concepts and associated terms (VIM) [9] for the two concepts on which this paper is focused are:

− **measurement uncertainty** non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used

and

− **metrological traceability** property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty" [10].

The stability of the metrological characteristics of the volume measures were determined based on the concordance factor En according to SR EN ISO/CEI 17043:2010 [11], between the value obtained from the last standardization and the value obtained during the measurements is calculated according to the formula:

$$E_n = \frac{x_{lab} - x_{ref}}{\sqrt{U_{lab}^2 + U_{ref}^2}}, \qquad\qquad (2)$$

where : x_{lab} - the result reported by the participating laboratory;

x_{ref} - the reference value (obtained from reference laboratory determinations);

U_{lab} - the uncertainty estimated by the participating laboratory;

U_{ref} - the uncertainty associated with the reference value (estimated by the reference laboratory).

Thus for:

a) $|E_n| \leqslant 1.0$ shows „satisfactory" performance and does not generate any signal;

b) $|E_n| > 1.0$ shows „unsatisfactory" performance and generates action signals.

The measurement uncertainty was calculated based on the SM ISO/IEC Guid 98-3:2017 Uncertainty of measurement. Part 3: Guide to the expression of uncertainty of measurements [12].

Table 2

The values received as a result of the measurements performed for volume standard measurement, type, M1P-10 ГР-01, M1P-50 ГР-01, M1P-100 ГР-01

Standard volume measure type	Date of measurement	Nominal value, dm³	Conventional volume, dm³		Measurement uncertainty, dm³		En
			X_m	X_{CE}	U_m	U_{CE}	
M1P-10 ГР-01	06.08.2018	10	10.013	10.013	0.00040	0.00019	0.74
	10.10.2018		10.013	10.013	0.00044	0.00019	0.88
M1P-50 ГР-01	08.08.2018	50	50.076	50.077	0.00117	0.00124	0.29
	08.10.2018		50.076	50.077	0.00099	0.00124	0.54
M1P-100 ГР-01	13.08.2018	100	100.126	100.128	0.00142	0.00196	0.65
	17.10.2018		100.126	100.128	0.00142	0.00196	0.65

The standard volume measures whose metrological characteristics have been analyzed are a component part of the national standard [13] of the unit of measurement of the volume of liquids, thus the results obtained confirm the arrangement in the means of measurement held by the National Institute of Metrology and ensure the traceability of the unit of volume measurement both nationally [14] and internationally [15].

Analyzing the data from the tables above, taking into account all the measurement, we can conclude that En factor is less than 1, what means that the standard [11] criteria is met.

4. Conclusions

Standard volume measures, as a component part of the national standard of the liquid volume measurement unit, contribute directly to ensuring the reproduction, preservation and transmission of the volume measurement unit, through the calibration process of standards and measuring instruments, in order to ensure uniformity, the accuracy and traceability of the measurements performed throughout the territory of the Republic of Moldova.

Analyzing the results of the measurements performed, it is observed that the concordance factor En between the value obtained from the last standardization and the value obtained during the measurements for the nominal value (10 dm³, 50 dm³ and 100 dm³) is < 1.0, so according to SR EN ISO/CEI 17043:2010 it is confirmed that the standard volume measure can be used without any intervention. Therefore, it can ensure the traceability of the

volume measurement unit to other standard volume measures of another category. In this way the uniformity, accuracy and legality of the measurements performed in different fields where the volume is measured - for example wine industry, fuel industry is ensured.

Conflicts of Interest: The authors declare no conflict of interest.

References
1. GOST 8.400-2013 State system for ensuring the uniformity of measurements. Standard metallic gauges. Verification procedure. Approved: Federal Agency for Technical Regulation and Metrology, 30 December 2013 [in Russian].
2. EURAMET CG-21:2020 Guidelines on the Calibration of Standard Capacity Measures Using the Volumetric Method. Available online: https://www.euramet.org/publications-media-centre/calibration-guidelines (accessed on 20 October 2022).
3. ECA EURAMET CG-19:2012 Guidelines On The Determination Of Uncertainty In Gravimetric Volume Calibration. Available online: https://www.euramet.org/publications-media-centre/calibration-guidelines (accessed on 20 October 2022).
4. Sabadas, A.; Ensuring the traceability of measurement units. In *International Student Scientific Communications Session, ElStud XII-Editiona*, 21-23 June 2018 [in Romanian].
5. Velichko, O. N. Traceability of measurement results at different levels of metrological work. *Meas Tech* 2009, 52, pp. 1242–1248. Available online: https://link.springer.com/article/10.1007/s11018-010-9428-7 (accessed on 6 October 2022).
6. Research report SC 017:2022. Available online: https://inm.md/uploads/0/images/large/545c0635609f49bfc777cc620daefd99_sc-017-201-vulpe-roman.pdf, (accessed on 19 October 2022) [in Romanian].
7. Sabadas, A. Contributions to ensuring the traceability of volume measurements. In *IX International Scientific-Practical Conference Physical and Technological Problems of Transmission, Processing and Storage of Information in Infocommunication Systems*, 21-23 October 2021.
8. SM EN ISO/IEC 17025:2018 General requirements for the competence of testing and calibration laboratories [in Romanian].
9. ISO/IEC Guide 99:2017 International vocabulary of metrology. Basic and general concepts and associated terms (VIM). Available online: https://www.iso.org/obp/ui/#iso:std:iso-iec:guide:99:ed-1:v2:en (accessed on 25 October 2022).
10. Bich, W. Interdependence between measurement uncertainty and metrological traceability. *Accred Qual Assur* 2009, 14, pp. 581–586. Available online: https://link.springer.com/content/pdf/10.1007/s00769-009-0500-4.pdf (accessed on 27 October 2022).
11. SM SR EN ISO/CEI 17043:2011 Conformity assessment. General requirements for proficiency testing [in Romanian].
12. SM ISO/IEC Guid 98-3:2017 Uncertainty of measurement. Part 3: Ghuide to the expression of uncertainty of measurements (GUM:1995).
13. Metrology Law No. 19 of 03 April 2016. Published: 15 April 2016 in the Official Gazette No. 100-105 art. 190 [in Romanian]. Available online: https://www.legis.md/cautare/getResults?doc_id=106060&lang=ro (accessed on 3 November 2022).
14. General Regulation of Legal Metrology RGML 17:2015 "Traceability schemes of measurement units. Establishing principles. The method of elaboration, approval and use", Annex to the Order of the Ministry of Economy No. 13 of 09 February 2015. Available online: https://inm.md/static-files/data/415/file_1634_0.pdf (accessed on 28 October 2022) [in Romanian].
15. Mutual Recognition of National Measurement Standards and of Calibration and Measurement Certificated Issued by National Metrology Institutes, Paris:BIPM (1999), revised October 2003. Available online: https://www.bipm.org/documents/20126/43742162/CIPM-MRA-2003.pdf (accessed on 21 October 2022).

Publisher's Note: JES stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.

Submission of manuscripts: jes@meridian.utm.md

OPEN ACCESS CC BY

REMOTE SURVEILLANCE: A MEANS OF INTELLIGENCE GATHERING FOR MINIMIZING SECURITY CHALLENGES IN NIGERIA

Anas Shehu [1*], ORCID: 0000-0002-5307-1457,
Hadiza Aliyu Kangiwa [1], ORCID: 0000-0002-7363-9353,
Abubakar Sani [2], ORCID: 0000-0001-9616-0647

[1] Department of Computer Science, Kebbi State Polytechnic Dakin-gari, Dakin-gari, 862106, Nigeria.
[2] Department of Computer Science, Yusuf Maitama Sule University, Kano, 700214, Nigeria.
*Corresponding author: Anas Shehu, anasshehu8@gmail.com

Abstract. Nowadays, national security issues are increasing day by day in most countries. A multitude of measures to reduce the challenges have been presented and even implemented by many authors, but without exhaustive results. The use of computers and sophisticated IT tools by the terrorist group, increasing number of citizens, lack of social amenities and other factors have made some of them inadequate enough to control the problems in Nigeria. The purpose of this paper is to highlight national security challenges in Nigeria and how security oversight is operated. To achieve this, the authors analyze available secondary data, investigating national security modus operandi and presenting the general concept of surveillance. Related works were also investigated for discussion. Remote surveillance, wiretapping, geospatial intelligence and a consolidated national database are proposed to achieve digital intelligence collection for insecurity management.

Keywords: *geospatial intelligence, national database, national security, lawful interception, soft wiretapping, SIM card.*

Rezumat. În zilele noastre, problemele de securitate națională cresc zi de zi în majoritatea țărilor. O multitudine de măsuri pentru a reduce provocările au fost prezentate și chiar implementate de mulți autori, dar fără rezultate excaustive. Utilizarea de computere și instrumente sofisticate IT de către grupul terorist, creșterea numărului de cetățeni, lipsa de facilități sociale și alți factori au făcut ca unele dintre ele să fie suficient de inadecvate pentru a controla problemele din Nigeria. Scopul acestei lucrări este de a evidenția provocările de securitate națională din Nigeria și modul în care este operată supravegherea securității. Pentru a atinge acest scop, autorii efectuează analiza datelor secundare la dispoziție, investigând modus operandi de securitate națională și prezentarea conceptului general de supraveghere. Au fost investigate și lucrări conexe pentru discuții. Pentru a realiza colectarea de informații digitale în vederea gestionării insecurității, se propune supravegherea de la distanță, interceptările telefonice, informațiile geospațiale și o bază de date națională consolidată.

Cuvinte cheie: *informații geospațiale, baza de date națională, securitate națională, interceptare legală, interceptare soft, cartelă SIM.*

1. Introduction

Security is a feeling of safety, protection, and relative freedom [1]. This security is always the number one priority according to Maslow's order of needs besides psychology and as such, each nation's responsibility is to provide its citizen security. And has become a basic necessity of human beings and society [2].

Any security threats are known as security challenges [3]. The rate at which security challenges are progressing in countries of the world is ubiquitous, Nigeria as a sovereign nation will not be exempted. Many Nigerians will agree to the fact that many disparate security mechanisms vis-à-vis human and technological were suggested and even implemented to minimize the menace, but to no avail; due to its pervasive nature and the huge threats it poses to human lives and material belongings. The aforesaid proposal and strategies suggested by previous writers are insufficient enough, and therefore; it has to call need for other means and strategies to curb it.

The notion of insecurity may be seen as subjective depending on an individual defining it based on a situation and particular place. But, two things might lead to its definition and meaning. 1. *Public upheaval* 2. *Threat to peace* [4]. Violation of state criminal law can destroy human and other resources leading to human abduction, murder, suicide bombing, armed robbery, armed banditry and so forth [5]. These criminal activities that cause insecurity are engaged with the aid of tools of information and communication technology such as mobile phones, the internet, software, and the like [6].

Additionally, these security challenges are facing Nigeria for more than two decades. Recent among them are kidnapping, banditry, and herdsmen/farmers clashes [7]. Though their root causes are not far from politics, sectional agitations, ethnic & religious crises, militias, boundary disputes, cultism, criminality, and organized crimes" [8].

Technology has revolutionized how humans interact and communicate day by day. This revolution has evolved the world and made it a global village where humans pass messages - text, audio, and video comfortably from any angle wirelessly. Because of the pervasive nature of technology, criminals, and terror groups leverage this technology for national insecurity.

Therefore, a plethora of modern technological means was suggested by researchers to minimize the vices, that include human policing, *close circuit television* (CCTV) cameras, Data mining, and biometric-based system. They remain insufficient, taking cognizance of Nigeria's increased number of citizens and other factors.

Therefore, there is a need for cutting-edge technological ways of digital intelligence gathering and analysis that will match the challenges. Consequently, a strategy of *digital surveillance* is proposed to detect communication contents, location, time, and other communication metadata of communicants—citizens communicating over telecommunication or social media networks.

2. Contributions

Contributions of this work include a brief overview of Nigeria's national security challenges and how it's currently being operated conventionally. Modern IT-based surveillance for tackling insecurity in Nigeria will be seen as second contribution. The third contribution will cover surveillance concept.

And lastly, the discussion and way forward to Nigeria security community to handling these challenges will be covered.

3. Methodology

In this section, we will present the methodology used in the conduct of this work including analysis of secondary data at our disposal, investigating Nigeria national security modus operandi, presentation of general concept of surveillance, related work and finally, discussions on the way forward towards tackling insecurity in Nigeria.
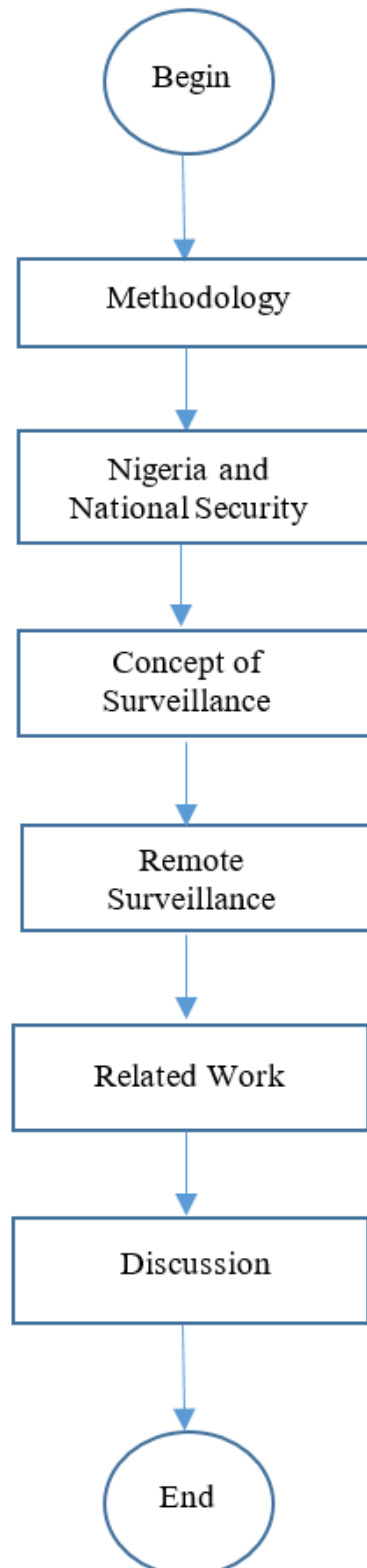
Figure 1. Activity diagram.

4. Nigeria and National Security

Nigeria as a nation is struggling with security challenges—*Southeast*, Indigenous People of Biafra (IPOB), *Northeast*, Boko Haram, *Northwest*, banditry, cattle rustling, kidnapping for ransom, *Southwest*, herdsmen/farmers clash, and *Southsouth* with militancy and oil bunkering. All these could not be possible without the aid of communication technologies such as cell phones and social networks [9]. For instance, the bombing of Super screen TV stations in Lagos in 2009 was coordinated by mobile communications. Another example noted in [10] where Boko Haram sects use computer and other information communication technology (ICT) tools in achieving their objectives on social media platforms and also communicate among themselves. Another famous example was Khalid Almidhar and Nawaf Alhazmi who used mobile networks (though arguable) to coordinate the 9/11 attack in the USA[11]. In Nigeria, cyberspace insecurity activities and actions are taking place with no fear. Nigeria, as of July 2022, has recorded a total number of active SIM cards for cell phone users to be (208, 61,000) making calls and other network-related engagements [12]. With the orthodox security architecture in Nigeria [13], it is impossible to track down and acquire sufficient intelligence in large numbers. To this end, the country's security agencies have at their disposal abundant sources of intelligence gathering—digital devices owned by individuals and internet facility. But, only if Nigerian security abolishes traditional and paper-based security operations and information sharing [14] and embraces modern IT equipment blended with our proposal for systematic and efficient adoption. That could only be the alpha and omega solutions to modern security issues that are going digital day by day.

5. Concept of Surveillance

Surveillance is legal if the government has lawful facts to do it without using it as a vehicle of political intimidation or oppression or reducing citizens' autonomy [15]. Surveillance as mentioned in [16] is "monitoring of behaviors, activities or other changing information to influence, manage, directing or protecting people. State surveillance is lawful when undertaken for legitimate means [15] as technology permeates our daily lifestyle such as in education, transactions, manufacturing, and production, defense, etc., coupled with the digitization of many activities, items, and events indicating individuals produce a significant amount of communication data. These communication data produce an output report of one's movement over time which, in turn, becomes an integral part of the nation's surveillance. Additionally, electronic surveillance gives an idea of what communicants are saying or planning. The majority of foreign governments at the national level embrace digital surveillance because the vast volume of communications moving over the internet is beyond human understanding. The nations are the United State of America [16], the United Kingdom [17], Australia, China [18], and Bangladesh which avert insecurity challenges that going digital and moving over communication networks within society. Government surveillance programs include *landlines, cell phones, mobile devices—tablets, laptop computers, online communications, search engine queries, social media,* and the like [16]. Through this surveillance, intelligence would be deduced and analyzed for further actions. As noted in [19] "*intelligence*" are series of actions and policies observed by any nation to forestall internal/ external threats to its territories, economy, and stability. The authors in [2] Argue that intelligence is a cluster of people and organizations that carry-out missions of collections and analysis, counterintelligence, and covert action. With modern information and communication technology, executing digital intelligence is simpler and, allows joining

together communication data that gives a big and comprehensive picture of an individual's communication pattern and behaviors that point to threats to national security [16]. Consequently, UK intelligence & security services were reported to have used the bulk communication data for citizen protection of human rights—security. Finally, this special information gained is what provides a state of the nation, organization, or individual basis to progress its action plan toward securing the country and other individuals.

6. Remote Surveillance
6.1. Wiretapping

National security agency (NSA) via their ex-contractor was reported to have said that NSA has been collecting US citizen's calls logged in greater number. And this called for the interpretation of some sections of the patriot Act [20]. The act was implemented in full capacity in the United State of America and also inspired intelligence gathering in various means [21]. The act provides legal and constitutional backing for forestalling terrorist acts and maintaining the security of the nation. Wiretapping was one of the techniques employed by the US intelligence community for call logs and other things. Wiretapping is a way of interception and listening to communication over a network without the consent of communicants [22].

The act of interrupting and taking total control of oral and electronic communication media is also called wiretapping [23]. Lawful wiretapping is practical in every democratic state [24] the patriot act bill might be activated or implemented in Nigeria towards achieving maximum security via eavesdropping. Couple with other plethora of laws/acts by Nigerian government which   provide lawful interception and surveillance such as: *Lawful Interception of Communication Regulations* (preventing, investigating and protecting crime)*, Cybercrime Act 2015* (warrant to search data deposited in computer or network and to apply any technology to decode, decrypt messages)*, Part V of Mutual Assistance in Criminal Matters Act 2019* (for surveillance, interceptions of communications and postal items)*, Terrorism Prevention Amendment Act 2013* (power to intercept communications to forestall act of terrorisms and offences)*, Nigerian Section 147 of Communication Commission Act 2003* (provides capability to intercept digital communication [25] Nigeria security agencies are obligated to launch the desired eavesdropping gadgets and software to intrude all cell phone network communications of all callers or suspected callers for state security (if at all the SIM cards registration database is active and running).

Intercepting mobile phone calls is much easier because its signals are on the air. It needs only unwired devices of interception [26]. As noted in [27] when insecurity issues arise, there is a significant rise in domestic and international surveillance which call the use of wiretapping. But as cited by the aforementioned authors, the reverse is the case in Nigeria. Thus, activating laws/bills is essential for interrupting or hijacking cell phone communication for state security. This proves to be more probable and less difficult than on landline conversation. The most suitable wiretapping is *soft wiretapping,* where information over communication lines on the ply on phone devices is being studied. Its implementation is on network provider's equipment [28] and is more covert, user-friendly and inexpensive [29]. It is a common culture of intelligence agencies, larger organizations, and the black box in a developed country [30]. A research conducted by [31] proved that 96% of intercepted communications display and present location data to intelligence communities. For instance, in 2010, the USA army succeeded in tracking down Abu Ahmad al-Kuwaiti who was a courier

service provider working for Osama bin Laden. And this led to the death of Bin Laden in a city called Abbottabad, Pakistan [32]. Intelligence communities are always on alert for suspicious conversations, that is when they will access relevant wiretapping systems for location information to further their actions. As pointed out by Nigerian Communication Commission Act 2003, the Nigeria government could be made it necessary for all mobile network providers working in the country to install and activate wiretap capability network (if not installed) as stated in Nigerian Communication Commission Regulations 2011, Nigerian Communication Commission Regulations 2019 and the USA Communications Assistance for Law Enforcement Act (CALEA) 1994 [32].

As reported in [15,25], the Nigerian government procured surveillance technologies from Israel in 2013, and also collaborated the same year with Firm Circle - a telecom Spy Company; to spy on political opposition in Nigeria. These revelations of surveillance acquisitions and domestic laws/acts in place, plus witnessing a high increase in security challenges; indicated little or no application of wiretapping by security agencies for state security. Even if wiretapping exist in Nigeria then, is for tracking the opposition as noted in [25]. Nigerian security community can choose to adopt one of these or some combinations of *soft wiretapping, hard wiretapping, and* use of *active* and *passive devices* for its covert intelligence operations. Both active and passive technologies were used sometime in 1995 to fish out notorious hackers.

Wiretap terms

Hard Wiretapping: Physical wire is attached for signal interception.

Soft Wiretapping: Network information is analyzed as it moves on. Is achieved through mobile phone software.

Passive Device: a technology use for intercepting close-range mobile phones signal and that of service provider's networks. It takes advantage of soft encryption or absence of any during signal transmission. This technology is more invisible during its operation.

Active Device: a technology used for interception of communication contents between mobile phones. Takes advantage of the absence of authentication of base station by mobile phone. Is less invisible as they produce telltale light.

6.2. Geospatial Intelligence (GEOINT)

The contemporary world now is seeing a lot of technological changes with rapid phase day in and day out by making us understand what is going on at a particular time in the exact venue. This is only achievable with ICT and, geospatial intelligence is component of it [33]. Geospatial intelligence is the defined as the "exploitation and analysis of imagery and geospatial information to describe, assess and visually depict physical features and geographically referenced activities on the earth" [34]. Geospatial intelligence comprises imagery, imagery intelligence, and geospatial information. Imagery, also known as "photograph intelligence" [35], gives thorough analysis of the images in question (whether still picture or full-motion video that was reproduced electronically or by optical means) [34] data to locate, group, and recognize objects or any establishment [36]. GEOINT data is gathered through space-borne, airborne sensors stationed on the ground. GEOINT covers large geographical distance and helps in providing scientific geolocation of the target. Through the air, imagery could be acquired via satellite unmanned aerial vehicles and reconnaissance aircraft. Videos and pictures acquired or posted by terrorist groups will be analyzed for scientific location when blended with geospatial information techniques.

Geospatial intelligence was used to locate the Iraqi terror group training camp which even revealed the timing of the coverage and the camera location. Furthermore, with this geospatial intelligence, the exact place where a United States journalist was executed was spotted after a photo examination was carried out via video posted on social media by the sect [37]. Again, the geolocation of social media users was used by the US intelligence service to map out refugees fleeing Syria [34]. These were and would be achieved when optimum utilization of *Geospatial Technologies* by Nigerian security agencies such as the State Security Service (SSS), Defense Intelligence Agency (DIA), and other security communities is in full force [38] to know the whereabouts of victims kidnapped by bandits for ransom, bandits themselves or Boko haram sect.

For law enforcement agencies to start the war against insecurity without using GEOINT, is as simple as fighting nobody[33]. For the security community to apply imagery intelligence, Geospatial intelligence toolkits should be employed.

6.3. National Database

As in other developed countries such as the US, UK, Germany, Canada, China, and so on, the Nigerian security architecture should have a consolidated database that contains comprehensive information on citizens and foreigners living in the country. It is the same database that will be used to link individuals using wiretap contents or metadata as the case may be and/ or image analysis was done to identify the suspects. The database holds many advantages to the security community by using it to play back on how crime has happened and who was involved. Also to use this data store is like hastening investigation because of available information that can be reached quickly. It is essential to note that developing this data store is simple and easy. Since Nigeria had claimed to have updated the Independent National Electoral Commission (INEC) database, National Identity Management Commission (NIMC) database, National Population Commission (NPC) database, bank database, driver's license and plate number database, SIM card registration database, and so on. Further, an electronic transport system (as a mobile app) should be implemented, plus automatic vehicle plate number systems for vehicle movement in real-time should exist. These disparate data could be integrated and consolidated into the single and robust data warehouse for intelligence analysis and the like.

7. Related Work

This section will present works related to digital surveillance or the application of any ICT tools/systems in observing nation-state security.

Presentations were made by [9] that explain how information and communication technology is used to create unrest and security issues in the country. Also, the same technology could be used to stabilize national security. Though no clearer direction was provided on how to achieve that. The strategy proposed by [39] pointed out the value to be achieved when geospatial intelligence is used to track down the insurgency. Photographs and videos taken from the satellite give much idea on the location of the terrorist camp, the objects spotted in the photograph will also be different from one another base on the type (person for person, sea for sea, and tree for tree, etc.). Global Positioning System (GPS) is one of the emerging technology used in recent times for counter-terrorism [6] which uses triangulation to measure and determine the position, time, and speed of an object at a point attached to the system.

Data mining technology was proposed as a means of discovering notorious personalities, financial movements, unusual patterns, and behavior through intelligence to

fight crime-related acts associated with them [39]. The data mining schemes like *Classification* and *Link Analysis* would help to ascertain genuine patterns of information and behaviors which, in the end, meet intelligence objectives. Additionally, activities like phone calls visited places and email contacts could be linked to one another statistically for additional intelligence analysis. In [12] biometrics means were proposed to curb the insecurity in the country. And it is an automated way of fishing out a personality using physical, physiological, or behavioral characteristics through identification and verification. The features to consider in this process are facial features, retina, fingerprint, and iris. [40] Proposed a system that is viable to point out a suspected terrorist under the security watch list during screening in the airport and whose facial features were pre-processed in biometric systems. This system is so profound that it did not label innocent as culprits or the other way round. The contribution in [40], a biometric system based on fingerprint was designed to authenticate personality gaining access to the building with an IP-video surveillance camera connected to it serving as an intelligence vehicle with the capability to report to the terminal any suspicious movement and unwanted gatherings. In the work of [13], digital devices and software were mentioned in the presentation and deployed for usage to Nigerian security agencies but had little or no impact on minimizing the security challenges facing Nigeria. Amongst the technology is: *close circuit television* (CCTV) supposed to be installed in Lagos and Abuja in large numbers but, only a few were installed. *Global Positioning Systems* (GPS) are supposed to be installed in cars, *Pre-Arrival Assessment Reports (PAAR)* for customs operations, *biometric technology, and SIM card registration* for all Nigerians, *Integrated (IPPIS)* and *Bank Verification Number (BVN)*. Though listed some elements militating the successful implementation of these technologies that included 1. Digital divide 2. Insufficient training for security personnel and 3. The dearth of political will.

Presentation of essential use, nature, and analysis of intelligence sources by the US intelligence community were made [34]. In the contribution, sources of intelligence were stated such as Human Intelligence (HUMINT), Geospatial Intelligence (GEOINT), Signal Intelligence (SIGINT), Measurement and Signature Intelligence (MASINT), and Open Source Intelligence (OSINT). Detail role of each source and the problems associated−i.e. financial, were elaborated. The composition and designation of some US intelligence communities were also mentioned. In another research conducted by [41], the history of imagery intelligence (IMINT), its origin, and previous applications in world wars I and II were highlighted. Modern-era UAVs and the future of IMINT for the usage of the intelligence community (more specifically military) were discussed. In another work presented by [42] application of information technology via Geographic Information Systems was proposed to tackle crime and insecurity. In the document, the author provides a model where crime can be mapped and located. Finally, hotspot mapping techniques were elaborated on in the report. The work of [43], provide insecurity issues in Nigeria emanating from Niger Delta militancy, kidnapping from South East, Boko haram insurgency rooted in North East, the herdsmen crisis in Benue, and banditry centered in the Northwestern states. However, the author proper technological solution to this menace by employing aerial drones for state surveillance. He however further itemized the functions of some drones for deployment. Another proposal by [44], pointed to security challenges posed by Boko Haram and their application of information and communication technology (ICT) for intelligence gathering and launching mayhems to citizens. The authors noted that ICT gadgets and systems should

be used for surveillance and intelligence gathering. Also, a central intelligence unit and development of any single identification means were proposed.

In another work done by [45], techniques were proposed where personnel can intercept wireless communications by sending a signal through the jamming antenna to hear out the transmitting messages. In a similar research by [46] techniques of using a receiver that can divide the message communicated into two−interception and spoofing is used. The technique employed optimizes the eavesdropping rate more than traditional eavesdropping does. New means of checkmating insecurity in public safety networks, national security centers and national databases were among them [47]. The authors also listed some ICT gadgets/means to improve national security architecture. Intelligence gathering, interception of messages, use of geographic information systems, launching fingerprint systems in almost all endeavors, deployment of the crime management systems and so on were also itemized in the report. Furthermore, the research outlined a list of basic technological devices and means to help control insurgency in Nigeria.

The use of big data analytics to analyze social media contents for intelligence gathering by citing free analytics for security agencies to use in the country, Nigeria. Specifically, there are some tips that will aid intelligence community for effective social media analysis[48]. Integration of data modeling with the Geographic Information System (GIS) database for profiling terror group location and predicting the sect pattern of activities for better close observations was proposed in[49]. Research work conducted by [50] proposed the use of a Global Positioning System (GPS) chip by an individual on their body for easy tracking and locating when kidnapped or in danger. Also, issues facing the application of technology in Nigeria's security domain such as insufficient training, dearth of the fund, consolidated national database, and absence of security satellites were expatriated. In another research conducted by[51], weighted linear combination was used on top of geospatial intelligence for identification and fishing out terror groups' location and territory in Sulawesi Province, Indonesia. Among the beauty of their work is using water source, slopes, settlements and land cover as research parameters. On the other hand, [52]made presentation on how geospatial technology could be of help in surveillance and security research within the academia. They stressed how importance it is to acquire such knowledge for proper tracking, mapping and digitizing terrorist actions on crime map. Consequently, GEOINT research group was formed to cover six geological zones in Nigeria with one university in each zone covering the activities, plus the authors base university.

Authors in [53] made presentations as surveyed papers on the applicability of GEOINT by counter-terrorism squads to subduing insecurity. It was reported by many of the respondents that GEOINT has a role to play in conquering terrorism in the country. The work specifically focused on the Boko Haram insurgency between 2015 to 2018. In another work prepared and presented by[54], the history of the US laws mandating legal wiretap was expatriated. Also, wiretapping concepts and a few of its devices were mentioned. The researchers argued the use of wiretapping techniques citing harm caused to US citizens in the process, but; suggested the use of vulnerabilities technological devices to listen to conversations and other digital messages which the US security community are using secretly and in small quantity. Analyzing social network content gathered by its users over time for solving issues of terrorism was proposed by[55]. Examples of how SNA helped mapped out terrorist groups and their networks were stated in detail. The author highlighted two sub-division of SNA - data collectors and modelers. Nigerian government should take hold of

information communication technology (ICT) in checkmating insecurity in its bushes, forests, and other terrorist hiding places. There exist national space laws in the country that can be relied on in using drones, helicopters, close circuit television (CCTV), and satellite to guard them which in turn keep an eye on and terrorize criminals [56]. Based on the survey results compiled, they conclusively made recommendations that the Federal Government of Nigeria should fund technology in the security cycle. Train them on modern technology and data analysis. The national database was among the recommendation also. An interesting proposal was made in [57] where, smart objects and surveillance technologies could be fused into one single system for proper monitoring, controlling, and overseeing of citizens' affairs for security and other purposes. It was assumed in the framework that all other security agencies have dedicated system for their affairs, including office of the national security adviser. These systems are then connected to national secured and centralized systems via secured internet connection and pass on data to and fro. However, on one hand, smart objects, GPS, surveillance camera and the like are connected and transmit needed data through a common gateway to national database.

From the literature above, it is evident that none of the author(s) made wider coverage/ proposal of using wiretapping for all incoming and outgoing calls by Nigerian citizens for the sake of intelligence gathering. Also applying geospatial intelligence couple with wiretapping for detecting images and locations of criminals/terrorist and warehouse them in a single data store for analyses and interrogations were not fully addressed. Based on these, we can say that Nigeria security community is operating in a conventional way — human intelligence for tackling insecurity, hence the need for our proposal to Nigeria's security personnel.

8. Discussion

From the above literatures and other submissions, we can observe that some authors suggest application of GPS only for tackling insecurity in Nigeria. Other writers only proposed the use of data mining to fish out the criminals when a correlation was found. For some authors, we can see that their proposal was only for the use of biometric to identify criminal at the gate of domestic or national airport in the country. Other group of papers were based on deploying ICT hardware and software (national database and CCTV) alone for neutralizing insecurity in Nigeria. And finally, some authors suggest the Nigeria security to deploy Ariel drones for surveillance. This proposal was not feasible because all the five satellites belonging to Nigerian government, none is allocated for gaining intelligence [39], so Ariel drones might be difficult. It is important to note that, data mining, CCTV, GPS, GIS, biometric, Ariel drone and national database as independent system alone may not solve Nigeria security issues without having digital intelligence gathering in place, couple with consolidated national database for linking the information and then employ analysis and trigger actions. It is worthy to note that many wars against insecurity were fought with success because of IT-based intelligence facilities [58]. It is equally important to know that counter-terrorism flourishes with the help of digital intelligence in this information age. Nigeria as a nation is and has witnessed security crises which inspired federal government to have pointed it as a reason for surveillance [25]. Hence the need of our proposal.

9. Conclusions

Security is the number one priority of every country to give to its citizen. The need for a suitable and trusted strategy to relax the insecurity issues is timely. Our Remote surveillance proposal only needs Nigeria's espionage/intelligence community to collaborate

with mobile phone service providers to access their call record database/ duplicate the contents or mount intelligence devices on their respective towers or apply the two. Nigeria security agencies can also learn from or corroborate with Egypt, Libya, Bahrain, Saudi Arabia, United Arab Emirates, and India to acquire the intelligence technology as them as noted by another author above. To our knowledge, succeeding in this domain of security, a citizen must sacrifice their privacy in exchange for security. Of importance, to keep crimes and insecurity in check in this information age, technology-based techniques should be embraced, deployed, and relax the orthodox methods in use since Nigeria's independence. Since Nigerian government procured surveillance technologies from Israel and also formed a collaboration with a spy company. Then, using physical power by law enforcement agencies might not win the war against insecurity without the acquisition/deploying greater intelligence. In future work, the authors will cover the nitty-gritty of how modern IT-based intelligence-gathering/ surveillance devices function plus their classifications and architecture.

Conflicts of Interest: The authors declare no conflict of interest.

References
1. Abdullahi, A.S.; Mukhtar, J.I. Armed Bnaditry as a Security Challenges in Northwestern Nigeria. *African Journal of Sociological and Psychological Studies* 2022, 2, pp. 45-62.
2. Abiodun, T.F.; Oludotun, A.; Chukunyere, N.; Oladejo, A.A. Security Intelligence Cooperation and the Coordinated War on Terror Among Nigeria's Security Agencies: Panacea to Stable National Security. *Global Scientific Journals* 2019, 7, pp. 542-566.
3. Eke, C.C. Terrorism And The Dilemmas Of Combating The Menace In Nigeria,. *International Journal Of Humanities And Social Science* 2013, 3, pp. 1-7.
4. Okeke, G.N. A Legal Approach To Combating Terrorism: Modern Dimension. *Journal Of International Law & Jurisprudence* 2011, 2, pp. 293-303.
5. Gony, A. Red Teaming The Red Team: Utilizing Cyber Espionage To Combat Terrorism. *Journal Of Strategic Security* 2013, 6, pp. 1-9.
6. Akinode, J.L.; Alawode, A.J.; Ojuwo, O.O. Improving National Security Using GPS Tracking System Technology. *African Society for Scientific Research* 2011, pp. 634-645.
7. Emmanuel, A.O.; Eyinnaya, O.L. Critical Thinking in Information Technology and Management for National Security in Nigeria. *Asian Journal of Applied Science and Technology* 2019, 3, pp. 41-52.
8. Okechukuwu, A.T.; Onyekaneze, O.O. Forensics TechnologyDeoxyribonucleic Acid (DNA) Profiling in Security Management and Fight Against Insecurity/Terrorism in Nigeria. *Journal of Humanities and Social Science* 2019, 24, pp. 36-45.
9. Maryam, U.B. Available online: http//:punchng.com (accessed on 12 October 2022).
10. Chinda, F.E.; Shuaibu, A.N.; Dyikuk, J. Information Technology as an Indispensable Security Tool: Nigeria's Boko Haram Sect in Focus. *International Journal of Applied Research and Technology* 2018, 7, pp. 39-51.
11. Hewitt, M. *Wiretapping: A Neccessity for Effectively Combating Terrorism in the 21st Century*. Liberty University, 2008; pp. 1-49.
12. Chima, P.; Joseph, I.S. Adoption of Digital Solutions in Managing Security Challenges of the 21st Century in Nigeria Option for Effective Responses. *Journal of Management Sciences Research* 2022, 2, pp. 334-341.
13. Tanriverdi, H.; Chen, H. Government Digital Surveillance and Citizan's Self-censorship of Technology Use. In *Proceedings of the Thirty ninth International Conference of Information Systems*, San Fransisco, USA, 2018.
14. The Role of Information Technology in Enhancing National Security in Nigeria (2001-2020). *Pinisi Journal of Art, Humanity and Social Studies* 2021, pp. 44-53.
15. Oloyede, R.; Roberts, T.; Mohamed, A.A.; Farahat, M.; mutung'u, g. *Surveillance Law in Africa: A Review of Six Countries*; Brighton, 2021; pp. 2-203.
16. Milanovic, M. Human Rights Treaties and Foreign Surveillance: Privacy in the Digital Age. *Havard International Law Journal* 2015, 56, pp. 81-146.
17. Milward, J.; Peterson, D. China's System of Oppression in Xinjiang: How it Developed and How to Curb it. *Global China: Assessing China's Growing* 2022, pp. 1-25.
18. Eke, G.E.; Okechi, H.A.; Uche-Nwachi, E.O.; Ekeh, L.K. Science-Systems as a Catalyst to Sustainable National Security in Nigeria. *Science World Journal* 2021, 16, 3 pp. 97-401.

19. Richard, H.J.; Don, G. Intelligence And Its Role In Protecting Against Terrorism. *Journal of Strategic Security* 2010, 3, pp. 31-38.

20. Soghoin, C.; Pell, S.K. A Lot More Than A Pen Register, And Less Than A Wiretap: What The StingRay Teaches us About How Congress Should Approach The Reform of Law Enforcement Surveillance Authorities. *Yale Journal of Law & Technology* 2013, 134, pp. 134-171.

21. American Heritage Dictionary. Available online: www.legal-dictionary.thefreedictionary.com/wiretapping (accessed on 12 August 2015).

22. Steven, B. Law.illinois. Available online: http://www.law.illinois.edu/bljournal/post. (accessed on 2 September 2012).

23. Steven, B.M.; Matt, B.; Sandy, C.; Susan, L. Lawful Hacking: Using Existing Vulnerabilities For Wiretapping On The Internet. *Northwestern Journal Of Technology And Intellectual Property* 2014, 12, pp. 1-64.

24. Accountable Wiretapping-or- I Know They Can Hear You Now. *Journal of Computer Security* 2015, 23, pp. 167-195.

25. *Surveillance Law in Africa: A Review of Six Countries.* Institute of Development Studies: Brighton, 2021, pp. 103-131.

26. Pell, K.S.; Soghoian, C. Your Secret Stingrays No Secret Anymore: The Vanishing Government Monopoly Over Cell Phone Surveillance and Its Impact on National Security and Consumer Privacy. *Havrd Journal of Law & Technology* 2014, 28, pp. 1-76.

27. The Correlation Between Wiretapping and Terrorism: A Comparative Analysis of American and European Societal Views on Government Surveillance. *ILSA Journal of International & Comparative Law* 2016, 23, pp. 55-74.

28. Yee, M.J.; Philips, S.; Condon, G.R.; Jones, P.B.; Kao, E.K.; Smith, S.T.; Anderson, C.C.; Waugh, F.R. Network Discovery with Multi-Intelligent Source. *Lincoln Laboratory Journal* 2013, 20, pp. 31-46.

29. Your Secret Stingrays No Secret Anymore: The Vanishing Government Monopoly Over Cell Phone Surveillance and Its Impact on National Security and Consumer Privacy. *Havrd Journal of Law & Technology* 2014, 28, pp. 1-76.

30. Blake, N.C. The Constitution And The NSA Warrantless Wiretapping Program: A Fourth Amendment Violation?? *Yale Journal Of Law And Technology* 2009, pp. 228-259.

31. Surveillance, T. seussbeta.tripod.com. Available online: http:///seussbeta.tripod.com (accessed on 2 September 2015).

32. Ryan, D.; Glenn, G.; Laura, P. firstlook. Available online: "www.firstlook.org (accessed on 13 October 2022).

33. Understanding Geospatial Intelligence and the Challenge of Effective Counter-Terrorism Strategy: A Case Study of Nigeria's Boko Haram Challenge. *Unnes Law Journal* 2020, 6, pp. 163-185.

34. Intelligence Sources in the Process of Collection of Information by the US Intelligence Community, "Security Dimensions", 2019, pp. 82-105.

35. Staff, I.O.S. *Operations Security Intelligence Threat Handbook.* 1996, pp. 1-80.

36. Margaret, Hu. Available online: http://www.washingtonpost.com (accessed on 5 September 2022).

37. Sani, A.; Paki, Z.S.; Hadiza, A.U. Location Information: Another Perspective of Intelligence Gathering for Minimizing Terrorism in Nigeria. *International Journal of Scientific & Engineering Research* 2015, 6.

38. Emenari, S.U. The Application Of Geospatial Intelligence In National Security For Sustainable Development To Combat Terrorism Insurgence In Nigeria. *Journal Of Environmental Science, Toxicology And Food Technology* 2014, 8, pp. 2319-2402.

39. Okonkwo, R.O.; Enem, F.O. Combating Crime And Terrorism Using Data Mining Techniques. In *Proceedings of the Nigeria computer society (NSC): 10 th International conference*, 2011.

40. Akinmosin, A.S.; Yusuf, S.E.; Dada, A.M. Combating Terrorism With Biometric Authentication Using Face Recognition. In *Proceedings of the Nigeria computer society (NSC): 10 th International conference*, 2011.

41. Guide to Imagery Intelligence. *Journal of U.S. Intelligence Studies* 2011, 18, pp. 61-64.

42. Crime Mapping in GIS by Using Hotspot. *SJCMS* 2018, 2, pp. 13-19.

43. Usage of Drones or Unmanned Aerial Vehicles (UAVs) for Effective Aerial Surveillance, Mapping System and Intelligence Gathering in Combating Insecurity in Nigeria. *African Journal of Social Sciences and Humanities Research* 2020, 3, pp. 29-44.

44. Information Technology as an Indispensable Security Tool: Nigeria's Boko Haram Sect in Focus. *International Journal of Applied Research and Technology* 2018, 7, pp. 39-51.

45. Xu, J.; Duan, L.; Zhang, R. Proactive Eavesdropping Via Copgnitive Jamming in Fading Channels. *arXiv* 2017, pp. 1-16.

46. Zeng, Y.; Zhang, R. Wireless Information Surveillance Via Proactive Eavesdropping With Spoofing Relay. In *Proceedings of the 4th IEEE International Conference on Acoustics, Speech and Signal Processing*, Shanghai, China, 2016.
47. Oludare, A.I.; Omolara, O.E.; Umar, A.M.; Kenni, D.V.; Ezenwosu, A.C. Harnessing Information Communication Technology (ICT) in Handling Crime and Terrorism in Nigeria. *Journal of Social Science and Public Politics* 2015, 5, pp. 1-25.
48. Jibril, M.L.; Mohammed, I.A.; Yakubu, A. Social Media Analytics Driven Counterterrorism Tool to Improve Intelligence Gathering Towards Combating Terrorism in Nigeria. *International Journal of Advance Science and Technology* 2017, 107, pp. 33-42.
49. Idhoko, K.E.; Ojaiko, J.C. Integration of Geographic Information Systems (GIS) and Spatial Data Mining Techniques in Fight Against Boko Haram Terrorist in Nigeria. *International Journal of Science and Research* 2015, 4, pp. 1932-1934.
50. Udochukwu, E.; Christian, S.I.; Adebayo, A. The Application of Geospatial Intelligence in National Security for Sustainable Development to Combat Terrorism Insurgence in Nigeria. *Journal of Environmental Science, Toxicology and Food Technology* 2014, 8, pp. 11-16.
51. Utomo, A.M.; Wijayanto, G.N.; Yusfan, M.A.; Wardani, M.; Poniman, A.; Supriyadi, A.A.; Gultom, R.; Martha, S. Geospatial Intelligence Analysis to Support National Defense Interest. In *Proceedings of the 2021 International Conference on Advanced Computer Science and Information Systems*, 2021.
52. Nwachukwu, M.A.; Nwachukwu, J.; Anyanwu, J.; Babatunde, A.; Ekweogu, C.; Nwachukwu, A.N. Geospatial Intelligence Training Concept for Terrorism Surveillance, Nigeria to Infusive Sub-Saharan African Countries. *American Journal of Geosptial Technology* 2022, 1, pp. 44-51.
53. Nte, N.D.; Abdulaziz, A.B.; Uzorka, M. Understanding Geospatial Intelligence and the Challenge of Effective Counter-Terrorism Strategy: A Case Study of Nigeria's Boko Haram Challenge. *Unnes Law Journal* 2020, 6, pp. 163-185.
54. Bellovin, S.M.; Clark, S.; Landan, S. Lawful Hacking: Using Existing Vulenarabilities For Wiretapping on The Interner. *Northwestern Journal of Technology and Intellectual Property* 2014, 12, pp. 1-64.
55. Ressler, S. Social Network Analysis as an Approach to Combat Terrorism: Past, Present and Future Research. *Homeland Security Affairs* 2006, 2, pp. 1-10.
56. Chima, U.H.; Muritala, O.; Elisha, N.C. Harnessing Information and Communication Technology (ICT) For The Management of Ungoverned Spaces in Nigeria: Policy and Strategic Way Out. *International Journal of Development and Management Review* 2020, 15, pp. 17-31.
57. Obodoeze, F.C.; Ozioko, F.E.; Okoye, F.A.; Mba, C.N.; Ozue, T.I.; Ofeogbu, E.O. The Escalating Nigeria National Security Challenge:Smart Objects and Internet-of-Things to The Rescue. *International Journal of Computer Networking and Communication* 2013, 1, pp. 81-94.
58. Michael, H. *Wiretapping: A Necessity For Effectively Combating Terrorism in 21st Century*; liberty university: 2008, pp. 1-49.

Publisher's Note: JES stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.

Submission of manuscripts: jes@meridian.utm.md

THE DIFFERENCE BETWEEN CYBER SECURITY VS INFORMATION SECURITY

Arina Alexei *, ORCID: 0000-0003-4138-957X,
Anatolie Alexei, ORCID: 0000-0002-0570-4854

Technical University of Moldova, 168 Stefan cel Mare Blvd., Chisinau, Republic of Moldova
Corresponding author: Arina Alexei, arina.alexei@tse.utm.md

Abstract. The terms cyber security and information security are often used interchangeably, both in the academic literature and by organizations. This article aimed to identify whether these terms can be used interchangeably, analyzing the differences between the terms, based on several definitions identified in scientific articles and books, as well as definitions presented by specialized organizations. The analysis consists in determining the keywords, that define the terms, based on which conclusions were made. It has been determined that information security places a strong emphasis on the protection of information, cyber security refers to all assets that are part of cyberspace, such as end devices (including IoT), network devices, communication media and information. Cybersecurity is an umbrella term that includes: electronic communications network and computer security, physical security, infrastructure security, personnel security, hardware and application security, business process security. Through the identified results, important contributions are made both for the academic environment, as well as for governmental and non-governmental organizations, for the understanding and correct use of terms, to reduce uncertainty and to correctly approach specialist terminology, because the field of security is increasingly important, the challenges are complex and diverse.

Keywords: *asset, availability, confidentiality, cyber, information, integrity, threat.*

Rezumat. Termenii de securitate cibernetică și securitate a informației sunt adesea folosiți în mod interschimbabil, atât în literatura academică, cât și de către organizațiile specializate. Acest articol și-a propus să identifice dacă acești termeni pot fi utilizați interschimbabil, analizând diferențele dintre termeni, pe baza mai multor definiții identificate în articole științifice și cărți, precum și definițiile prezentate de organizațiile specializate. Analiza constă în determinarea cuvintelor cheie, care definesc termenii, în baza cărora s-au făcut concluzii. S-a stabilit că securitatea informației pune un accent puternic pe protecția informației, securitatea cibernetică se referă la toate activele care sunt parte a spațiului cibernetic, cum ar fi dispozitivele finale (inclusiv IoT), dispozitivele de rețea, mediile de comunicații și informația. Securitatea cibernetică este un termen umbrelă care include: securitatea rețelelor de comunicații electronice și a computerelor, securitatea fizică, securitatea infrastructurii, securitatea personalului, securitatea hardware și a aplicațiilor, securitatea proceselor de afaceri. Prin rezultatele identificate se aduc contribuții importante atât pentru mediul academic, cât și pentru organizațiile guvernamentale și neguvernamentale, pentru

înțelegerea și utilizarea corectă a termenilor, pentru reducerea incertitudinii și pentru abordarea corectă a terminologiei de specialitate, deoarece domeniul securității devine tot mai important, provocările fiind complexe și diverse.

Cuvinte cheie: *activ, disponibilitate, confidențialitate, cibernetică, informații, integritate, amenințare.*

1. Introduction

Currently, most of the economic, commercial, cultural, social and governmental activities and interactions of countries at all levels, including individuals, non-governmental and governmental organizations are carried out through the cyber environment [1], which has developed rapidly with the advent of the Internet. Along with the intense digitization attested at the international level, through the massive growth of electronic services, which are becoming more and more popular, the risks associated with cyber security also increase. The importance of cyber security increased substantially as a result of the use of the Internet between the 1980s and 1990s when the Internet became a quality resource for the consumer [2, pp. 33-39]. So that in the report presented by ENISA (The European Union Agency for Cybersecurity), for the years 2020-2021, cyber-attacks continued to grow, both in the diversity of the attack vectors used, as well as in the number of attacks and the impact they had [3]. The attack vectors were very diverse including: phishing and RDP attacks, DDoS campaigns, software supply chain, malware distribution, website compromise, vulnerable web applications, email-relevant vectors including phishing, spear phishing, whaling, business email compromises, etc.

The impact of the security breach can be estimated financially and in terms of the volume of compromised information. Thus, according to the annual report, produced by the Ponemon Institute and sponsored, analyzed, and published by IBM Security, which studied 550 organizations, from 17 countries and 17 industries, affected by data breaches that occurred between March 2021 and March 2022, reached the following results, the reported data breach losses are continuously increasing, so that from 2017 to 2022, they increased from $3.62 million to $4.35 million [4], with a percentage increase of about 12%. ENISA also published in July 2022, a worrying report on ransomware attacks, which have adapted and evolved to become more and more effective and cause more and more devastating damage. The monthly impact on the volume of data stolen, associated only with ransomware attacks, increased from 8 TB in May 2021 to 136 TB in June 2022 [5].

Cyber-attacks with a major impact on state infrastructures, at the international level, have devastating outcomes, and their number increases exponentially every year, The Center for Strategic and International Studies (CSIS), research organization dedicated to advancing practical ideas to address the world's greatest challenges, publishes troubling data on security incidents on a monthly basis. This data relates to the theft of personal data, such as the attack on Chinese police databases in July 2022, in which 1 billion records were compromised, this data was later put up for sale online; the attacks on the largest natural gas distributors from Greece in August 2022, caused a system outage; the DDOS attacks, which hit multiple public and private sector websites of the Romanian ministry of defense, border police, national railway company, and the OTP Bank in April 2022; the ransomware attacks in January 2022 on the Ukrainian government, which compromised the computers of government agencies [6]. Another cyber-attack, from October-November 2022, targeted the social media accounts of several officials from the Republic of Moldova, in the end all private conversations were made public.

With the start of the war in Ukraine and complicated geopolitical relations, the aggressiveness of attacks led by state-sponsored groups has essentially increased, according to the report presented by Microsoft, the most targeted industries in 2022 were Information Technology, NGOs and the education sector. The education and research sector were declared the most targeted in the report also presented by Check Point Software, multinational provider of software and combined hardware and software products for IT security [7]. A good example of this is the multiple attacks on China's Northwestern Polytechnical University, China accuses the U.S. National Security Agency (NSA), of infiltrating the university's electronic communications networks and stealing data [6].

In addition to all the challenges in the field, which are becoming more complex every year, an additional point of uncertainty is the interchangeable use of the term cyber security and information security. In the name of the governmental organizations of different countries, whose purpose and goals are similar, the terms cyber security and information security seem to have the same meaning, for example: Information Technology Service and Cyber Security (Republic of Moldova), the Federal Office for Information Security (Germany), National Cyber Security Center (Great Britain), Cybersecurity & Infrastructure Security Agency (USA), French National Agency for the Security of Information Systems (France). The same situation refers to the national state strategies, whose names also vary, as follows: Information Security Strategy of the Republic of Moldova 2019-2024, the German Cybersecurity Strategy, the Cyber Security Strategy of the United Kingdom, The National Cyber Strategy of the United States of America or The French national digital security strategy and not least the Cybersecurity Strategy adopted by the European Union in December 2020; using both terms, which creates uncertainty.

Based on the issues mentioned above, this research paper will try to identify the similarities and differences between cyber security and information security, to see if these two terms can be used interchangeably. The difference between this article and other articles that had the same purpose it to present different types of security, which are part of the umbrella term: cyber security; and based on the examples presented, to make a difference between cyber security and information security, which allows the correct use of the terms, when discussing issues related to national security, because the targets of cyber-attacks could be: individuals, states, connected devices and of course information, so it cannot be said that only information requires protection. Expanding the knowledge bases by presenting several definitions both taken from the academic and industrial environments, contributes to a better understanding of the studied terms and allows their use as appropriate. Also, the presentation of the trends of the last 18 years, in order to be able to analyze the international use of the terms in this field, contributes to highlighting the term cyber security versus other types of security.

The second section of the article is reserved for the literature review, the third section will contain various reflections and discussions on the research question, and the last section is reserved for conclusions.

2. Literature review

The definitions that will be presented in this section have been divided into those given by the academic environment, through books and published scientific articles and the industrial environment.

2.1 Information Security

Information security is a science that was tackled many centuries ago. The Encyclopedia Britannica records the use of cryptology in 400 BC by the Spartans, who used a device called the scytale for secret communication between military commanders [8]. During the 4th century, A.H. Aeneas Tacticus wrote a work entitled "On the Defense of Fortifications", one chapter of which was devoted to cryptography, making it the earliest treatise on the subject [8]. Since then, the importance of information security has been addressed and studied to meet the challenges of data protection, first for the classic form of data retention and analyzed also in the case of electronically stored data, with the evolution of modern technologies.

Over time, this term has been analyzed and addressed both by the industrial and the academic environment. Several definitions have been presented for a more accurate approach to the term. To achieve the purpose of this paper, an attempt will be made to analyze information security from different perspectives to understand whether it is an interchangeable term with cyber security or not. The fundamental principles of both terms are the same: confidentiality which ensures the access of authorized persons to information, integrity which refers to the accuracy of data and the availability of data to authorized users at the time of the request. In the meantime, some additional properties have been attributed to information security, by International Organization for Standardization (ISO), such as: authenticity, accountability, non-repudiation, and reliability [9]. ISO defines information security as: "the preservation of confidentiality, integrity and availability of information" [10], and extended the definition by adding the previously mentioned properties.

Several published scientific papers were identified, the purpose of which was to identify the meaning of the term information security, the purpose of which was similar to that of the present paper, namely to better understand the term to have a correct approach. Researchers from the Republic of Moldova define information security as a process that refers to "any information, IT or non-IT (books, reports, documents, etc.), on any traditional supports (paper, cloth, etc.) or electronic media (magnetic tape, CD, etc.), in any form (text, graphics, audio, video), communicated traditionally (written, oral, regular mail) or electronically (e-mail, chat, mobile phone)" [11]. The researcher's von Solms & van Niekerk, who over time have carried out several types of research in this field, define Information security as: "the protection of information, which is an asset, from possible harm resulting from various threats and vulnerabilities" [12], in other words, information is the asset that needs to be protected, information security is seen to be the process [12]. Having clearly defined the intention to identify a theory relevant to information security, Craig A. Horne et al. [13], concluded that no theory on information security was apparent in the literature, and there does not seem to be a way to measure when information has been protected enough. In his book, Whitman & Herbert define information security as" the protection of information and the systems and hardware that use, store, and transmit that information" [14, p.3]. According to the definition given by Ogbanufe, information security refers to the protection of digital and physical data against unauthorized access, disclosure, modification, or deletion [15].

Definitions were also identified in the most developed dictionaries and specialized organizations, so Oxford Dictionary defines information security as "ways of protecting information, especially electronic data, from being used or seen without permission". The National Institute of Standards and Technology (NIST) defines information security as "the protection of information and information systems from unauthorized access, use, disclosure,

disruption, modification, or destruction to provide confidentiality, integrity, and availability". Information security, according to the Escal Institute of Advanced Technologies (SANS Institute) which specializes in information security and cybersecurity training, refers to "the processes and methodologies which are designed and implemented to protect the print, electronic, or any other form of confidential, private and sensitive information or data from unauthorized access, use, misuse, disclosure, destruction, modification, or disruption" [16]. ISACA (Information Systems Audit and Control Association) treats information security as assurances within the enterprise that information is protected against disclosure to unauthorized users (confidentiality), improper modification (integrity), and non-access when required (availability) [17].

From the analysis of the definitions presented above, proposed by both researchers and specialized organizations, it can be stated that information security is particularly oriented towards information, as an important asset, which requires protection, regardless of its state: digital or printed. In other words, the main emphasis is placed specifically on information, ICT (information and communication technology) systems are targeted only from the perspective of protecting the information that resides in these systems as well as the physical environments in which the information could be located, such as cabinets, storage areas and buildings.

2.2 Cyber Security

The term cyber security appears in the literature and various studies, in two different forms. As a combination of 2 words "cyber security" or as a single word with the prefix "cybersecurity", in any case, it has 2 components, just like the term information security. In the case of information security, it is clear that it mainly refers to information, as the definitions in the previous section have also shown. In the case of cyber security, various discussions arise concerning the word "cyber". The analysis of specialized literature showed that the terms "Cybersecurity" and "Cyber Security" can be used interchangeably, because they have the same object of study, to demonstrate this, the definitions related to cyber security in this section will be given with the terms used by the authors.

The word "cyber" evolved from the scientific work of Norbert Wiener [9], in his work "Cybernetics; or control and communication in the animal and the machine", from 1948, which described the term cybernetics as the interaction between man and machine, the resulting system can create an alternative environment of interaction [18]. The etymological meaning of the term "cybernetics", according to Wiener's notes [18], is the Greek word "kybernetes", which was selected due to the sense of control over actions, comes from the Greek word "steersman", meaning "the one who steers" [19].

Meanwhile, multiple definitions have been given to the term "cyber". Thus, NIST defines cyber as "referring to both information and communications networks" [20]. Merriam-Webster Dictionary and Cambridge Dictionary, define cyber: as relating to or involving computers or computer networks (such as the Internet). To expand the contemporary definitions and make a connection with the initial definition given by Wiener, the definition of the term "cyber" must include the human factor, without which the interaction of systems and technologies would not exist, the interaction environment called by Wiener "cybernetic" does not can be formed, which directly contradicts the original definition of the term. The hypothesis is also supported by researchers Thomas Edgar & David Manz, in the book "Research Methods for Cyber Security", who claim that: "Cyber systems would not have a

function without human intervention" [4, pp. 33-39]. Cyber systems are part of cyberspace, they represent the physical manifestations of cyberspace activity, with which humans interact, and cyber security is part of every interaction [4, pp. 33-39].

The history of cyber security began much later, researchers refer to the first research published in 1970 [21], the paper was a technical report that analyzed and presented the security issues of computer systems and presented the comprehensive approach to security as a mix of hardware, software, communications, physical, personal and administrative controls, which require an implementation to ensure security [21].

Next, as in the previous section, various definitions for the term cyber security, addressed by researchers and the industrial environment, will be presented.

Researchers Bragaru & Briceag presented a definition adapted from the ISO/IEC 27032 standard, such that cyber security has the meaning of "...any security related to cyberspace, which is a complex environment that occurs in the process of interaction between people, software and internet services provided through technological devices or integrated networks" [11]. Edgar & Manz define: cyber security as a domain that includes the technologies, policies, and procedures to secure something within cyberspace [4, pp. 33-39]. Cyberspace was defined by NIST as: "a global domain within the information environment consisting of the interdependent network of information systems infrastructures including the Internet, telecommunications networks, computer systems, and embedded processors and controllers" [20].

Von Solms & van Niekerk define cyber security as "the protection of cyberspace itself, the electronic information, the ICTs that support cyberspace, and the users of cyberspace in their personal, societal and national capacity, including any of their interests, either tangible or intangible, that are vulnerable to attacks originating in cyberspace" [12]. The tangible asset is e.g. the protection of network devices, and the intangible assets are e.g. all the ethical aspects, vulnerable to cyber-attacks [12]. Researchers Li & Liu define cyber-security as" includes practical measures to protect the information, networks, and data against internal or external threats" [22]. Another definition characterizes cyber security as that which refers to the understanding of the problems that can arise as a result of various cyber-attacks and the development of defense strategies (e.g countermeasures) that preserve the confidentiality, integrity and availability of any digital and information technologies [23]. Researcher Shahid Alam defines cybersecurity as the security of the new digital age, also known as Cyberspace [24]. The CIA triad, analyzed from the perspective of cyber security refers to confidentiality as control over access, operations and disclosure of system elements; integrity as modification, manipulation, or destruction of elements in the system; availability as well as access to the services of elements in the system [24]. Researchers Schatz & al defined cyber security as "The approach and actions associated with security risk management processes followed by organizations and states to protect confidentiality, integrity and availability of data and assets used in cyberspace. The concept includes guidelines, policies and collections of safeguards, technologies, tools and training to provide the best protection for the state of the cyber environment and its users" [25].

In the industrial environment, cyber security has many definitions, which will allow us to analyze this term even more deeply. Cybersecurity is "the practice of protecting systems, networks and programs from digital attacks," according to Cisco, "these attacks are usually aimed at accessing, changing, or destroying sensitive information; extorting money from users; or interrupting normal business processes". IBM defines Cybersecurity as" the practice

of protecting critical systems and sensitive information from digital attacks. Also known as information technology (IT) security, cybersecurity measures are designed to combat threats against networked systems and applications, whether those threats originate from inside or outside of an organization". The International Telecommunication Union (ITU) defines cybersecurity as " the collection of tools, policies, security concepts, security safeguards, guidelines, risk management approaches, actions, training, best practices, assurance and technologies that can be used to protect the cyber environment and organization and user's assets. Organization and user's assets include connected computing devices, personnel, infrastructure, applications, services, telecommunications systems, and the totality of transmitted and/or stored information in the cyber environment" [26]. The cyber environment includes users, networks, devices, all software, processes, information in storage or transit, applications, services, and systems that can be connected directly or indirectly to networks [26]. ISO/IEC describes cybersecurity as the " preservation of confidentiality, integrity and availability of information in Cyberspace"[27]. ENISA defines that "cybersecurity shall refer to the security of cyberspace, where cyberspace itself refers to the set of links and relationships between objects that are accessible through a generalized telecommunications network, and to the set of objects themselves where they present interfaces allowing their remote control, remote access to data, or their participation in control actions within that Cyberspace" [28]. ISACA refers to the cybersecurity as "the protection of information assets by addressing threats to information processed, stored, and transported by internetworked information systems". In Gartner Glossary, cybersecurity is the combination of people, policies, processes, and technologies employed by an enterprise to protect its cyber assets.

The Oxford Dictionaries defines cybersecurity as: "the state of being protected against the criminal or unauthorized use of electronic data, or the measures taken to achieve this". The Merriam –Webster dictionary defines cybersecurity as: "measures taken to protect a computer or computer system (as on the Internet) against unauthorized access or attack".

From the definitions presented above, it can be concluded that cyber security is an umbrella term, which includes several types of security, the common property of which is the connection to communication networks, the services provided to users are electronic services because cyber security does not exist outside cyberspace. Figure 1 shows all the components of cyber security from the authors' perspective, as a result of analyzing the key words in the definitions presented above.

Figure 1. Cyber security - umbrella term.

Infrastructure Security refers to protecting electronic systems and responding to unauthorized incidents involving a country's infrastructure [29]. Physical security refers to all the devices, technologies and materials used for the perimeter, external and internal protection, which includes: doors, barriers, sensors, access control systems, etc. [30], to protect an enterprise's cyber assets. Business process security is based on the integration of security in the life cycle of business processes that use modern technologies, such as Cloud Computing [31]. Hardware security refers to the protection of devices that are part of cyberspace, that is, they are capable of being connected to communication networks. Information security as part of cyber security is based on the protection of electronic information. Personnel security refers to intangible cyber assets and ethical aspects of protecting human beings as part of the cyber environment. Computer security in the context of cyber security refers to computer systems that connect to telecommunications networks to communicate, excluding computers that are not connected to the network [12]. Network security refers to the security of network devices and connection media. Application security refers to the development of secure software. Communications security refers to "the protection of all communications media, technology, and content" [12].

3. Discussion

In cyber security, electronic information is an asset as important as devices, applications, telecommunications networks and people, is part of the cyber assets; more than that information can be seen as an important vulnerability in cyberspace. Whereas in information security, information is the primary asset that requires protection [12].

To identify the important differences between cyber security and information security, an attempt will be made to analyze the nature of attacks in cyberspace, to elucidate whether the ultimate target is information, or not.

When referring to the attack vectors described in the first section of this article, the vast majority are aimed at information security in cyberspace, that is, in this context cyber security and information security are interchangeable terms since the asset to be protected is electronic information, concerning such attacks as phishing, spear phishing, whaling, business email compromises, DoS and DDoS, malware distribution especially ransomware, website compromise, vulnerable web applications, etc. The variety of security threats in cyberspace is much more complex than those described here.

Definitions given by researchers, concerning the nature of cyber-attacks, are further confirmation of the differences between the terms. Researchers Bullock et al, define cyber security as addressing different cyber threats, such as cyber war, cyber terrorism, cyber-crime, cyber espionage and cyber sabotage [32], which do not necessarily target information, targets can also be other cyber assets: telecommunications networks, computers, IoT devices, and humans.

Cyberspace is very dynamic, so changes in cyberspace are happening all the time and at an accelerated pace, based on the constant development of communication and computing technologies [22]. The dependence of states on the cyber environment has greatly increased for communication and control over physical environments [22], unknown vulnerabilities appear with every change in modern technologies and provision of new electronic services.

In Table 1, several common cyber-threats that do not target information can be reviewed.

Threats on cyber assets except for information

No	Target	Vulnerable cyber assets	Vulnerable protocols	Attack Types	Recent cases
1.	IoT devices	Smart homes, smart offices, smart classroom or smart lab devices: smart lock, web cameras, home gateway, voice-activated home automation device, etc.	TCP UDP Non-standard Wireless protocols: Wi-Fi, Z-wave, XBee, Zigbee, Bluetooth, LoRA	DDoS Buffer Overflow Backdoor Installation Address Spoofing Man in the Middle Session Hijacking SQL Injection Routing Attack Malicious codes [33]	The Mirai malware, which mainly compromises IoT devices, diversified its number of variants by 57% in 2019 compared to the same period in 2018 [34].
2.	Person	Users' end devices, social media accounts	TCP UDP	Cyber bullying	The Cyberbullying Research Center in the USA has conducted 12 research studies from 2007 until now, the data is worrying, from May 2007 to April 2021, the percentage of teenagers aged 12-17 years, who were victims of cyberbullying have increased from 18.8% to 45.5%.
3.	End devices	Users' end devices and their resources, like: RAM, CPU, SSD/HDD, tec.	RDP HTTP	Malware	The Emotet malware in 2019 created 100,000 more botnets than in 2018, representing a 913% increase in the number of botnetized end-user devices [34].
4.	Digital media	Network devices: routers, switches, servers.	TCP UDP FTP DNS, etc.	Malware	Sony Pictures (2014), Netflix, ABC and HBO (2015). Unauthorized posting of media products in the public environment reduces the income of the production companies, because the public can access them for free, without having to go to cinemas or pay monthly subscriptions to gain access.
5.	Critical infrastructure	Network devices: routers, switches, servers; and network services	All network protocols	DDoS Malware	In June 2022, Hackers targeted Lithuania's state railway, airports, media companies, and government ministries with DDoS attacks [6]. The target of these attacks is oriented toward the interruption of critical national services.

So, where the target of cyber-attacks is information, the terms cyber security and information security can be used interchangeably. In the case of cyber-attacks targeting: IoT equipment, organizational business processes or critical state infrastructure, people or actions leading to financial harm (as in the case of digital media), cyber security is a different term. In the case of information printed and stored in physical format, the protection of this information relates to information security, but not to cyber security, as it is not a cyber asset.

To present how widely used the terms information security and cyber security are, as well as other terms such as network security, infrastructure security, or computer security, the Google Trends tool was used to analyze internationally, in the period 10.01.2004-10.01.2022, the results can be analyzed in figure 2.

From figure 2 it can be seen that Google searches show a line with an increasing trend for the term cyber security, especially since 2009, when it was used by the ex-president of the USA, Barack Obama, in his press release referring to the importance of cyber security [25]. Trend lines are used to reflect the total number of searches for a term compared to other terms. The other terms proposed for analysis, including information security, have negative trend lines.
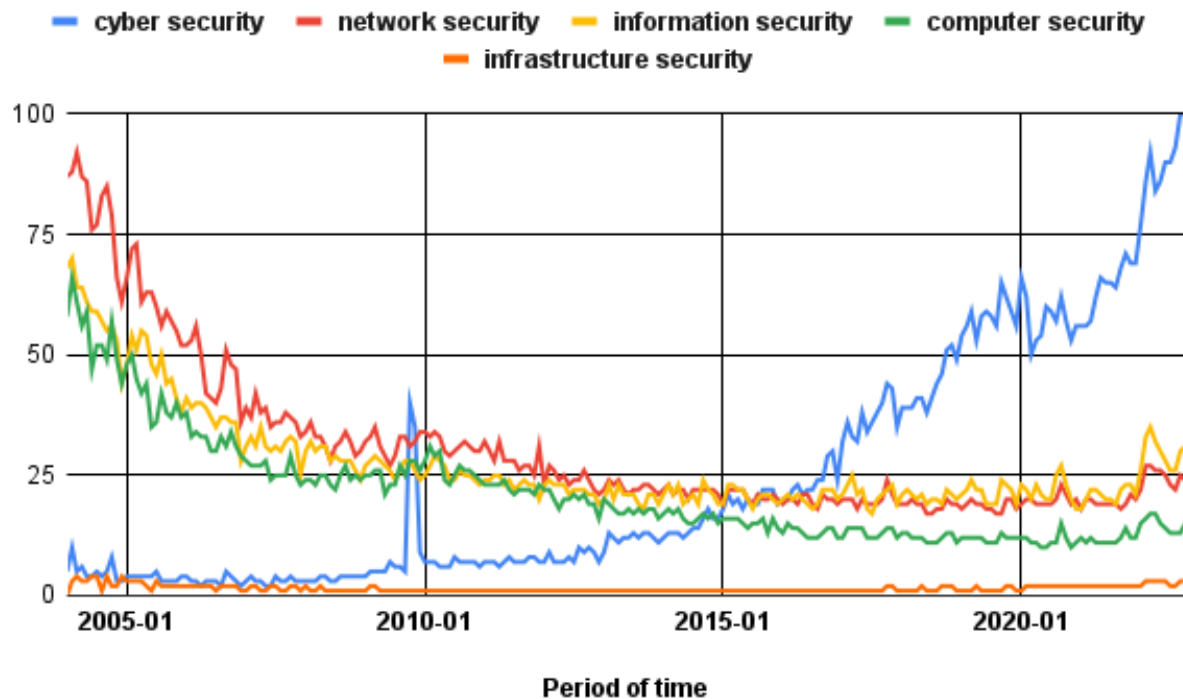
Figure 2. Google search trends for security terms 2004-2022 [35].

These results are indicative but important to identify trends [36]; but also, to present further proof of the hypothesis that cyber security represents an umbrella term for several types of security.

4. Conclusions

With the outbreak of the war in Ukraine, but also before, with the Covid-19 pandemic and the shift to remote work, cyber-attacks have steadily increased, with targets ranging from information and devices to people. Until now, states have each defined a strategy to be implemented to defend against cyber-attacks, where the terms information security and cyber security are used interchangeably.

But as discussed in this article, these two terms can be used in this way when the target of the attacks is information, when the target is various infrastructure services, cyber assets, or people, as a constituent part of cyberspace, they can no longer use the term information security, but rather the term cyber security. Several definitions have been presented, in order to be able to define these two terms as comprehensively as possible and to improve the knowledge base in this field, international trends can be used as an additional argument to justify the use of the term cyber security, as a term that covers more technological issues than information security.

Conflicts of Interest: The authors declare no conflict of interest.

References
1. Al-Ghamdi, M.I. Effects of knowledge of cyber security on prevention of attacks. *Materials Today: Proceedings* 2021. DOI: 10.1016/J.MATPR.2021.04.098.
2. Edgar, T.W.; Manz, D.O. *Research Methods for Cyber Security.* 1st ed., Elsevier, United States, 2017, 402 p.

3   Enisa Threat Landscape 2021. Available online: https://www.enisa. europa. eu/publications/ enisa-threat-landscape-2021 (accessed on 05.09.2022).

4. Cost of a Data Breach Report 2022. Available online: https://www.ibm. com /downloads/cas/3R8N1DZJ (accessed on 06.09.2022).

5. Enisa Threat Landscape for Ransomware Attacks. Available online: https:// www.enisa. europa.eu/ publications/enisa-threat-landscape-for-ransomware-attacks (accessed on 05.09.2022).

6. Center for Strategic and International Studies (CSIS). Significant Cyber Incidents. 2022. Available online: https://www.csis.org/programs (accessed on 12.09.2022).

7. Check Point Research. Cyber Security Report. 2022. Available online: https://www.checkpoint.com (accessed on 20.10.2022).

8. Encyclopedia Britannica. Available online: https://www.britannica.com/topic/ cryptology (accessed on 06.09. 2022).

9. Bay, M. What is cybersecurity? *French Journal for Media Research* 2016, 6, pp 1-28. ISSN 2264-4733.

10. ISO/IEC 27001: Information Security Management, International Organization for Standardization, Geneva, Switzerland, 2013. Available online: https://www.iso.org/isoiec-27001-information -security.html (accessed on 02.09.2022).

11. Bragaru, T; Briceag, V; Malcoci, V; Galaicu, V. Information Security vis-à-vis Informational Security. *Studia Universitatis Moldaviae* 2019, 2 (122), pp. 38–47. [in Romanian].

12. Von Solms, R.; Van Niekerk, J. From information security to cyber security. *Computer Security* 2013, 38, pp. 97–102. DOI: 10.1016/j.cose.2013.04.004.

13. Horne, C.A; Ahmad, A; Maynard, S.B. A Theory on Information Security. In: *The 27th Australasian Conference on Information Systems*, Wollongong, Australia, 2016.

14. Whitman, M. E.; Mattord, H.J. *Principles of Information Security*. 7th ed., Cengage Learning, 2021; p. 658.

15. Ogbanufe, O. Enhancing End-User Roles in Information Security: Exploring the Setting, Situation, and Identity. *Computer Security* 2021, 108, pp. 102340. DOI: 10.1016/ J.COSE. 2021.102340.

16. SANS Institute. Information Security Resources. Available online: https://www. sans.org/information-security (accessed on 06.09.2022).

17. Cooke, I. Doing More with Less. *ISACA* 2017, 5, pp. 6-9.

18. Wiener, N. *Cybernetics; or control and communication in the animal and the machine*. John Wiley, USA, 1948; 231 p.

19. Merriam-Webster.com Dictionary. Available online: https://www.merriam-webster.com/dictionary/steersman (accessed on 10.09.2022).

20. National Institute of Standards and Technology. Information Security. Available online: https://csrc.nist.gov/ glossary/term/cyberspace (accessed on 10.09.2022).

21. Ware, W. H. *Security controls for computer systems*. Rand Corp Santa Monica CA, USA, 1970, 80 p.

22. Li, Y.; Liu, Q. A comprehensive review study of cyber-attacks and cyber security; Emerging trends and recent developments. *Energy Reports* 2021, 7, pp. 8176–8186. DOI: 10.1016/J.EGYR.2021.08.126.

23. Jang-Jaccard, J.; Nepal, S. A survey of emerging threats in cybersecurity. *Journal of Computer and System Sciences* 2014, 80 (5), pp. 973–993. DOI: 10.1016/j.jcss.2014.02.005.

24. Alam, S. *Cybersecurity: Past, Present and Future*. Lambert Academic Publishing, Germany, 2022; pp. 2-8. DOI: https://doi.org/10.48550/arXiv.2207. 01227.

25. Schatz, D.; Bashroush, R.; Wall, J. Towards a More Representative Definition of Cyber Security. *The Journal of Digital Forensics, Security and Law* 2017, 12(2), pp. 53-74. DOI: 10.15394/jdfsl. 2017.1476.

26. ITU-T X.1205. Overview of cybersecurity. Available online: https:// www.itu.int/ en/ITU-T/study groups/com (accessed on 10.09.2022).

27. ISO/IEC 27000: Information technology − Security techniques − Information security management systems − Overview and vocabulary, "International Organization for Standardization," Geneva, Switzerland, 2018. Available online: https://www.iso.org/standard /73906 (accessed on 22.09.2022).

28. National Cybersecurity Strategies. European Network and Information Security Agency. Available online: https://www.enisa.europa.eu/publications/national-cyber-security-strategies-an-implementation-guide (accessed on 04.09.2022).

29. Abouzakhar, N. Critical Infrastructure Cybersecurity: A Review of Recent Threats and Violations. In: *12th European Conference on Cyber Warfare and Security* 2013, pp. 1–10.

30. Al-Fedaghi, S.; Alsumait, O. Towards a conceptual foundation for physical security: Case study of an IT department. *International Journal of Safety and Security Engineering* 2019, 9 (2), pp. 137–156. DOI: 10.2495/SAFE-V9-N2-137-156.
31. Farah, A.; Saida, B.; Mourad, O. C. On the security of business processes: classification of approaches, comparison, and research directions. In: *International Conference on Networking and Advanced Systems (ICNAS)*, 2021, pp. 1–8. DOI: 10.1109/ICNAS53565.2021.9628908.
32. Bullock, J. A.; Haddow, G. D.; Coppola, D. P. *Cybersecurity and critical infrastructure protection. Introduction to Homeland Security*. Butterworth-Heinemann, 2021; pp. 425–497. DOI: 10.1016/B978-0-12-817137-0.00008-0. ISBN: 978-0-12-817137-0.
33. Alexei, A.; Alexei, A. Analysis of IoT security issues used in Higher Education Institutions. *International Journal of Mathematics and Computer Research* 2021, 9 (5), pp. 2277-2286. DOI: 10.47191/ijmcr/v9i5.01.
34. Enisa Threat Landscape Botnet 2020. Available online: https://www.enisa. europa. eu/ topics/threat-risk-management/threats-and-trends/etl-review-folder/etl-2020-botnet (accessed on 12.09.2022).
35. Data source: Google Trends. Available online: https://trends.google.com/trends/explore?date=all&q=cyber%20security,network%20security,information%20security,computer%20security,infrastructure%20security (accessed on 20.09.2022).
36. Choi, H.; Varian, H. Predicting the Present with Google Trends. *Economic Record* 2012, 88, pp. 2–9. DOI: 10.1111/j.1475-4932.2012.00809.x.

Publisher's Note: JES stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.

Submission of manuscripts: jes@meridian.utm.md

OPEN ACCESS   CC BY

THIN FILMS OF COPPER OXIDE NANOSTRUCTURED VIA RAPID THERMAL PROCESSING

Cristian Lupan [1], ORCID: 0000-0003-2268-6181,
Vasilii Crețu [1*], ORCID: 0000-0002-3096-5976,
Abhishek Kumar Mishra [2*], ORCID: 0000-0002-6129-5653,
Nicolae Magariu [1], ORCID: 0000-0001-7006-3443,
Nicolae Manciu [1], ORCID: 0000-0003-2993-6593,
Nicolai Ababii [1], ORCID: 0000-0001-5046-8611,
Soni Mishra [3], ORCID: 0000-0003-3511-8319,
Serghei Railean [1], ORCID: 0000-0001-5478-4363,
Lukas Zimoch [4], ORCID: 0000-0002-4860-065X,
Vardan Galstyan [5,6], ORCID: 0000-0002-0615-3097,
Sandra Hansen [4], ORCID: 0000-0001-5906-8075,
Thierry Pauporté [7], ORCID: 0000-0001-5906-8075

[1] *Department of Microelectronics and Biomedical Engineering, Center for Nanotechnology and Nanosensors, Technical University of Moldova, 168 Stefan cel Mare Blvd., Chisinau, Republic of Moldova*
[2] *Department of Physics, Applied Science Cluster, School of Engineering, University of Petroleum and Energy Studies, Dehradun 248007, Uttarakhand, India*
[3]*Department of Physics, Graphic Era Hill University, Dehradun 248002, India*
[4] *Department of Materials Science, Chair for Functional Nanomaterials, Faculty of Engineering, Kiel, Kaiserstraße 2, D-24143 Kiel, Germany*
[5] *Sensor Laboratory, Department of Information Engineering (DII), University of Brescia, Via Valotti 9, 25133 Brescia, Italy*
[6]*Department of Engineering "Enzo Ferrari", University of Modena and Reggio Emilia, Via Vivarelli 10, 41125 Modena, Italy*
[7] *Institut de Recherche de Chimie Paris-IRCP, CNRS – Chimie ParisTech, 75231 Paris Cedex 05, France*
Corresponding authors: Vasilii Cretu, vasilii.cretu@mib.utm.md,
Abhishek Kumar Mishra, *akmishra@ddn.upes.ac.in*

Abstract. Nanostructured copper oxide films were obtained by the method of chemical synthesis from solutions (SCS) and exposed to post-growth rapid thermal processing (RTP) in air at different temperatures to study the influence of annealing temperature on morphological, chemical, structural and sensing properties. Controlled modification of surface morphology, in the particular size of nanostructures, crystallinity and phase can be achieved by RTP, which is preferred due to saving of energy budget nowadays. Detailed physico-chemical analysis of the films was performed using the scanning electron microscopy (SEM), X-ray diffraction (XRD), Raman and energy dispersive X-ray (EDX) techniques. Sensors based on the copper oxide nanostructured films after RTP for 30 s only were tested with 100 ppm hydrogen gas at an operating temperature range from 250 ºC to 350 ºC. The difference

in the response to 100 ppm hydrogen gas of the sensors based on thermally processed films at different temperatures was determined. We also noted that the change in the response of the sensing structure is correlated with its surface morphology controlled by RTP regime with a short duration. A detection mechanism to hydrogen gas has been proposed as well.

Keywords: *copper oxide, nanostructures, SCS, rapid thermal processing, DFT.*

Rezumat. Peliculele de oxid de cupru nanostructurat au fost obținute prin metoda sintezei chimice în soluții (SCS) și expuse după depunere la tratament termic rapid (RTP) în aer la diferite temperaturi pentru a studia influența temperaturii tratamentului termic asupra proprietăților morfologice, chimice, structurale și senzoriale. Modificarea controlată a morfologiei suprafeței, în particular a dimensiunii nanostructurilor, cristalinității și fazei au fost obținute cu ajutorul RTP, care este preferat datorită necesității consumului de energie în prezent. Analiza fizico-chimică detaliată a peliculelor a fost realizată cu ajutorul microscopului electronic de baleiaj (SEM), difracție cu raze X (XRD), Raman și tehnici pe baza dispersiei razelor X (EDX). Senzorii pe baza peliculelor de oxid de cupru nanostructurat după RTP numai de 30 s au fost testate la 100 ppm hidrogen la temperaturi de operare între 250 °C și 350 °C. Diferența în răspuns la 100 ppm hidrogen a senzorilor bazați pe peliculele tratate termic rapid la diferite temperaturi au fost determinate. Noi am observat că modificarea în răspuns a structurilor senzoriale corelează cu morfologia suprafeței controlată de regimul RTP cu durată foarte mică. De asemenea, un mecanism de detecție a hidrogenului a fost propus și confirmat prin calculele DFT.

Cuvinte cheie: o*xid de cupru, nanostructuri, SCS, tratament termic rapid, DFT.*

1. Introduction

Copper oxides are non-toxic and can be synthesized by various simple and low-cost methods. The low-dimensional structures of copper oxides (CuO and Cu_2O) with high surface-to-volume ratio have been used in various fields such as photovoltaics, gas sensing of volatile organic compounds and toxic gases, lithium-ion batteries, electrochromic devices, super-capacitors, field emission devices, non-enzymatic biosensors for glucose detection, antimicrobial applications, microfluids and photo catalysis [1, 2].

Copper oxide can have two oxidation states and three crystalline phases with distinct parameters: pure CuO or Cu (II) cupric oxide with monoclinic crystalline structure, pure Cu_2O or Cu (I) oxide with cubic crystalline structure oxide, and Cu_4O_3 paramelaconite, a mix of Cu (II) and Cu (I) with tetragonal crystalline structure. The more stable phases at relatively high temperature (up to 500 ºC, which is suitable for gas sensing applications) and ambient pressure are CuO and Cu_2O [1, 2].

Various methods for the growth of copper-based nano- and micro-oxide materials were developed, such as chemical based synthesis methods [3], thermal decomposition [1-3], thermal oxidation of Cu metal [4], etc. The controlled post-growth modification of surface morphology, in particular size of nanostructures, crystallinity and phase can be achieved by thermal processing at different temperatures and duration. Nowadays, rapid thermal processing is preferred due to energy efficiency nowadays. For example, by rapid thermal processing of Cu_2O films the non-planar CuO/Cu_2O with unique gas sensing properties heterostructures was reported [2,4]:

$$2Cu_2O + O_2 \rightarrow 4CuO \tag{1}$$

The choice of methods for obtaining the detector surface is an important step to realize the gas detectors with necessary properties. The morphology of the obtained surface determines the ratio of surface area to volume, which determines the amount of gas that reacts at a given time, which in turn determine the conductivity of the sensor with and without the gas atmosphere. The surface morphology significantly affects not only the sensitivity of the sensor, but also indicative characteristics such as the time-dependent response of the sensors, the long-term stability, the selectivity and the optimal operating temperature [5].

Homogeneous detection surfaces with a well-defined structure can be obtained by direct synthesis of the Cu_2O oxide phase, or by the temperature reduction reaction of Cu_2O to CuO. Copper oxide in the CuO phase is a semiconductor with a *p*-type electrical conductivity and a bandwidth of approximately 1.1-2.0 eV, which has been widely implicated in VOCs gas detection tasks [6]. In this context, single crystalline CuO nanowires demonstrated high performance for the detection of ethanol vapors, even at room temperature [7]. It is also known that copper oxide is an excellent catalyst for volatile organic compounds, and thus they were used for the detection of hydrogen, mainly by surface metallization, due to its very active surface [7]. The oxidative dehydrogenation process and mechanism of ethanol (C_2H_5OH) molecules and oxidation of acetaldehyde molecules were recommended and illustrated in previous work [8]. Experimental studies demonstrated that the morphology of the oxide surface affects the selectivity of the sensor. Thus, morphology control by post-deposition rapid thermal annealing (RTA) for a short duration is an interesting topic for research. Here, the hydrogen gas sensing properties of prepared structures based on CuO/Cu_2O films have been studied and presented in detail. Density Functional Theory (DFT) based calculations have presented insights into H_2 gas sensing on CuO surfaces.

2. Experimental
A. Synthesis of CuO/Cu₂O and Characterization

Copper oxide (Cu_2O) layers were synthesized on glass/quartz substrates by growth from chemical solutions, which was described in detail in a previous paper [8]. The growth solution is mixed on the basis of copper thiosulphate, as a cationic precursor, in which 1 M of Cu sulphate pentahydrate ($CuSO_4 \bullet 5H_2O$) and 1 M Na thiosulfate ($Na_2S_2O_3 \bullet 5H_2O$) were mixed. The complex solution is diluted to 0.1 M Cu ion using deionized water (DI). The complex anion solution consists of 2 M NaOH. Single SCS cycle of Cu_2O thin film deposition can be described as follows: immersion of the glass in anion solution for adsorption of hydroxyl groups, then immersion of the same glass in the Cu^+ ions solution for reaction with OH^- groups. An experimentally elaborated graph is used to control the film thickness. The optimal film thickness was determined to be 0.7 µm [8]. After chemical deposition of the nano-layers, rinsing in DI water takes place and finally exposed in a stream of hot air (~ 150 °C) for 1 min.

The rapid thermal processing (RTP) method is used to change the surface morphology and the crystal phase composition of the copper oxide nanostructured films. The samples were exposed for 30 s at different temperatures (650 °C, 700 °C and 750 °C) by using a high-intensity infrared emitting *lamps*, followed by deposition of gold contacts for electrical connection and studies of gas sensing properties. This short period of RTP allows us to save

the thermal budget for technological processes significantly if compare with previous works [9, 10].

The electrical measurements of the sensors were carried out by the two-probe method in a homemade installation. The electric current as a function of the gas pulses applied to the surface of the nanostructured films was recorded using the Keithley 2400 sourcemeter. The electrical resistance was measured under the ambient atmosphere and under influence of the hydrogen gas at relative low concentration (100 ppm). The relative humidity was the same as in the ambient (40%), and was measured using an electronic hygrometer [9].

Hydrogen was chosen as a test gas with a flow rate of 500 sccm (ml/min), generated using a pre-calibrated mass flow controller [10] and the gas concentrations were controlled using the following expression [11, 12]:

$$C(ppm) = \frac{C_1 * F_{gas}}{F_{tot}}, \tag{2}$$

where: C is the necessarily gas concentration; C_1 is the initial concentration *(ppm)* of the tested gas; F_{gas} is the flow of the H_2 gas; and F_{tot} is the final flow of the mixed gases.

B. Computational Methodology

We used the Vienna ab initio simulation package (VASP) employing a plane-wave basis set [13-16], the projector augmented wave (PAW) method for depicting the ions and electrons interactions [17], and Perdew– Burke–Ernzerhof functionals [18, 19] to evaluate the nonlocal exchange -correlations. We used DFT+U method to treat strong correlations through the Hubbard model in the Dudarev formalism [20] for modelling copper oxides, as this was found to predict accurate structural, electronic magnetic as well as surface properties in our earlier works [21, 22]. Here, we used a more accurate method of dispersion corrections, i.e. DFT-D3 approach by Grimme [23], while all other details related to computations remain unchanged [21, 22].

3. Results and Discussion

A. Morphological characterization

After the post-deposition rapid thermal processing (RTP), the color of the deposited film has visually changed. Figure 1 shows the SEM image of RTP copper oxide films at 650 °C for 30 s. The scale bars on the SEM images are 200 nm. It can be demonstrated that the surface morphology of the film shows a conglomeration of interconnected nano-crystallites with random shape with a diameter of about 200 nm. It can be observed that in the SEM image of the nanostructured copper oxide film with RTP at 700 °C for 30 s, the nano-crystallites have the same structure in the environment, but locally agglomerations of nano-crystallites with a diameter of 100-150 nm are observed. The edge of nano-crystallites can be observed. In a previous work [24], it was demonstrated that the processing at a higher temperature than 700 °C with a treatment time of 60 s will lead to the formation of crystals highlighted more strongly, which achieve a defined form. Thus, we can assume that differentiation of the surface morphology is dependent on both the RTP temperature and time.

In figure 2 (a) the SEM image of the copper oxide film with rapid thermal processing at 700 °C for 30 s is depicted, showing the morphology of the film. In figure 2 (b), the energy dispersive X-ray (EDX) mapping analysis of copper oxide was performed; showing roughly even distributed copper (Cu) and oxygen (O) atoms.
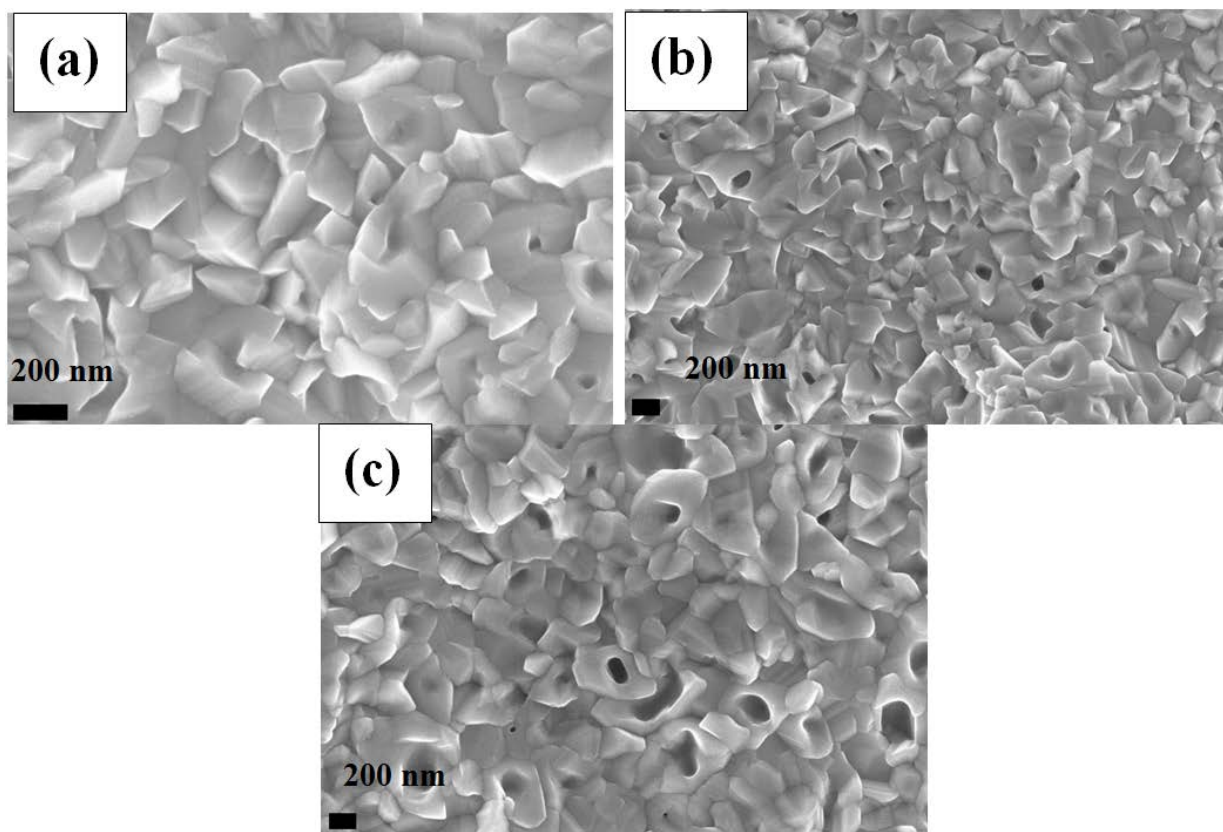
Figure 1. SEM image of Cu_2O/CuO thin nano-layers synthesized by SCS method and hermally processing after deposition at: (a) 650; (b) 700; (c) 750 °C by RTP method for 30 s.

B. Energy dispersive X-ray characterization
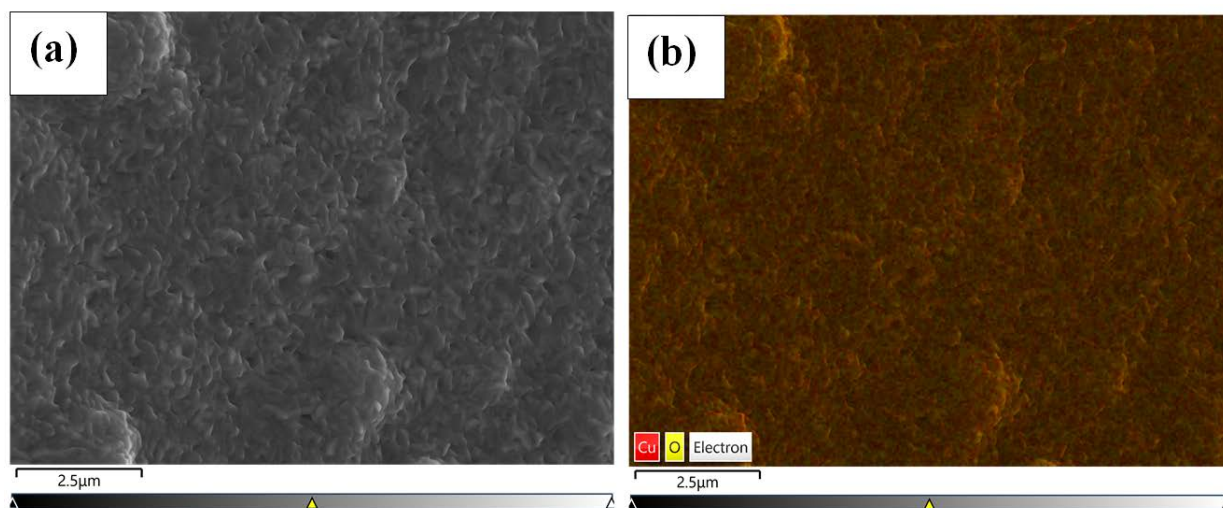
Figure 2. Energy dispersive X-ray (EDX) mapping analysis of copper oxide (CuO) film with rapid thermal processing at 700 °C (R700): (a) SEM image of the film for EDX mapping; (b) EDX mapping of the detected elements.

In figure 3, the individual EDX map of the detected elements copper and oxygen are presented, showing that these are distributed evenly on the surface. In figure S1, a line-scan EDX mapping of the copper oxide (CuO) film with RTP at 700 °C (R700), and results of the measurements are summarized in table 1, showing the atomic % of detected elements. On this film oxygen at% are slightly higher than the ones of copper.
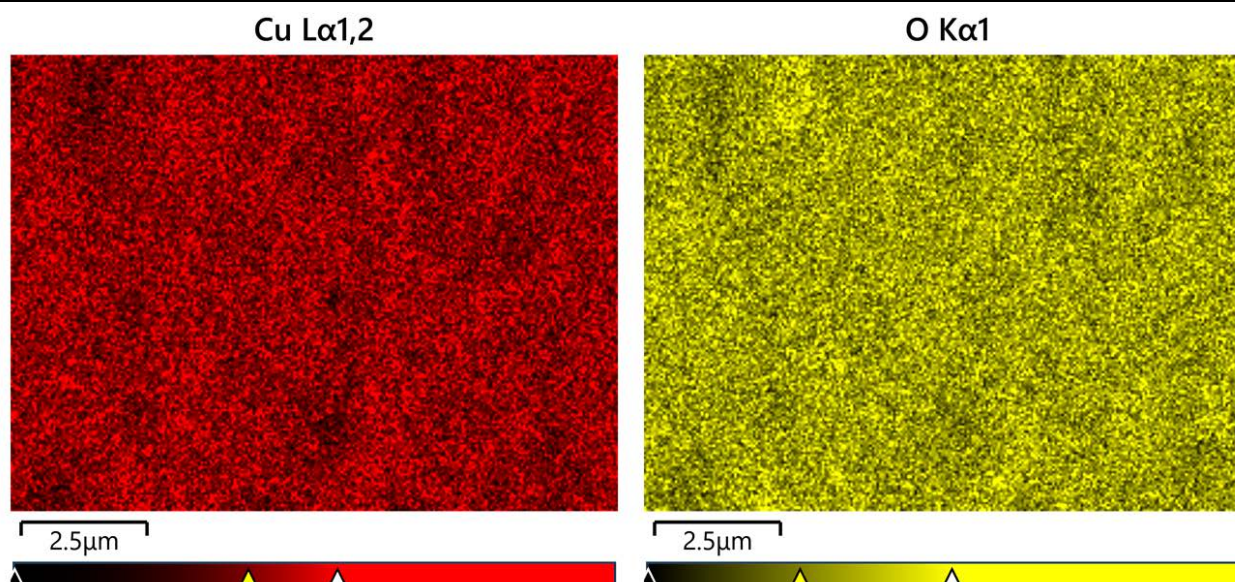
Figure 3. EDX mapping of individual elements on the copper oxide (CuO) film with rapid thermal processing at 700 °C (R700) for 30 s.

Table 1

Distribution of detected elements on the copper oxide (CuO) film with rapid thermal processing at 700 °C (R700 sample set)

Element	At%
O	54.72%
Cu	45.28%
Total	100%

C. XRD and Raman part characterization

X-ray diffraction (XRD) and micro-Raman spectra were used to analyze the crystal structure of copper oxide films at different rapid thermal processing temperatures. Figure 4 (a) shows the XRD patterns of CuO films with rapid thermal processing at 650 °C (curve 1), 700 °C (curve 2), and 750 °C (curve 3).
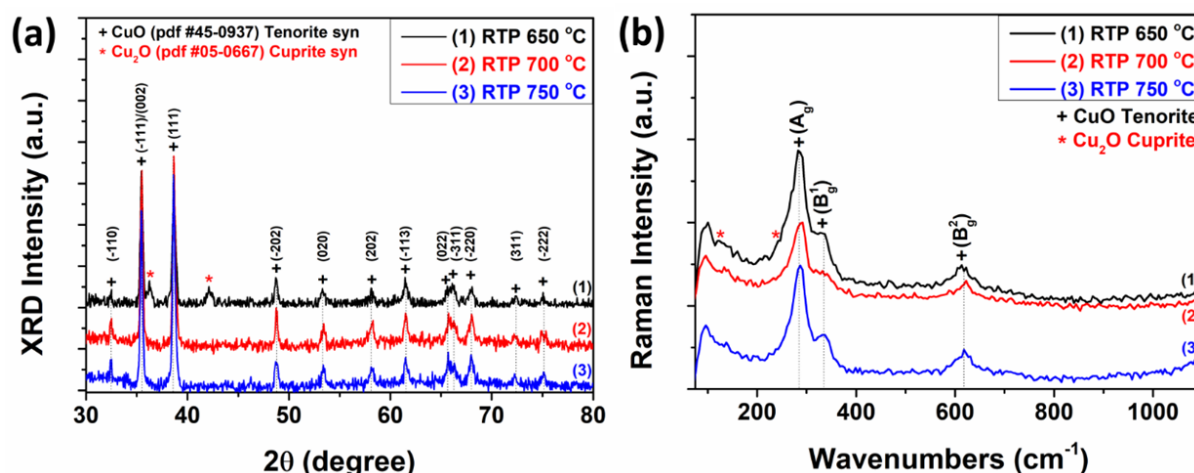
Figure 4. (a) XRD patterns and (b) micro-Raman spectra scanned of CuO films with rapid thermal processing of 650 °C (curve 1), 700 °C (curve 2), and 750 °C (curve 3).

From the Figure 4 (a) it can be seen that in the case of samples after RTP of 650 °C (curve 1), reflections at 2θ of 36.25° and 42.1° are assigned to the Cu_2O (Cuprite) phase, and at temperatures above of 700 °C (curve 2), and 750 °C (curve 3), these reflections disappear, which would mean the transition of the phase of Cu_2O (Cuprite) in CuO (Tenorite) phase. Cu_2O has a cubic crystalline structure with a space group $T_h^2 - Pn3[65W]$ or $O_h^4 - Pn3[72P]$, and the parameters of the unit cell are: a = 4.27 Å at $p \approx 0$ GPa, a= 4.18 Å at $p \approx 10$ GPa [82W], [10]. For all RTP regimes, the reflections (*hkl*) attributed to the CuO phase (Tenorite) were found, which correspond to the indices of the crystal lattice (-110), (-111)/(002), (111), (-202), (202), (-113), (022), (-311), (-220), (311), (-222) and (-131) Miller planes at 2θ values of 32.5°, 35.45°, 38.65°, 48.7°, 53.3°, 58.1°, 61.45°, 65.75°, 66.1°, 68.05°, 72.4° and 75.05°, respectively. CuO has a crystalline structure in monoclinic symmetry with the *C2/c* space group [10, 25] and lattice constants a = 4.684 Å, b = 3.423 Å, c = 5.129 Å, and β = 99.54° [10, 26].

To confirm XRD data, micro-Raman studies were performed. Figure 4 (b) shows a micro-Raman spectrum of CuO films with RTP at 650 °C (curve 1), 700 °C (curve 2) and 750 °C (curve 3). From Figure 4 (b) it can be seen that for samples with RTP at 650 °C (curve 1) five peaks are observed, where the three at the wavelength of about 287, 331 and 617 cm^{-1}, can be attributed to the CuO phase, and two peaks at the values of about 124 cm^{-1} and 242 cm^{-1} can be ascribed to the Cu_2O phase [10]. Also, from Figure 4 (b) it can be seen that at RTP of 700 °C (curve 2), and 750 °C (curve 3), the Cu_2O phase disappears, which would demonstrate the transition of the Cu_2O phase to the CuO crystal phase at temperatures higher than 650 °C for 30 s, which is in agreement with XRD data.

D. Gas sensing properties

For the investigation of the gas sensing properties of the CuO/Cu_2O nanostructured films, the meander-shaped gold contacts with an inter-distance of 0.9 mm were deposited on the surface of the samples. The response was investigated in atmospheric air towards 100 ppm of hydrogen gas at different temperatures. The response to gas was determined as the ratio of the sample electrical currents under exposure to ambient (I_{air}) and under exposure to hydrogen gas (I_{gas}):

$$S = \frac{I_{air}}{I_{gas}} \tag{3}$$

In figure 5 is compared hydrogen gas responses of the copper oxide sensors with different rapid thermal processing temperatures at different operating temperatures. The concentration of hydrogen gas is 100 ppm. The operating temperatures are 250 °C, 300 °C and 350 °C. At these three operating temperatures, all the investigated samples showed a high response to hydrogen gas after RTP regime. At lower operating temperatures the sensors show negligible responses to hydrogen gas, and therefore were not included.

At operating temperatures of 300 °C, the maximum response to the hydrogen atmosphere is observed for all three types of investigated sensor structures. Thus, at the indicated optimal operating temperature the samples with RTP at 650 °C, 700 °C, and 750 °C have a response of 3, 3.7, and 3.5, respectively.

The dependence of the gas sensitivity on the RTP temperature and the operating temperature (OPT) is obvious. From Figure 5 we can conclude that, in the case of H_2 measurement, the highest response demonstrates the thin films subjected to RTP at 700 °C and OPT 300 °C.
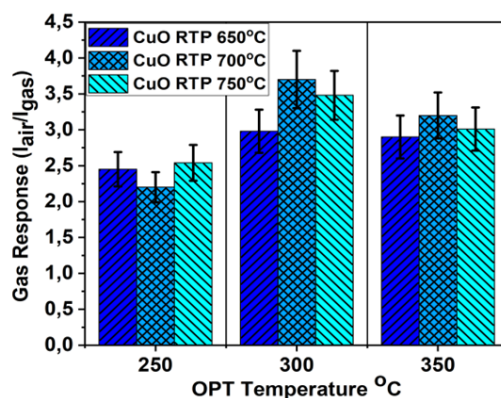
Figure 5. Responses of copper oxide nanostructured films with RTP at 650 °C, 700 °C and 750 °C in air to 100 ppm of H_2 gas versus operating temperature.

The higher gas response for samples with RTP of 700 °C can be attributed to the transition from Cu_2O phase to CuO phase in the RTP temperature range of 650 °C–700 °C by controlling the thickness ratio between CuO and Cu_2O layers, demonstrated by Lupan et al. [8].

Figure 6 shows the dynamic H_2 response of Cu_2O/CuO nanostructured films with RTP at 700 °C at an OPT of 300 °C. Two pulses of H_2 gas were applied to verify the repeatability of the device structures. All pulses indicate a response of ~ 3.7 with an error rate of no more than 10%. The response time from the application of the gas atmosphere to the gas response of 3.3 is ~ 14 s. Recovery of the sample from the maximum value up to the ratio of 1.37 takes place in 20 s.

It was also noted that the response curve does not return to the unit ratio in a reasonable time, probably due to the adsorption/desorption of gas species that takes place slowly on the surface of the films during hydrogen exposure.
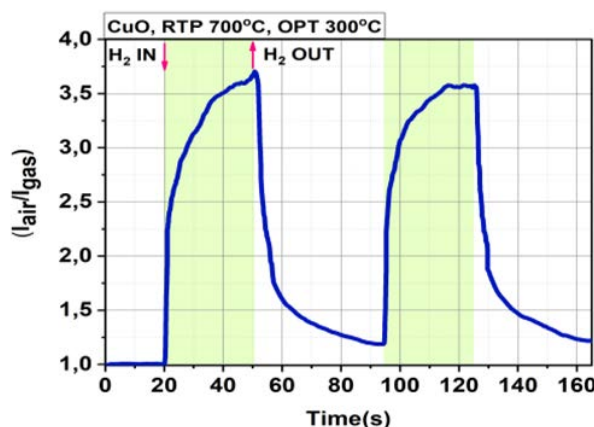
Figure 6. Dynamic hydrogen gas response of Cu_2O/CuO nanostructured films with rapid thermal processing at 700 °C for 30 s and working temperature OPT of 300 °C.

E. Proposed Hydrogen Gas Sensing Mechanism

The value of the response of CuO is mainly dependent on the type of surface vacancies, morphology and different impurities formed on the top of the nanostructured film [27]. Thus, we can say that surface phenomena play a critical role in the gas detection mechanism for the semiconductor metal oxides with *p*-type conductivity, like CuO.

Oxygen species at the crystal facets are absorbed in molecular form O_2^- or in atomic form O^-/O^{2-}. At temperatures below 200 °C, the majority species on the oxide surface are O_2^- [28]:

$$O_{2(g)} \rightarrow O_{2(ad)} \tag{4}$$

$$O_{2(ad)} \rightarrow O_{2(ad)}^{-} + h_{(lattice)}^{+} \tag{5}$$

At the same time, for OPT above 200 °C, the dominant species are O^{-} or O_2^{-} [28]:

$$\frac{1}{2}O_{2(ad)} \rightarrow O_{(ad)}^{-} + h_{(lattice)}^{+} \tag{6}$$

Adsorbed O^{-} or O_2^{-} generates the surface states with acceptor effect and captures electrons (e^{-}) from the valence band, and as a result the concentration of the holes (h^{+}) increases and the electrical resistivity decreases. For *p-type* semiconducting metal oxides at the surface, the holes accumulation layer is generated. The thickness of this region is directly proportional to the Debye length (λ_D) [8]. The conduction of electrical current takes place through the region of the accumulation layer, while the internal site of the oxides have a higher electrical resistance. The following reactions occur when semiconductor oxide is applied to the atmosphere with hydrogen (we assume that $h^{+} + e^{-} = Null$) [28]:

$$H_{2(g)} + O_{(ads)}^{-} + h^{+} \rightarrow H_2O_{(g)} + Null \tag{7}$$
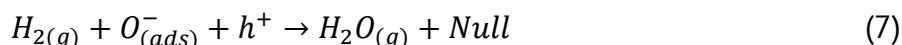
After placing the *p-type* semiconductor oxides in the atmosphere with the presence of H_2 gas, the electrical and chemical interaction between the O^{-} species and the hydrogen will take place. With the release of e^{-}, the e^{-} recombines with the surface h^{+}, which leads to the decrease in the holes accumulation layer and accordingly to lower electric current flowing through the crystallites.

The generated temperature at the surface of the films is one of the most important conditions of its work, it ensures the activation of adsorption-desorption processes that take place on the surface of the sensitive film of metal oxide. The operating temperature largely determines the selectivity and sensitivity of the *p-type* semiconducting metal oxides to a certain gas. This is dependent on the different activation energies of the adsorption processes for various gas species.

At an operating temperature lower than the optimal operating temperature, the desorption of the resultant reaction elements between the oxygen adsorbed on the surface and the reducing gas will be slower or will be absent at all, so that the surface will not recover for the subsequent adsorption of the gas. At a temperature higher than the optimal OPT, the gas does not absorb on the surface of the semiconductor oxide. Vacancies in the structure of the crystal facets of oxide have gas adsorption centers that allow changing in the response.

Another method of controlling the sensitive and selective properties is to change the surface morphology. It modulates the response according to the surface/volume principle. This approach is effective for both types of conductivities [8].

Post-growth thermal process at different temperatures allows the formation of various morphologies with the formation of crystallites with different dimensions and shapes that affect and permits control on the sensing mechanism.

Furthermore, it has been determined that the morphology with a smaller diameter of crystallites has better gas detection properties [28, 29]. The smaller diameter of the crystallites has a higher surface-to-volume ratio which results in the concomitant reaction of a larger number of *oxygen* species, which ultimately leads to improved gas detection properties [28].

F. DFT Calculations: H_2 gas molecule interaction with copper oxides

The exact oxidation state of copper oxide is challenging to determine as it consists of mixed phases of Cu_2O and CuO. The (111) plane has been found to be the most dominant plane in Cu_2O as well as in CuO morphologies [21, 22]. In our recent works based on DFT+U Hubbard parameter approach simulations, we found that $U_{eff} = 7$ eV value is appropriate to model both phases of copper oxide. Here, we used the same methodology to model the copper oxide surfaces, where we kept 3 top layers fixed to their bulk positions in 5 layers model of Cu_2O, while top two layers were fixed in 4 layers model of CuO (111) surface.

The Cu_2O (111) top surface consists of saturated and coordinatively unsaturated copper atoms, where most of the surface reactions take place at these coordinatively unsaturated copper atoms, acting as Lewis acid sites [30]. Here, H_2 molecule interacts with the surface through this copper atom as shown in figure 7, with interaction energy of -51.4 kJ/mol, making bonds of about 1.61 Å with the surface.
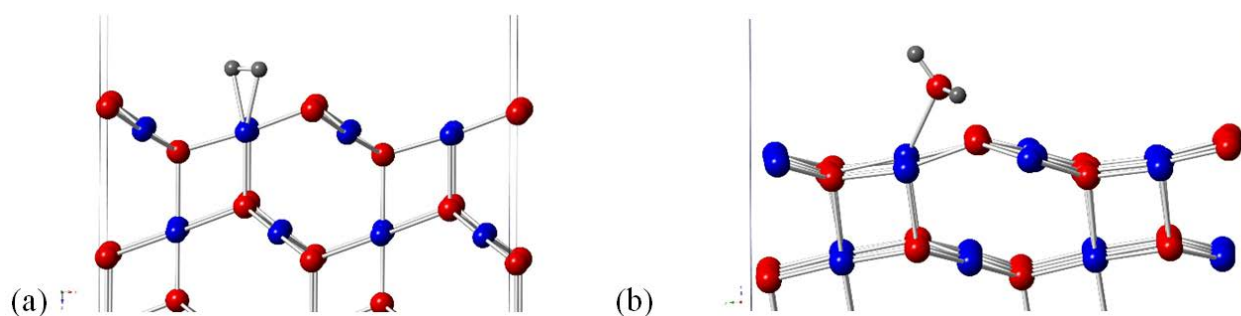
Figure 7. (a) H_2 molecule interaction with (a) Cu_2O (111) surface and (b) CuO (111) surface. Red and blue color balls indicate O and Cu atoms, while H atoms are represented by small grey color balls.

We next investigate the behaviour of H_2 gas molecule over CuO (111) surface, consisting of 3- and 4-coordinated copper and oxygen atoms in the top surface layer. As soon as H_2 gas molecule comes in contact with most exposed surface oxygen atoms, it forms molecularly adsorbed water molecule over the surface as shown in figure 7b. Here, H_2 molecule exhibits interaction energy of -55.3 kJ/mol, resulting in the reduction of the copper oxide surface.

We found that H_2 gas molecule interacts with both copper oxides with almost the same binding energy, whereas water molecules are expected to be formed as a result of H_2 gas molecule interaction with pristine CuO phases, resulting in the reduction of copper oxides.

4. Conclusions

Hydrogen is extensively used in research laboratories, biomedical systems, and auto transportation areas. In this work, the copper oxide nanostructured films were developed and investigated as hydrogen gas sensors. The aspect of controlling the response of the sensor structure by modifying the operating temperature of the film in the range of 250-350 °C and changing the post-deposition RTP temperature in the range of 650-750 °C for only 30 s has been demonstrated.

It was established that for the detection of relative low concentrations of hydrogen (100 ppm), the most optimal rapid thermal processing is at a temperature of 700 °C for 30 s. In this case, the structure showed a gas response of 3.7 at the operating temperature of 300 °C with the response and recovery times of about 14 s and 20 s, respectively.

It was observed that the film sensitivity is changed depending on its surface morphology, determined by the RTP temperature and time. EDX, XRD and Micro-Raman investigations demonstrated the transition of the film from the tenorite-cuprite mixed phases structure to the film with a surface tenorite structure at 700 °C for 30 s. Compared to the conventional thermal processing of CuO films in furnace described in [31], there is a significant enhancement in hydrogen sensitivity and an enormous energy budget saving. The proposed gas detection mechanism of oxides has been expressed. DFT found that H_2 gas molecule interacts with both copper oxides with almost the same binding energy. We consider that these results will be of a big interest for the elaboration of nanotechnology for low-energy-budget-oxide based detectors by a cost-effective approach and to develop a sensing mechanism.

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Zoolfakar, A.S.; Rani, R.A.; Morfa, A.J.; O'Mullane, A.P.; Kalantar-Zadeh, K. Nanostructured copper oxide semiconductors: A perspective on materials, synthesis methods and applications. *J. Mater. Chem. C* 2014, 2, pp. 5247–5270.

2. Zhang, Q.; Zhang, K.; Xu, D.; Yang, G.; Huang, H.; Nie, F.; Liu, C.; Yang, S. CuO nanostructures: Synthesis, characterization, growth mechanisms, fundamental properties, and applications. *Prog. Mater. Sci.* 2014, 60, pp. 208–337.

3. Postica, V.; Ababii, N.; Creţu, V. Synthesis of Al-doped ZnO nanostructured films by SCS method. In *Conferinţa Ştiinţifică a Colaboratorilor, Doctoranzilor şi Studenţilor UTM*, 2013, 1, pp. 112-115.

4. Xu, C.H.; Woo, C.H.; Shi, S.Q. Formation of CuO nanowires on Cu foil. *ChemicalPhysics Letters* 2004, 399, pp. 62–66.

5. Choi, Y.; Kim, D.; Hong, S.; Hong, K. S. H_2 and C_2H_5OH sensing characteristics of mesoporous p-type CuO films prepared via a novel precursor-based ink solution route. *Sensors and Actuators B* 2013, 178, pp. 395-403.

6. Hoa, N. D.; Quy, N. V.; Tuan, M. A.; Hieu N. V. Facile synthesis of p-type semiconducting cupric oxide nanowires and their gas-sensing properties sensor. *Physica E* 2009, 42, pp. 146–149.

7. Lupan, O.; Postica, V.; Ababii, N.; Hoppe, M.; Cretu, V.; Tiginyanu, I.; Sontea, V.; Pauporté, Th.; Viana, B.; Adelun, R. Influence of CuO nanostructures morphology on hydrogen gas sensing performance. *Microelectr. Eng.* 2016, 164, pp. 63-70.

8. Lupan, O.; Cretu, V.; Postica, V.; Polonskyi, O.; Ababii, N.; Schütt, F.; Kaidas, V.; Faupel, F.; Adelung, R. Non-Planar Nanoscale p-p Heterojunctions Formation in $Zn_xCu_{1-x}O_y$ Nanocrystals by Mixed Phases for Enhanced Sensors. *Sensors and Actuators B: Chemical* 2016, 230, pp. 832-843.

9. Postica, V.; Gröttrup, J.; Adelung, R.; Lupan, O.; Mishra, A.K.; De Leeuw, N.H.; Ababii, N.; Carreira, J.F.C.; Rodrigues, J.; Ben Sedrine, N.; Correia, M.R.; Monteiro, T.; Sontea, V.; Mishra, Y.K. Multifunctional Materials: „A Case Study of the Effects of Metal Doping on ZnO Tetrapods with Bismuth and Tin Oxides". *Adv. Funct.Mater.* 2017, 27, 1604676, http://dx.doi.org/10.1002/adfm.201604676.

10. Lupan, O.; Ababii, N.; Mishra, A.K.; Gronenberg, O.; Vahl, A.; Schürmann, U.; Duppel, V.; Krüger, H.; Chow, L.; Kienle, L.; Faupel, F.; Adelung, R.; De Leeuw, N.H.; Hansen, S. Single $CuO/Cu_2O/Cu$ microwire covered by a nanowire network as agas sensor for the detection of battery hazards. *ACS Appl. Mater. Interfaces* 2020,12, pp. 42248–42263, http://dx.doi.org/10.1021/acsami.0c09879.

11. Helwig, N.; Schüler, M.; Bur, C.; Schütze, A.; Sauerwald, T. Gas mixing apparatusfor automated gas sensor characterization. *Meas. Sci. Technol.* 2014, 25, 055903, http://dx.doi.org/10.1088/0957-0233/25/5/055903.

12. Luo, S.; Shen, Y.; Wu, Z.; Cao, M.; Gu, F.; Wang, L. Enhanced ethanol sensingperformance of mesoporous Sn-doped ZnO. *Mater. Sci. Semicond. Process* 2016, 41, pp. 535–543, http://dx.doi.org/10.1016/j.mssp.2015.10.001.

13. Kresse, G.; Hafner, J. Ab initio molecular dynamics for liquid metals. *Phys. Rev. B: Condens. Matter Mater. Phys.* 1993, 47, pp. 558–561, https://doi.org/10.1103/PhysRevB.47.558.

14. Kresse, G.; Hafner, J. Ab initio molecular-dynamics simulation of the liquid-metal–amorphous-semiconductor transition in germanium. *Phys. Rev. B: Condens. Matter Mater. Phys* 1994, 49, pp. 14251–14269, https://doi.org/10.1103/PhysRevB.49.14251.

15. Kresse, G.; Furthmüller, J. Efficiency of ab-initio total energy calculations for metals and semiconductors using a plane-wave basis set. *Comput. Mater. Sci.* 1996, 6, pp. 15–50, https://doi.org/10.1016/0927-0256(96)00008-0.

16. Kresse, G.; Furthmüller, J. Efficient iterative schemes for ab initio total-energy calculations using a plane-wave basis set. *J. Phys. Rev. B: Condens. Matter Mater. Phys.* 1996, 54, pp. 11169–11186, https://doi.org/10.1103/PhysRevB.54.11169.

17. Blochl, P. E. Projector augmented-wave method. *Phys. Rev. B: Condens. Matter Mater. Phys.* 1994, 50, pp. 17953–17979, https://doi.org/10.1103/PhysRevB.50.17953.

18. Perdew, J. P.; Burke, K.; Ernzerhof, M. Generalized Gradient Approximation Made Simple. *Phys. Rev. Lett.* 1996, 77, pp. 3865–3868, https://doi.org/10.1103/PhysRevLett.77.3865.

19. Perdew, J. P.; Burke, K.; Ernzerhof, M. Generalized Gradient Approximation Made Simple. *Phys. Rev. Lett.* 1997, 78, pp. 1396, https://doi.org/10.1103/PhysRevLett.78.1396.

20. Dudarev, S. L.; Savrasov, S. Y.; Humphreys, C. J.; Sutton, A. P. Electron-energy-loss spectra and the structural stability of nickel oxide: An LSDA+U study. *Phys. Rev. B: Condens. Matter Mater. Phys.* 1998, 57, pp. 1505–1509, https://doi.org/10.1103/PhysRevB.57.1505.

21. Mishra A. K, Roldan A., H. De Leeuw N., CuO Surfaces and CO_2 activation : A dispersion corrected DFT+U study. J. Phys. Chem. C 2016, 120 (4), 2198.

22. Mishra A. K, Roldan A., H. De Leeuw N., Density Functional Theory Study of the Adsorption Behaviour of CO_2 on Cu_2O Surfaces. *J. Chem. Phys.* 2016, 145, pp. 044709-13.

23. Grimme, S. Semiempirical GGA-type density functional constructed with a long-range dispersion correction *J. Comput. Chem.* 2006, 27, pp. 1787–1799, https://doi.org/10.1002/jcc.20495.

24. Cretu, V.; Postica, V.; Mishra, A. K.; Hoppe, M.; Tiginyanu, I.; Mishra, Y. K.; Chow, L.; De Leeuw, N. H.; Adelung, R.; Lupan, O. Synthesis, characterization and DFT studies of zinc-doped copper oxide nanocrystals for gas sensing applications. *Journal of Materials Chemistry A.* 2016, 4, pp. 6527–6539.

25. Åsbrink, S.; Norrby, L. J. Refinement of the Crystal Structure of Copper(II)Oxide with a Discussion of Some Exceptional E.s.d.`s. *Acta Crystallogr. Sect. B Struct. Crystallogr. Cryst. Chem.* 1970, 26, 8 p.

26. Tiginyanu, I.M., Lupan, O., Ursaki, V. V., Chow, L., Enachi, M. Nanostructures of Metal Oxides. In: P. Bhattacharya, R. Fornari, H. Kamimura (Eds.). *Comprehensive Semiconductor Science and Technology*, Elsevier Science, Amsterdam, 2011, 3, pp. 396-479.

27. Hübner, M.; Simion, C. E.; Tomescu-Stănoiu, A.; Pokhrel, S.; Bârsan N.; Weimar, U. Influence of humidity on CO sensing with p-type CuO thick film gas sensors. *Sensors and Actuators B: Chemical* 153, (2011), pp. 347-353.

28. Zoolfakar, A.; Ahmad, M.; Rani, R.; Ou, J.; Balendhran, S.; Zhuiykov, S.; Latham, K.; Wlodarski, W.; Kalantar-Zadeh, K. Nanostructured copper oxides as ethanol vapour sensors. *Sensors and Actuators B: Chemical* 2013, 185, pp. 620-627.

29. Yu, H.; Yu, J.; Liu, S.; Mann, S. Template-free hydrothermal synthesis of CuO/Cu_2O composite hollow microspheres. *Chemistry of Materials* 2007, 19, pp. 4327-4334.

30. Sun B. Z., Chen W. K., Wang X., Lu C. H., A density functional theory study on the adsorption and dissociation of N_2O on Cu_2O(1 1 1) surface. *Appl. Surf. Sci.* 2007, 253, pp. 7501, https://doi.org/10.1016/j.apsusc.2007.03.042.

31. Cretu, V.; Ababii N.; Chistruga A.; Magariu N.; Postica V.; Lupan O. CuO/Cu_2O Nanostructured Films for Gas Sensors In: *2019 IEEE 9th International Conference Nanomaterials: Applications & Properties (NAP),* 2019 15-20 Sept. DOI: 10.1109/NAP47236.2019, vol.2, pp-02NEE19

Publisher's Note: JES stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.

Submission of manuscripts: jes@meridian.utm.md

Supporting Information

Figure S1. Linescan EDX mapping of the copper oxide (CuO) film with rapid thermal processing at 700 °C (R700).

SOLVING THE DAYS-OFF SCHEDULING PROBLEM USING QUADRATIC PROGRAMMING WITH CIRCULANT MATRIX

Vasile Moraru *, ORCID: 0000-0002-6400-1374,
Daniela Istrati, ORCID: 0000-0002-1607-92731,
Sergiu Zaporojan, ORCID: 0000-0001-5928-4229

Technical University of Moldova, 168 Stefan cel Mare Blvd., Chisinau, Republic of Moldova
*Corresponding author: Vasile Moraru, *vasile.moraru@ia.utm.md*

Abstract. The purpose of this paper is the approach of a mathematical model dedicated to planning the consecutive days off of a company's employees. Companies must find a flexible work schedule between employees, always considering the satisfaction of work tasks as well as guaranteeing consecutive days off. The analysis is based on solving a quadratic programming problem with binary variables. The proposed method uses the properties of the circulant symmetric matrix in the researched model, which allows the transformation of the considered problems into an equivalent separable non-convex optimization problem. A practical continuous convex relaxation approach is proposed. DC Algorithm is used to solve relaxed problems. A solved numerical example is presented.

Keywords: *scheduling problem, binary quadratic programming, circulant matrix, separable optimization, convex relaxation.*

Rezumat. Scopul acestei lucrări este abordarea unui model matematic dedicat planificării zilelor libere consecutive ale angajaților unei companii. Companiile trebuie să găsească un program de lucru flexibil între angajați, având întotdeauna în vedere satisfacerea sarcinilor de lucru cât și garantarea zilelor libere consecutive. Tratarea se bazează pe rezolvarea unei probleme de programare pătratică cu variabile binare. Metoda propusă utilizează proprietățile matricei simetrice circulante din modelul cercetat, care permite transformarea problemei considerate într-o problemă echivalentă de optimizare neconvexă separabilă. Este propusă o abordare practică de relaxare continuă convexă. Pentru rezolvarea problemei relaxate se utilizează Algoritmul DC. Se prezintă un exemplu numeric rezolvat.

Cuvinte-cheie: *problemă de planificare, programare pătratică binară, matrice circulantă, optimizare separabilă, relaxare convexă.*

1. Introduction

In both production organizations and service companies (IT companies, airlines, security services, fire stations, restaurants, hospitals, etc.) a specific concern is the planning of days off and working days for a week (or multiples thereof). Work schedules should require

employees to be present at work on different days to maintain the admissible quality of services. At the same time, employees must have the desired number of consecutive days off during the week. The adaptability for days-off planning in companies is a serious problem, especially in recent years, during the Covid-19 pandemic. Companies need to find a flexible work schedule between employees, always considering the satisfaction of work tasks as well as the guarantee of consecutive days off. A comprehensive study of the literature on this subject can be found in [1, 2].

In this paper, the mathematical model proposed in [3] is developed, in which the problem of free days planning is reformulated as a binary problem of non-convex quadratic programming. In the case of convex, effective (polynomial) solving algorithms have been proposed [4]. But if the quadratic programming problem is nonconvex or includes integer variables, the problem is NP-hard [5, 6]. No known algorithm can solve such problems efficiently.

The paper is organized as follows: Sections 3 and 4 describe the issue of day planning and formulated as a binary problem of quadratic programming. Section 5 investigates the properties of the symmetric matrix that form the purpose matrix. In Section 5 the quadratic programming problem turns into an equivalent separable programming problem.

2. Problem definition

We will consider that the working week is 5 days, i.e., the company or institution has a compressed and flexible work schedule. The days are marked to be 1, 2, 3, 4, 5. The planning of days off and working days per week or the multi-week work cycle assumes that the following assumptions and constraints are met [7, 3]:

1. The total number of employees m and the number of workers n_k required during the day are known: $k \in \{1, 2, 3, 4, 5\}$.
2. Each employee $i \in \{1, 2, …, m\}$ has a fixed number d_i of days off per week.
3. Each employee has at least 2 consecutive days off per week.
4. Some specific tasks need to be assigned to employees who have the skills to perform them.

3. The 0-1 Quadratic Programming Model [3]

For each worker, we enter the binary variable x_{ik} so that:

$$x_{ik} = \begin{cases} 1, \text{if day } k \in \{1,2,3,4,5\} \text{ is an off day for worker } i \in \{1,2,…,m\} \\ 0, \quad \text{otherwise} \end{cases}$$

We have the matrix (Eq. (1)):

$$X = \begin{pmatrix} x_{11} & x_{12} & x_{13} & x_{14} & x_{15} \\ x_{21} & x_{22} & x_{23} & x_{24} & x_{25} \\ … & … & … & … & … \\ x_{m1} & x_{m2} & x_{m3} & x_{m4} & x_{m5} \end{pmatrix} \in \mathbb{R}^{m \times 5} \tag{1}$$

We convert this matrix into a column vector consisting of its lines (Eq. (2)):

$$x = (x_{11} \quad x_{12} \quad … \quad x_{15} \quad x_{21} \quad x_{22} \quad … \quad x_{25} \quad x_{m1} \quad x_{m2} \quad … \quad x_{m5})^T. \tag{2}$$

For every day $k \in \{1,2,3,4,5\}$, the number of workers taking this day off is $m - n_k$. This assumption can be written as:

$$\begin{cases} x_{11} + x_{21} + \cdots + x_{m1} = m - n_1 \\ x_{12} + x_{22} + \cdots + x_{m2} = m - n_2 \\ \dots \dots \dots \dots \dots \dots \dots \dots \dots \dots \dots \dots \dots \\ x_{15} + x_{25} + \cdots + x_{m5} = m - n_5 \end{cases}.$$

Or in the matrix form

$$Ax=b$$

where $b = (m - n_1 \quad m - n_2 \quad m - n_3 \quad m - n_4 \quad m - n_5)^T \in \mathbb{R}^5$ and

$$A = (I \quad I \quad \dots \quad I) \in \mathbb{R}^{5 \times (5m)}.$$

Here *I* is the 5×5 identity matrix.

The assumption that each worker (employee) $i \in \{1, 2, \dots, m\}$ has d_i days off per week can be written as follows:

$$\begin{cases} x_{11} + x_{12} + x_{13} + x_{14} + x_{15} = d_1 \\ x_{21} + x_{22} + x_{23} + x_{24} + x_{25} = d_2 \\ \dots \dots \dots \dots \dots \dots \dots \dots \dots \dots \dots \dots \dots \\ x_{m1} + x_{m2} + x_{m3} + x_{m4} + x_{m5} = d_m \end{cases}$$

or *Ex=d* , where

$$d = (d_1 \quad d_2 \quad \dots \quad d_m)^T \in \mathbb{R}^m,$$

$$E = (E_1 \quad E_2 \quad \dots \quad E_m) \in \mathbb{R}^{m \times (5m)}.$$

The matrices $E_i \in \mathbb{R}^{m \times 5}$ are in the form

$$E_i = \begin{pmatrix} 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 \\ \dots & \dots & \dots & \dots & \dots \\ 1 & 1 & 1 & 1 & 1 \\ 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 \end{pmatrix} \leftarrow the\ row\ i \in \{1, 2, \dots, m\}.$$

The assumption that some specific tasks must be performed by a worker with the appropriate skill can be written as follows:

$$c_{ik} = \begin{cases} 1, \text{if the presence of the worker } i \in \{1, 2, \dots, m\} \text{ is needed for the day } k \in \{1,2,3,4,5\} \\ 0, \qquad \text{otherwise} \end{cases}$$

Then

$$Cx=0,$$

where $0 = (0 \quad 0 \quad \dots \quad 0)^T \in \mathbb{R}^m$ – is the zero-column vector and $C = \{0,1\}^{m \times (5m)}$ is the matrix with the c_{ik} elements defined above.

We need a function that allows us to maximize the number of consecutive days off per week [3]:

$$f(x) = \sum_{i=1}^{m} \left(\sum_{k=1}^{4} x_{ik} x_{i,k+1} + x_{i5} x_{i1} \right).$$

The objective function *f(x)* can be written as follows:

$$f(x) = x^T Q x.$$

Here the symmetric matrix Q is a block diagonal matrix:

$$Q = \begin{pmatrix} Q_0 & O & \dots & O \\ O & Q_0 & \dots & O \\ \vdots & \vdots & \ddots & \vdots \\ O & O & \dots & Q_0 \end{pmatrix} \in \mathbb{R}^{(5m)\times(5m)}.$$

Where zero matrix $O \in \mathbb{R}^{5\times5}$ is a matrix in which all of the entries are 0, and

$$Q_0 = \begin{pmatrix} 0 & \frac{1}{2} & 0 & 0 & \frac{1}{2} \\ \frac{1}{2} & 0 & \frac{1}{2} & 0 & 0 \\ 0 & \frac{1}{2} & 0 & \frac{1}{2} & 0 \\ 0 & 0 & \frac{1}{2} & 0 & \frac{1}{2} \\ \frac{1}{2} & 0 & 0 & \frac{1}{2} & 0 \end{pmatrix} \in \mathbb{R}^{5\times5}$$

Thus, maximizing the number of consecutive days off is to solve the following problem of quadratic programming with binary variables:

$$\left. \begin{array}{l} f(x) = x^T Q x \to max \\ \quad subject\ to \\ \quad Ax = b, \\ \quad Ex = d, \\ \quad Cx = 0, \\ \quad x \in \{0,1\}^n \end{array} \right\}$$ (QP)

When solving the problem (QP), we encounter great difficulties because the function is non-convex and the variables are integers (Boolean). The problem (QP) in general case is NP-hard [5, 8] and is very difficult to solve. The interest in such issues is the subject of a rich literature (see synthesis papers [9]) and has given rise to the development of numerous methods:

- Lagrangian relaxation to semi-defined relaxation [10, 11].
- Conical relaxation [12, 13].
- Continuous reformulation [14 - 16].
- Linear reformulation (reformulation-linearization technique RLT) [17].
- Heuristic and genetic methods [18, 19].

This paper proposes a method for solving the problem (QP), using the separable reformulation. This method is based on diagonalizing the Q matrix.

4. Q_0 matrix eigenvalues and eigenvectors

The matrix Q_0 is a symmetric circulant matrix [20]. It is immediately verified that the vector $x_p^{(1)} = (1 \quad 1 \quad 1 \quad 1 \quad 1)^T$ is an eigenvector. Indeed

$$Q_0 x_p^{(1)} = \left(\frac{1}{2} + \frac{1}{2}\right) x_p^{(1)} = x_p^{(1)}.$$

Thus $\lambda_1 = 1$ is an eigenvalue of the matrix Q_0. The other eigenvectors can be easily found using the primitive root of unity: $\omega_5 = exp(2\pi i/5)$, $i = \sqrt{-1}$.

In ω_5 terms, the the eigenvalues of the matrix Q_0 are [20]:

$$\lambda_k = \frac{1}{2}\omega_5^{k-1} - \frac{1}{2}\omega_5^{4(k-1)}, \quad k = 1,2,3,4,5.$$

So, we have (Eq. 3):

$$\left.\begin{array}{l} \lambda_1 = 1, \; \lambda_2 = \dfrac{1}{4}\sqrt{5} - \dfrac{1}{4}, \; \lambda_3 = -\dfrac{1}{4}\sqrt{5} - \dfrac{1}{4} \\[2mm] \lambda_4 = -\dfrac{1}{4}\sqrt{5} - \dfrac{1}{4} = \lambda_3, \; \lambda_5 = \dfrac{1}{4}\sqrt{5} - \dfrac{1}{4} = \lambda_2 \end{array}\right\}. \tag{3}$$

The eigenvectors are determined using the same primitive root of the ω_5 unit:

$$v_p^{(k)} = \left(\omega_5^{0\times(k-1)} \quad \omega_5^{1\times(k-1)} \quad \omega_5^{2\times(k-1)} \quad \omega_5^{3\times(k-1)} \quad \omega_5^{4\times(k-1)}\right)^T, \quad k = 1,2,3,4,5.$$

We note (Eq. 4):

$$\left.\begin{array}{l} \mu_2 = \dfrac{1}{4}\sqrt{2}\sqrt{5+\sqrt{5}}, \\[3mm] \mu_3 = \dfrac{1}{4}\sqrt{2}\sqrt{5-\sqrt{5}} \end{array}\right\}. \tag{4}$$

Performing the respective calculations and taking into account (Eq. 3 and Eq. 4), we obtain the eigenvectors of the matrix Q_0:

$$v_p^{(1)} = (1 \quad 1 \quad 1 \quad 1 \quad 1)^T = (\lambda_1 \quad \lambda_1 \quad \lambda_1 \quad \lambda_1 \quad \lambda_1)^T,$$

$$v_p^{(2)} = \begin{pmatrix} 1 \\ \frac{1}{4}\sqrt{5} - \frac{1}{4} + i\frac{1}{4}\sqrt{2}\sqrt{5+\sqrt{5}} \\ -\frac{1}{4}\sqrt{5} - \frac{1}{4} + i\frac{1}{4}\sqrt{2}\sqrt{5-\sqrt{5}} \\ -\frac{1}{4}\sqrt{5} - \frac{1}{4} - i\frac{1}{4}\sqrt{2}\sqrt{5-\sqrt{5}} \\ \frac{1}{4}\sqrt{5} - \frac{1}{4} - i\frac{1}{4}\sqrt{2}\sqrt{5+\sqrt{5}} \end{pmatrix} = \begin{pmatrix} \lambda_1 \\ \lambda_2 + i\mu_2 \\ \lambda_3 + i\mu_3 \\ \lambda_3 - i\mu_3 \\ \lambda_2 + i\mu_2 \end{pmatrix},$$

$$v_p^{(3)} = \begin{pmatrix} 1 \\ -\frac{1}{4}\sqrt{5} - \frac{1}{4} + i\frac{1}{4}\sqrt{2}\sqrt{5-\sqrt{5}} \\ \frac{1}{4}\sqrt{5} - \frac{1}{4} - i\frac{1}{4}\sqrt{2}\sqrt{5+\sqrt{5}} \\ \frac{1}{4}\sqrt{5} - \frac{1}{4} + i\frac{1}{4}\sqrt{2}\sqrt{5+\sqrt{5}} \\ -\frac{1}{4}\sqrt{5} - \frac{1}{4} - i\frac{1}{4}\sqrt{2}\sqrt{5-\sqrt{5}} \end{pmatrix} = \begin{pmatrix} \lambda_1 \\ \lambda_3 + i\mu_3 \\ \lambda_2 - i\mu_2 \\ \lambda_2 + i\mu_2 \\ \lambda_3 - i\mu_3 \end{pmatrix},$$

$$v_p^{(4)} = \begin{pmatrix} 1 \\ -\frac{1}{4}\sqrt{5} - \frac{1}{4} - i\frac{1}{4}\sqrt{2}\sqrt{5-\sqrt{5}} \\ \frac{1}{4}\sqrt{5} - \frac{1}{4} + i\frac{1}{4}\sqrt{2}\sqrt{5+\sqrt{5}} \\ \frac{1}{4}\sqrt{5} - \frac{1}{4} - i\frac{1}{4}\sqrt{2}\sqrt{5+\sqrt{5}} \\ -\frac{1}{4}\sqrt{5} - \frac{1}{4} + i\frac{1}{4}\sqrt{2}\sqrt{5-\sqrt{5}} \end{pmatrix} = \begin{pmatrix} \lambda_1 \\ \lambda_3 - i\mu_3 \\ \lambda_2 + i\mu_2 \\ \lambda_2 - i\mu_2 \\ \lambda_3 + i\mu_3 \end{pmatrix},$$

$$v_p^{(5)} = \begin{pmatrix} 1 \\ \frac{1}{4}\sqrt{5} - \frac{1}{4} - i\frac{1}{4}\sqrt{2}\sqrt{5+\sqrt{5}} \\ -\frac{1}{4}\sqrt{5} - \frac{1}{4} - i\frac{1}{4}\sqrt{2}\sqrt{5-\sqrt{5}} \\ -\frac{1}{4}\sqrt{5} - \frac{1}{4} + i\frac{1}{4}\sqrt{2}\sqrt{5-\sqrt{5}} \\ \frac{1}{4}\sqrt{5} - \frac{1}{4} + i\frac{1}{4}\sqrt{2}\sqrt{5+\sqrt{5}} \end{pmatrix} = \begin{pmatrix} \lambda_1 \\ \lambda_2 - i\mu_2 \\ \lambda_3 - i\mu_3 \\ \lambda_3 + i\mu_3 \\ \lambda_2 + i\mu_2 \end{pmatrix}.$$

Since Q_0 is a symmetric matrix with real elements and real eigenvalues, then we can always choose the corresponding eigenvectors with real inputs. Indeed, if

$$v = a + ib, \ ab \neq 0, \ i = \sqrt{-1},$$

is an eigenvector of the matrix M with eigenvalue λ. Then

$$M(a + ib) = \lambda(a + ib).$$

From here it follows that

$$Ma = \lambda a, Mb = \lambda b,$$
$$M(a + b) = \lambda(a + b),$$

i.e., the vectors a, b and $a + b$ are also eigenvectors associated with eigenvalue λ. Therefore, the real parts, the imaginary parts and their sum will in turn be their eigenvectors. Thus, we have the eigenvectors of the matrix Q_0 with the real elements:

$$p_k = Re\left(v_p^{(k)}\right) + Im(v_p^{(k)}), k = 1,2,3,4,5.$$

To establish other properties of the matrix Q_0 we consider the matrix whose columns are the eigenvectors p_k:

$$F_0 = \frac{1}{\sqrt{5}}(p_1 \quad p_2 \quad p_3 \quad p_4 \quad p_5) = \begin{pmatrix} \lambda_1 & \lambda_1 & \lambda_1 & \lambda_1 & \lambda_1 \\ \lambda_1 & \lambda_2 + \mu_2 & \lambda_3 + \mu_3 & \lambda_3 - \mu_3 & \lambda_2 - \mu_2 \\ \lambda_1 & \lambda_3 + \mu_3 & \lambda_2 - \mu_2 & \lambda_2 + \mu_2 & \lambda_3 - \mu_3 \\ \lambda_1 & \lambda_3 - \mu_3 & \lambda_2 + \mu_2 & \lambda_2 - \mu_2 & \lambda_3 + \mu_3 \\ \lambda_1 & \lambda_2 - \mu_2 & \lambda_3 - \mu_3 & \lambda_3 + \mu_3 & \lambda_2 + \mu_2 \end{pmatrix}.$$

The F_0 matrix is the Discrete Fourier Transform (DFT) matrix, with its orthogonal columns (orthonormal).

We will note the following properties of the F_0 matrix, properties that are verified by direct calculation:

1. $F_0 = F_0^T$
2. $F_0^2 = I$
3. $F_0^{-1} = F_0$
4. $\det(F_0) = 1$
5. $cond(F_0) = 1$

Thus, F_0 is a well-conditioned special orthogonal symmetric matrix. This is important from a numerical calculation point: small disturbances in the input data will not produce large variations in the calculations [21]. Using the orthogonal matrix F_0, the diagonalization of the circulating matrix Q_0 can be achieved (Eq. 5) [21]:

$$F_0 Q_0 F_0 = \Lambda_0 \tag{5}$$

where

$$\Lambda_0 = Diag(\lambda_1 \lambda_2 \lambda_3 \lambda_4 \lambda_5) = \begin{pmatrix} \lambda_1 & 0 & 0 & 0 & 0 \\ 0 & \lambda_2 & 0 & 0 & 0 \\ 0 & 0 & \lambda_3 & 0 & 0 \\ 0 & 0 & 0 & \lambda_4 & 0 \\ 0 & 0 & 0 & 0 & \lambda_5 \end{pmatrix},$$

namely (Eq. (6)):

$$Q_0 = F_0 \Lambda_0 F_0 \tag{6}$$

5. A separable reformulation of the quadratic program (QP)

Consider now the direct sum m of the matrix Q_0: $Q_0 \oplus Q_0 \oplus \cdots \oplus Q_0 \oplus Q_0$, which as a result gives us the matrix Q. Matrix Q is a block circulant matrix. The eigenvalues and eigenvectors of the matrix Q are the sum of the sets of 5 eigenvectors λ_1, λ_2, λ_3, λ_4, λ_5 and respectively the 5 eigenvectors p_1, p_2, p_3, p_4, p_5.

We note the diagonal block matrices:

$$F = F_0 \oplus F_0 \oplus \cdots \oplus F_0 \oplus F_0 = \begin{pmatrix} F_0 & O & \cdots & O & O \\ O & F_0 & \cdots & O & O \\ \vdots & \vdots & \ddots & \vdots & \vdots \\ O & O & \cdots & F_0 & O \\ O & O & \cdots & O & F_0 \end{pmatrix},$$

$$\Lambda = \Lambda_0 \oplus \Lambda_0 \oplus \cdots \oplus \Lambda_0 \oplus \Lambda_0 = \begin{pmatrix} \Lambda_0 & O & \cdots & O & O \\ O & \Lambda_0 & \cdots & O & O \\ \vdots & \vdots & \ddots & \vdots & \vdots \\ O & O & \cdots & \Lambda_0 & O \\ O & O & \cdots & O & \Lambda_0 \end{pmatrix}.$$

Then the matrix Q is diagonalized: $Q = F \Lambda F$.

Let be the column vector $\bar{x}_i = (x_{i1} \quad x_{i2} \quad x_{i3} \quad x_{i4} \quad x_{i5})^T$, $i=1,2, …, n$.

Then the vector x defined in (Eq. 2) can be written:

$$x = (\bar{x}_1^T \quad \bar{x}_2^T \quad \cdots \quad \bar{x}_m^T)^T \in \mathbb{R}^{5m}.$$

With these notations we rewrite the objective function as follows:

$$f(x) = x^T Q x = \begin{pmatrix} \bar{x}_1^T & \bar{x}_2^T & \cdots & \bar{x}_{m-1}^T & \bar{x}_m^T \end{pmatrix} \begin{pmatrix} Q_0 & O & \cdots & O & O \\ O & Q_0 & \cdots & O & O \\ \vdots & \vdots & \ddots & \vdots & \vdots \\ O & O & \cdots & Q_0 & O \\ O & O & \cdots & O & Q_0 \end{pmatrix} \begin{pmatrix} \bar{x}_1 \\ \bar{x}_2 \\ \vdots \\ \bar{x}_{m-1} \\ \bar{x}_m \end{pmatrix} =$$

$$= \bar{x}_1^T Q_0 \bar{x}_1 + \bar{x}_2^T Q_0 \bar{x}_2 + \cdots + \bar{x}_{m-1}^T Q_0 \bar{x}_{m-1} + \bar{x}_m^T Q_0 \bar{x}_m,$$

or, taking into account (Eq. 6):

$$f(x) = (F_0 \bar{x}_1)^T \Lambda_0 F_0 \bar{x}_1 + (F_0 \bar{x}_2)^T \Lambda_0 F_0 \bar{x}_2 + \cdots + (F_0 \bar{x}_m)^T \Lambda_0 F_0 \bar{x}_m.$$

We determine the column vector

$$\bar{y}_i = F_0 \bar{x}_i = (y_{i1} \quad y_{i2} \quad y_{i3} \quad y_{i4} \quad y_{i5})^T, \, i{=}1, 2, 3, \dots, m \,,$$

with the elements

$$y_{ij} = \frac{\sqrt{5}}{5} p_j^T \bar{x}_i, \, i = 1,2,\dots,m; \, j = 1,2,3,4,5,$$

where $\frac{\sqrt{5}}{5} p_j$ is the column j of the matrix F_0.

We note

$$y = (\bar{y}_1^T \quad \bar{y}_2^T \quad \dots \quad \bar{y}_m^T)^T \in \mathbb{R}^{5m}.$$

We have (Eq. 7)

$$y = Fx = \begin{pmatrix} F_0 \bar{x}_1 \\ F_0 \bar{x}_2 \\ \vdots \\ F_0 \bar{x}_m \end{pmatrix}. \tag{7}$$

Then in the variables y_{ij} the purpose function becomes a separable function:

$$g(y) = \bar{y}_1^T \Lambda_0 \bar{y}_1 + \bar{y}_2^T \Lambda_0 \bar{y}_2 + \cdots + \bar{y}_m^T \Lambda_0 \bar{y}_m =$$

$$= \lambda_1 \sum_{i=1}^{m} y_{i1}^2 + \lambda_2 \sum_{i=1}^{m} y_{i2}^2 + \lambda_3 \sum_{i=1}^{m} y_{i3}^2 + \lambda_4 \sum_{i=1}^{m} y_{i4}^2 + \lambda_5 \sum_{i=1}^{m} y_{i5}^2 = \sum_{k=1}^{5} \sum_{i=1}^{m} \lambda_k y_{ik}^2.$$

As $F^{-1} = F$, $F_0^{-1} = F_0$, from (Eq. 7), we have

$$x = Fy, \bar{x}_i = F_0 \bar{y}_i, \, i = 1,2,\dots,m,$$

$$x_{ik} = \frac{\sqrt{5}}{5} p_k^T \bar{y}_i, i = 1,2,\dots,m; \, k = 1,2,3,4,5.$$

Then the constraints

$$x_{1k} + x_{2k} + \dots + x_{mk} = m - n_k, k = 1,2,3,4,5,$$

$$x_{i1} + x_{i2} + x_{i3} + x_{i4} + x_{i5} = d_i, \, i = 1,2,3,\dots,m,$$

become (Eq. 8) and (Eq. 9):

$$p_k^T (\bar{y}_1 + \bar{y}_2 + \cdots + \bar{y}_m) = \sqrt{5}(m - n_k), \qquad k = 1,2,3,4,5, \tag{8}$$

$$(p_1 + p + p_3 + p_4 + p_5)^T \bar{y}_i = \sqrt{5} d_i, i = 1,2,\dots,m. \tag{9}$$

Considering

$$(p_1 + p_2 + p_3 + p_4 + p_5)^T = (5 \quad 0 \quad 0 \quad 0 \quad 0)^T,$$

from (Eq. 9) we obtain (Eq. 10):

$$y_{i1} = \frac{\sqrt{5}}{5} d_i, i = 1, 2, \ldots, m. \tag{10}$$

So

$$\bar{y}_i = \left(\frac{\sqrt{5}}{5} d_i \quad y_{i2} \quad y_{i3} \quad y_{i4} \quad y_{i5} \right)^T, \quad i = 1, 2, \ldots, m.$$

Thus, the problem (QP) was transformed into the following

$$g(y) = \sum_{k=1}^{5} \sum_{i=1}^{m} \lambda_k y_{ik}^2 \rightarrow max \tag{11a}$$

subject to

$$p_k^T \left(\sum_{i=1}^{m} \bar{y}_i \right) = \sqrt{5}(m - n_i), \qquad k = 1,2,3,4,5, \tag{11b}$$

$$\left(\sum_{k=1}^{5} p_k^T \right) \bar{y}_i = \sqrt{5} d_i, i = 1, 2, \ldots, m, \tag{11c}$$

$$CFy = 0, \tag{11d}$$

$$p_k^T \bar{y}_i \in \{0, \sqrt{5}\} \ k = 1,2,3,4,5; \ i = 1, 2, \ldots, m. \tag{11e}$$

Problem (Eq. 11) is a non-convex separable quadratic programming problem, except for constraints (Eq. 11e). The object function (Eq. 11a) is a non-convex quadratic function in which $\lambda_1 > 0$, $\lambda_2 > 0$, $\lambda_3 = \lambda_4 < 0$, $\lambda_5 = \lambda_2 > 0$ This function can be rewritten as the difference between two convex functions:

$$g(y) = \varphi_1(y) - \varphi_2(y),$$

where:

$$\varphi_1(y) = \sum_{i=1}^{m} \lambda_1 y_{i1}^2 + \sum_{i=1}^{m} \lambda_2 y_{i2}^2 + \sum_{i=1}^{m} \lambda_5 y_{i5}^2,$$

$$\varphi_2(y) = \sum_{i=1}^{m} (-\lambda_3 y_{i3}^2) + \sum_{i=1}^{m} (-\lambda_4 y_{i4}^2).$$

Nowadays there is an original theory for problems with functions represented as the difference between two convex functions, problems called DC programming (DC-Difference of Convex Functions). For such problems, effective solving algorithms have been developed, called DCA (DC Algorithm) [9, 18]. Constraints (Eq. 11b), (Eq. 11c), and (Eq. 11d) are linear with the decision variables y_{ik}. The last constraint (Eq. 11e) forces the variables $\frac{\sqrt{5}}{5} p_k^T \bar{y}_i$ to take the value 0 or 1 (zero or one). This makes it very difficult to solve the problem. A practical approach would be to relax these conditions by replacing (Eq. 11e) with (Eq. 11f):

$$0 \leq p_k^T \bar{y}_i \leq \sqrt{5}, k = 1,2, \dots ,5; \; i = 1,2, \dots, m. \tag{11f}$$

Thus, DCA methods can be used to solve the relaxed problem [9, 18]. The DCA method is of the primal-dual type and is based on the construction of two strings $\{y^{(k)}\}, \{u^{(k)}\}$ which are calculated at each iteration as follows:

Step 1. $y^{(0)}$ - the initial start approximation, $k=0$.

Step 2. It is determined $u^{(k)} = \nabla \varphi_1(y^{(k)})$.

Step 3. The solution $y^{(k+1)}$ of the convex separable programming problem is established:

$$\left. \begin{array}{c} \varphi_2(y) - \varphi_1(u^{(k)}) - (y - y^{(k)})^T u^{(k)} \to min \\ subject\ to\ (11b), (11c), (11d), (11f) \end{array} \right\}.$$

Step 4. If the stop criterion is checked, then STOP. Otherwise, take $k = k + 1$ and move on to Step 2.

6. A solved example

Consider the following example of planning consecutive days off with data:
- 5 days work week;
- Number of workers $m = 6$;
- Number of d_i days off per week for the worker $i = 1, 2, \dots, 6$:

$$d_1 = 2, d_2 = 2, d_3 = 2, d_4 = 3, d_5 = 3, d_6 = 4;$$

- n_k number of workers required on day k: $n_1 = 4, n_2 = 3, n_3 = 3, n_4 = 2, n_5 = 2$.

The optimal local solution of the model (Eq. (11a)), (Eq. (11b)), (Eq. (11c)), and (Eq. (11f)) provides the following values for $\bar{y}_i = (y_{i1}, \; y_{i2}, y_{i3}, y_{i4}, y_{i5})^T, i = 1, 2, 3, 4, 5, 6$:

$$y_{11} = 0.91423, y_{12} = -0.89614, y_{13} = -0.2341, y_{14} = -0.36601, y_{15} = 0.5002,$$
$$y_{21} = 0.91423, y_{22} = 0.95416, y_{23} = 0.4023, y_{24} = -0.20164, y_{25} = 0.20101,$$
$$y_{31} = 0.91423, y_{32} = 0.89614, y_{33} = -0.2341, y_{34} = -0.36601, y_{35} = 0.5002,$$
$$y_{41} = 1.0459, y_{42} = 1.09108, y_{43} = 0.05124, y_{44} = 0.42181, y_{45} = -0.51623,$$
$$y_{51} = 1.0459, y_{52} = -0.9172, y_{53} = -0.4023, y_{54} = 0.20164, y_{55} = -0.20101,$$
$$y_{61} = 1.5989, \; y_{62} = -0.50201 = y_{63} = y_{64} = \; y_{65}.$$

Considering that $\bar{x}_i = F_0 \bar{y}_i$, we obtain:

$$x_{11} = 5.7632 \cdot 10^{-2}, x_{12} = -8.8091 \cdot 10^{-3}, x_{13} = -1.4474 \cdot 10^{-2}, x_{14} = x_{15} = 1.011,$$
$$x_{21} = 0.99867, x_{22} = 0.95416, x_{23} = -3.6595 \cdot 10^{-2}, x_{24} = 0.10062, x_{25} = 2.7421 \cdot 10^{-2},$$
$$x_{31} = 5.7632 \cdot 10^{-2}, x_{32} = -8.8091 \cdot 10^{-3}, x_{33} = -1.4474 \cdot 10^{-2}, x_{34} = x_{35} = 1.011,$$
$$x_{41} = 1.0459, x_{42} = 0.96225, x_{43} = 0.90525, x_{44} = -0.25499, x_{45} = -0.21019,$$
$$x_{51} = -0.12208, x_{52} = -7.7861 \cdot 10^{-2}, x_{53} = 0.91319, x_{54} = 0.77597, x_{55} = 0.84918,$$
$$x_{61} = -0.18297, x_{62} = 0.93956 = x_{63} = x_{64} = x_{65.}$$

A schedule of consecutive days off generated by this model (rounding x_{ij} to integers) is given in the table 1:

A schedule of consecutive days off

Worker	Day					
	1	2	3	4	5	d_i
1	$x_{11} = 0$	$x_{12} = 0$	$x_{13} = 0$	$x_{14} = 1$	$x_{15} = 1$	2
2	$x_{21} = 1$	$x_{22} = 1$	$x_{23} = 0$	$x_{24} = 0$	$x_{25} = 0$	2
3	$x_{31} = 0$	$x_{32} = 0$	$x_{33} = 0$	$x_{34} = 1$	$x_{35} = 1$	2
4	$x_{41} = 1$	$x_{42} = 1$	$x_{43} = 1$	$x_{44} = 0$	$x_{45} = 0$	3
5	$x_{51} = 0$	$x_{52} = 0$	$x_{53} = 1$	$x_{54} = 1$	$x_{55} = 1$	3
6	$x_{61} = 0$	$x_{62} = 1$	$x_{63} = 1$	$x_{64} = 1$	$x_{65} = 1$	4
n_k	4	3	3	2	2	

7. Conclusions

In this paper we considered the problem of planning consecutive days off, formulated as a problem of non-convex quadratic binary programming, which is known to be NP-hard. The structure of the circulating matrix in the objective function allows its diagonalization. The main challenge is to quickly calculate the diagonal matrix Λ_0 and the Fourier matrix F_0, which in our approach reduces the considered problem to a separable programming problem. The characteristic of this transformation is that the matrix F remains well conditioned $(cond(F)) = 1$ regardless of the number of decision variables in the problem formulation. As far as we know, such circulating matrix approaches have not been studied in the literature. The proposed technique allows the use of the DCA algorithm to calculate local suboptimal solutions to the initiated problem, but it is also possible to find the global optimum using, for example, a branch and bound method, or in combination with the classical approximation results of the separable programming problem with a linear programming model.

Conflicts of Interest: The authors declare no conflict of interest.

References
1. Alfares, H.K. Survey, Categorization, and Comparison of Recent Tour Scheduling Literature. *Annals of Operations Research* 2004, 127, pp. 145–175.
2. Ernst, A.T.; Jiang, H.; Krishnamoorthy, M.; Sier, D. Staff scheduling and rostering: A review of applications, methods and models. *European Journal of Operational Research* 2004, 153 (1), pp. 3-27.
3. Loqman, Ch.; Ettaouil, M.; Hami, Y.; Haddouch, Kh. Convex Quadratic Reformulations for solving days-off scheduling problem. *Journal of Theoretical and Applied Information Technology* 2013, 49 (1), pp. 23-31.
4. Gould, N.; Toint, Ph. A Quadratic Programming Page. Available online: https://www.numerical.rl.ac.uk/people/nimg/qp/qp.html (accessed on 12.06.2022).
5. Pardalos, P.M.; Vavasis, S.A. Quadratic programming with one negative eigenvalue is NP-hard. *Journal Global Optimization* 1991, 1, pp. 15–22.
6. Furini, F.; Traversi, E.; Belotti, P.; Frangioni, A; Gleixner, A.; Gould, N.; Liberti, L.; Lodi, A.; Misener, R.; Mittelmann, M.; Sahinidis, N.V.; Vigerske, S.; Wiegele, A. QPLIB: a library of quadratic programming instances. *Mathematical Programming Computation* 2019, 11 (2), pp. 237-265.
7. Billionnet, A. Integer programming to schedule a hierarchical workforce with variable demands. *European Journal of Operational Research* 1999, 114 (1), pp. 105-114.
8. Pardalos, P. M.; Jha, S. Complexity of uniqueness and local search in quadratic 0-1 programming. *Operations Research Letters* 1992, 11 (2), pp. 119-123.
9. Floudas, C. A.; Visweswaran, V. Quadratic Optimization. In: *Handbook of Global Optimization. Nonconvex Optimization and Its Applications*. Horst, R., Pardalos, P.M. (eds), Springer New York, 1995, 2, pp. 217–269.

10. Shor, N. Z. Quadratic Optimization Problems. *Soviet Journal of Computer and Systems Sciences* 1987, 25, pp. 1-11.

11. Poljak, S.; Rendl, F.; Wolkowicz, H. A recipe for semidefinite relaxation for (0,1)-quadratic programming. *Journal of Global Optimization* 1995, 7, pp. 51–73.

12. Ito, N.; Kim, S.; Kojima, M.; Takeda, A.; Toh. K.-C. Equivalences and differences in conic relaxations of combinatorial quadratic optimization problems. *Journal of Global Optimization* 2018, 72. pp. 619–653.

13. Bomze, I.M.; Cheng, J.P.; Dickinson, J.C.; Lisser, A. A fresh CP look at mixed-binary QPs: new formulations and relaxations. *Mathematical Programming* 2017, 166, pp.159–184.

14. Burer, S. On the copositive representation of binary and continuous nonconvex quadratic programs. *Mathematical Programming* 2009, 120, pp. 479–495.

15. Billionnet, A.; Elloumi, S.; Plateau, M.-C. Improving the performance of standard solvers for quadratic 0-1 programs by a tight convex reformulation: The QCR method. *Discrete Applied Mathematics* 2009, 157 (6), pp. 1185-1197.

16. Poljak, S.; Wolkowicz, H. Convex Relaxations of (0, 1)- Quadratic Programming. *Mathematics of Operations Research* 1995, 20(3), pp. 550-561.

17. D.; Adams, W. P. A *Reformulation-Linearization Technique for Solving Discrete and Continuous Nonconvex Problems*. Springer, USA, 2013, 518 p.

18. Glover, F.; Kochenberger, G.A.; Alidaee, B. Adaptive Memory Tabu Search for Binary Quadratic Program. *Management Science* 1998, 44 (3), pp. 336–345.

19. Lodi, A.; Allemand, K.; Liebling, T.M. An Evolutionary Heuristic for Quadratic 0-1 Programming. *European Journal of Operations Research* 1999, 119(3), pp. 662-670.

20. Davis, P. J. D. *Circulant matrices*. Second Edition (American Mathematical Society). AMS Chelsea Publishing. An Imprint of the American Mathematical Society, 1994, 250 p.

21. Strang, G. *Linear algebra and its applications*. 4th edition. Academic Press, USA, 2022, 487 p.

Publisher's Note: JES stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.

Submission of manuscripts: jes@meridian.utm.md

ARCHITECTURE, CIVIL ENGINEERING, ACADEMIA AND DIPLOMACY: A COMPREHENSIVE OVERVIEW OF THE WORK OF LINO BIANCO

Svetlana Oleinic *, ORCID: 0000-0002-0133-0867

Technical University of Moldova, 168 Stefan cel Mare Blvd., Chisinau, Republic of Moldova
*Corresponding author: Svetlana Oleinic, *svetlana.oleinic@arh.utm.md*

Abstract. The practice of building bridges has been attributed to the profession of architects and civil engineers. The theory underlying their design is reserved to researchers mainly in academia. Metaphorically, bridge building is a term used to establish communication and, in particular, to develop friendly relations between parties, a role played by diplomats, especially ambassadors. A contemporary professional who is an architect, an academic and a diplomat is Lino Bianco, the present ambassador of the Republic of Malta to the Republic of Moldova. Based on literature available at the library of the University of Malta and on the internet, the article presents a comprehensive overview of the professional and academic work of Professor Bianco, work which has been acknowledged at both academic and diplomatic levels. It concludes by arguing that he is essentially a renaissance man.

Keywords: *academia, Bulgaria, contemporary architecture, diplomacy, Lino Bianco, Malta, Moldova, Romania.*

Rezumat. Practica construirii de poduri a fost atribuită profesiei de arhitecți și ingineri civili. Teoria care stă la baza designului podurilor este rezervată cercetătorilor, în principal din mediul academic. Metaforic, construirea de poduri este un termen folosit pentru stabilirea comunicării și, în special, pentru dezvoltarea relațiilor de prietenie între părți, rol jucat de diplomați, în special de ambasadori. Un profesionist contemporan care este arhitect, academic și diplomat este Lino Bianco, actualul ambasador al Republicii Malta în Republica Moldova. Bazat pe literatura disponibilă la biblioteca Universității din Malta și pe internet, articolul prezintă o privire de ansamblu cuprinzătoare asupra activității profesionale și academice a profesorului Bianco, activitate care a fost recunoscută atât la nivel academic, cât și la nivel diplomatic. Se încheie argumentând că el este în esență o personalitate renascentistă.

Cuvinte cheie: *mediu academic, Bulgaria, arhitectură contemporană, diplomație, Lino Bianco, Malta, Moldova, România.*

1. Introduction

The professions of an architect and a civil engineer have long been associated with buildings, whether they be edifices or engineering structures such as bridges. There are

countless number of contemporary architects and engineers who are practising either or both professions whilst in academia. but it is not common that such professionals share also a diplomatic posting. Such is Lino Bianco who is educated in architecture and civil engineering and practises the professions, teaches these disciplines at centres of excellence and is at the same time a non-resident ambassador.

Professor Bianco is a resident full-time academic at the Department of Architecture and Urban Design of the University of Malta where he teaches and directs research on the history and theory of architecture [1]. Although not a diplomat by career, he is the ambassador of the Republic of Malta to the Republic of Moldova as from 2018 [2]. He is also the ambassador of his motherland to Romania [3] and the ambassador emeritus to the Republic of Bulgaria [4]. This article gives a biographical outline and an overview of milestones in his work. To compile this article, several sources were used: the principal ones with respect to his professional and academic work are the website Lino Bianco and Associates [5] and the Online Access Repository of the University of Malta [6] respectively.

2. Biographical note

After completing his secondary studies at The Archbishop's Minor Seminary, Lino Bianco took his post-secondary education at St Aloysius' College, both in Malta [7]. His pre-university education spans several subjects from sciences to arts to commerce to the classics although, in the Maltese system of education, one follows one of them. He obtained the General Certificate of Education at Ordinary level (MQF Level 3) [8], primarily from the University of London, not only in the typical disciplines studied at school but also in subjects such as astronomy, ancient history, Arabic, Russian and Greek literature [7, 9]. His General Certificate of Education at Advanced level (MQF Level 4), mostly from the Associated Examination Board of the United Kingdom, were in pure mathematics, applied mathematics, physics, philosophy and religion [9]. Besides reading architecture and civil engineering at the University of Malta (MQF Level 6), and earning his PhD in architecture and politics from the University of Architecture, Civil Engineering and Geodesy, Sofia (MQF Level 8), Lino Bianco read philosophy and humanistic studies at the Institute of Philosophy and Humanistic Studies (MQF Level 6), geology at the University of Leicester (MQF Level 7) and history of architecture at the prestigious Bartlett Graduate School of Architecture at University College London (MQF Level 7) [1].

In his studies in philosophy he majored in logic with a dissertation based on the applicability of Aristotelian logic to Buddhist thought which work was under the direction of Peter Serracino Inglott, Malta's principal contemporary philosopher and later rector of the University of Malta, and Vincent Riolo [10]. At the Faculty of Architecture and Civil Engineering, where he was taught by Malta's leading architect Richard England, he majored in steel structures under the tutorship of Miloslav Tocháček, a Czech pioneer in the plastic design of steel structures, and wrote his dissertation, qualifying to MQF Level 7, in concrete technology [11]. Some weeks after completion of his studies, he was awarded a prestigious scholarship by the Office of the Prime Minister of Malta, to study and undertake research in the geology of the building limestones of Malta under Martyn Pedley and Anselm Dunham [12]. His studies in architectural history at The Bartlett, covered by a British Chevening scholarship, were undertaken under the mentorship of architectural historian Adrian Forty [13]. In Sofia, he completed his doctorate on the development of Sofia under the supervision of Veselina Troeva [14]. Professor Bianco holds a warrant to practise as a "perit" from the

Republic of Malta [15], a warrant to practise as an architect from the Republic of Bulgaria [4]. Lino Bianco has a title of EUR. ING. from the Fédération Européenne d'Associations Nationales d' Ingénieurs (Brussels). He is a fellow of the Royal Society of Arts (London) and of the Geological Society (London)[1, 16].

3. Architectural practice

Professor Bianco set up his professional practice *Lino Bianco and Associates* in 1997 to offer consultancy services in architecture, urban design, and environmental management and planning [17]. His philosophy of architecture is outlined in the website: "We perceive architecture as an expression of life and as a natural extension to our environment, in which technology, construction material, settings, purpose, detail and methods of construction are unified. We merge shape, colour, space, light and time with contextual sensitivity and design, while exploiting the uniqueness of the site's characteristics. Thus, our starting point with each project lies in exploring new ways to concatenate functionality, style and aesthetics. We integrate our work in connection with culture and the place's historical and geological importance, while accentuating sustainable building and site development as key to creative and quality design" [5]. A selection of his works is shown in a screenshot from the website (figure 1).

Selected Works

Architecture

Residential	Commercial	Public	Major Projects
Apartment at Qormi	Small shop	Interpretation facility	Artisan Urban Hub, Fez
Apartments at Ħamrun	Shopping complex	Entrances to WWII shelter	Grand Harbour Network Initiative
Alterations and extension to a house of character	Extension to an operating discotheque	Restoration of disused quarry	Malta Millennium Project
Apartments at Nadur	Wine-bar	Redesign of library at the public registry	Upgrading of caravan and bungalow site
Control room at Bulgaria	Agrarian farm	Redesign of the Grand Harbour breakwater	Grand Harbour initiatives

Environmental Planning and Management

Environmental Impact Statement / Traffic Impact Statement	Environmental Planning Statement	Project Description Statement	Other
Extension of a new hospital	New hardstone quarry	Mġarr ix-Xini Regional Park	Socio-cultural initiative for the locality of Xagħra
Tuna penning farm	New softstone quarry	Ħaġar Qim and Mnajdra Temples Conservation and Interpretation Project	Agrarian and arboreal assessment
Rehabilitation facility and day hospital	Extension to an existing dairy farm		Interim engineered landfill
Malta Millennium Project	Establishment of a new dairy farm	Rehabilitation facility and day hospital	Waste transfer facility at tal-Kus
Construction of a destination port	Rehabilitation of a disused softstone quarry	Proposal for a desalination plant	Corradino fuel depot
		Gozo biological treatment plant: A critique	
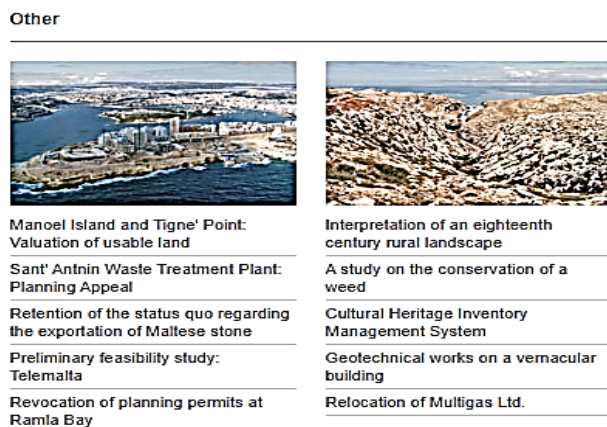
Figure 1. Lino Bianco and Associates (online version is in colour): **Selected works (©**
http://www.lino-bianco.com/selectedworks.php) [5].

They are classified under three headings: architecture; environmental planning and management; other. Residential designs include the alterations and extensions of traditional houses in Zabbar (figure 2) and Nadur (figure 3), both in Malta, which were winning entries at InterArch2012 [18] and InterArch2015 [19] respectively. Major projects include the entry competition for an artisan urban hub in Fez, Morocco (figure 4), and the upgrading of a caravan and bungalow site in Malta (figure 5). Other competition entries included the redevelopment of Via Roma, Agazzano [20], the upgrading and restoration of the "Boutilliere" in Cogne, Valle d'Aosta [21], redevelopment of the Parco della Rimembranza, Rapino [22], all in Italy, and the design of public spaces for Station Ceva Champel-Hôpital, Genève, Switzerland [23]. A comprehensive list of projects is given in table 1.

Lino Bianco is a strong proponent of values in architecture. His approach to environmental protection is innovative [25]. He is pro-development but against speculation [26]. Part of his consultancy includes challenging developments which have negative impact on heritage. For him, cultural and nature are linked. An example is his successful challenge of the multimillion touristic development of Ramla l-Ħamra, a site attributed to Homer's Calypso, which led to the revocation of its planning permission [27-29]. In parallel to running his practice, Professor Bianco was chairman of the Planning Council (Malta), member of the Planning Appeals Board (Malta), member of the Board of Directors of the Housing Authority (Malta), member of the Board of Directors of WasteServ Malta Ltd, and member of the General Services Board (Malta) [1, 24].

4. Academic research

Besides teaching in Malta, Lino Bianco held a number of teaching posts in Italy (Sapienza Università di Roma), Russia (Moscow Institute of Architecture), Bulgaria (University of Architecture, Civil Engineering and Geodesy, Sofia), Romania (Ion Mincu University of Architecture and Urbanism), Georgia (Georgian Technical University), Moldova (Technical University of Moldova), and the Philippines (University of the Philippines). He is a professor of the International Academy of Architecture [30] and a visiting professor in history and theory of urban design at the University of Architecture, Civil Engineering and Geodesy, Sofia [24]. Professor Bianco is a scholar on the architecture of the Bulgarian Capital Sofia and on Georgia, specifically on the development of poetics of architecture in this former Soviet Socialist Republic.
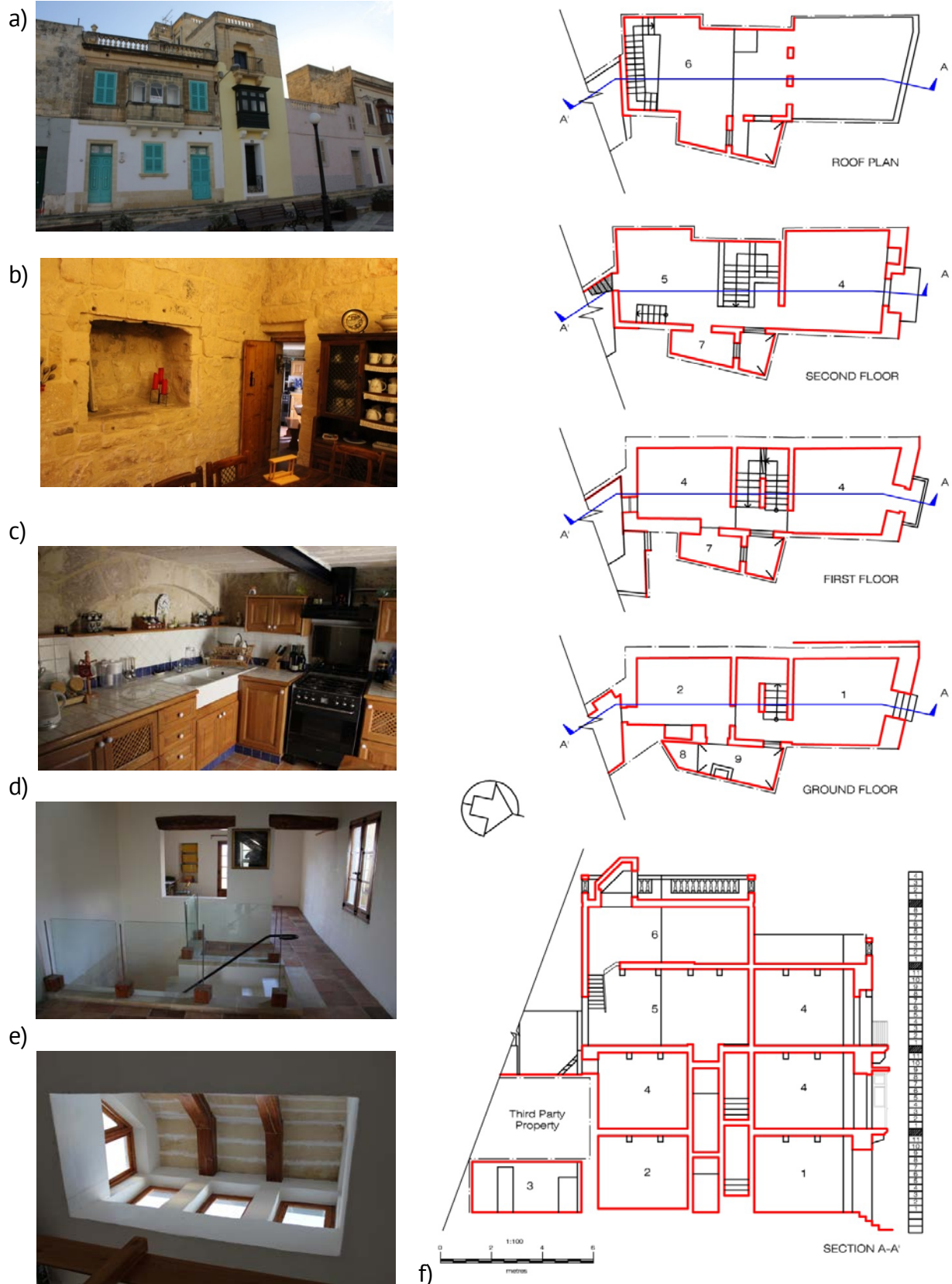
Figure 2. House of character, Zabbar (online version is in colour)**:**
a) street elevation with additional third level; b) dining room; c) kitchen; d) additional floor;
e) north lights at roof level; g) plans and section: 1– sitting room, 2– dining, 3– kitchen, 4–
bedroom, 5– living room, 6– washroom, 7– bathroom, 8– toilet, and 9– shaft [18].

a)

b)

c)
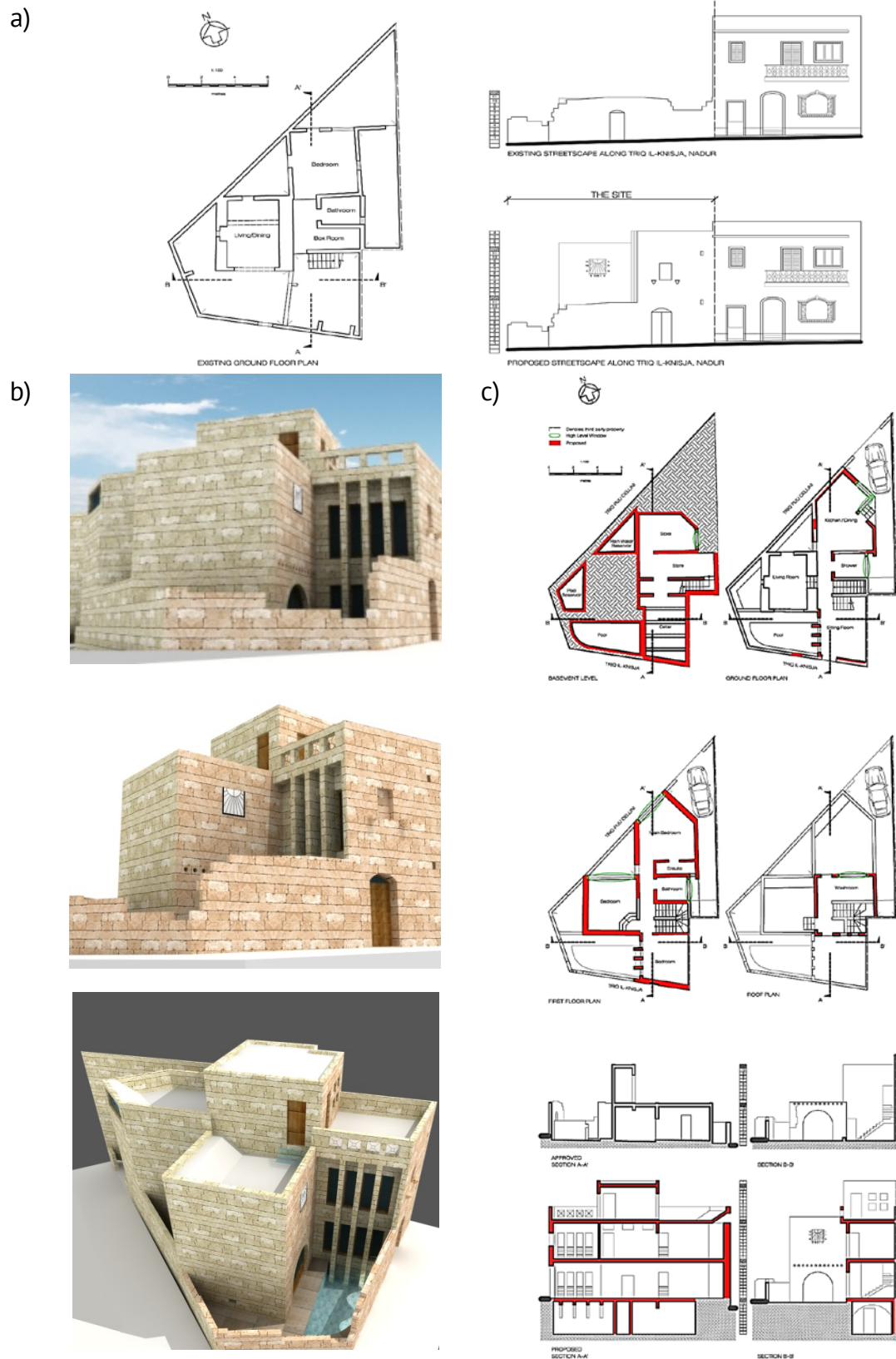
Figure 3. House of character, Nadur (online version is in colour): a) original plan, original and proposed elevation; b) computer generated models; c) design interventions [5].
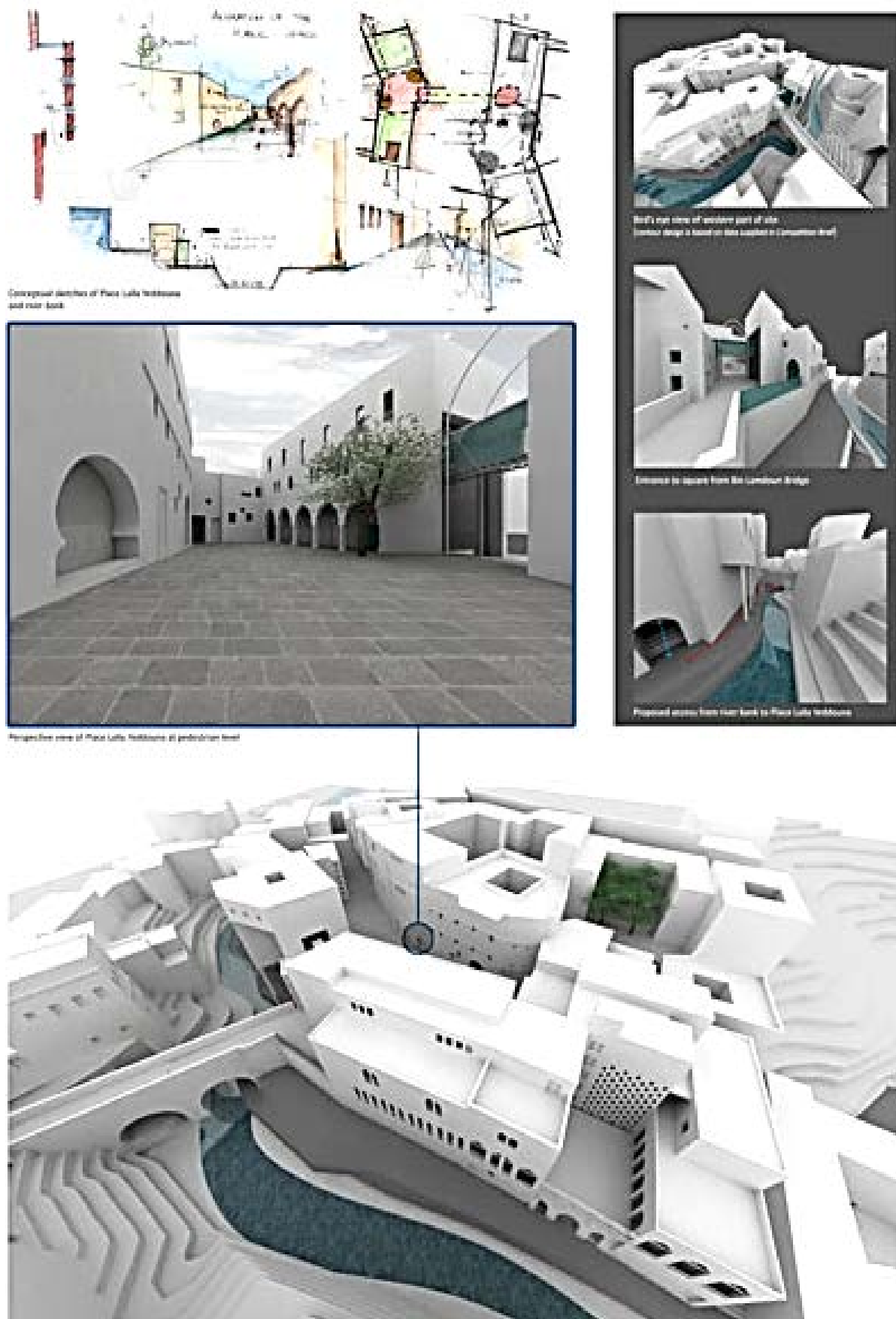
Figure 4. Artisan urban hub (online version is in colour): Competition entry for Place Lalla Yeddouna, Fez, Morocco [5].

a)

b)   c)

Figure 5. Upgrading of caravan and bungalow site in Malta (online version is in colour):
a) concept sketches; b) computer generated 3-image of proposed development of the site;
c) typical outdoor space to proposed units [5].

In recognition of the latter, he was appointed reviewer of the doctoral education programme at this university, the only PhD programme in architecture offered in the country [31]. His academic peer-reviewed publications fall under three broad categories: (I) history and theory of architecture, (II) philosophy and humanistic studies, and (III) geology.

Table 1

Participation in design projects: A selection [24]

Typology	Design project
Residential projects	1. Apartments, Nadur, Gozo (2009). 2. Single unit residence, Marsascala, Malta (2009). 3. Alterations and extension to a house of character, Zabbar, Malta (2008). 4. Block of two apartments overlying commercial premises, Birkirkara, Malta (2008). 5. Apartments, Ħamrun, Malta (2007).

	6.	Semi-detached corner dwelling, Nadur, Gozo (2006-2015).
	7.	Alterations and extension to a house of character, Zebbug, Malta (2006-2015).
	8.	Block of apartments, Xgħajra, Malta (2006–09).
	9.	Row of residential units, Xewkija, Gozo (2003–04).
	10.	Alterations and extension to a house of character, Zabbar, Malta (1998–2001).
Commercial projects	1.	Design of wine bar and cellar, Zabbar, Malta (2010).
	2.	Rehabilitation of residential unit into a shopping arcade, Paola, Malta (2008).
	3.	Redesign of a retail outlet into a catering establishment on a restricted site, Valletta, Malta (2007).
	4.	Alterations and conversion of substandard bachelor residence into shop, Qormi, Malta (2006).
	5.	Igeia clinics, Ħamrun, Malta (2005).
	6.	Offices and apartments at Rue D`Argens, Msida, Malta (2004–08).
	7.	The Limestone Heritage, Siggiewi, Malta (with Alex Torpiano and Louis Borg) (1999–2002).
	8.	Redesign of an established night club into a discotheque and pizzeria, San Gwann, Malta (1999).
	9.	Shop front for a class 4 shop, Gzira, Malta (1999).
Public projects	1.	Interpretation facility at Ta` Blankas, l/o Xewkija, Gozo (2008–2014).
	2.	Design for Grand Harbour Breakwater, Valletta, Malta (2008).
	3.	Redesigning access to Second World War shelter, Zabbar, Malta (2005–06).
	4.	Redesign of library at the Public Registry, Valletta (1998).
	5.	Landscape restoration of a disused open-pit quarry, Għarb, Gozo (1997).
Major projects	1.	L-Arka ta' Noe' Therapeutic Zoo, Siġġiewi, Malta (2014–2017).
	2.	Bamiyan Cultural Centre, Bamiyan Valley, Afghanistan (Competition entry, 2014).
	3.	Architectural design proposal for a gastronomical living museum, Valletta, Malta (2013).
	4.	Public spaces for Halte CEVA Champel-Hôpital, Geneve (Competition entry, 2012).
	5.	Upgrading and restoration of the "Boutilliere", Cogne, Valle d'Aosta, Italy (Competition entry, 2012).
	6.	Redevelopment of Via Roma, Agazzano, Italy (Competition entry, 2012).
	7.	Grand Harbour Network Initiative, Valletta, Malta (2011).
	8.	Design proposal for an artisan urban hub, Place Lalla Yeddouna, Fez, Morocco (Competition entry, 2010).
	9.	Upgrading of caravan and bungalow site, Għadira, Malta (2009).
	10.	Grand Harbour Initiatives: Twenty architectural design projects for the socio-economic regeneration of the littoral along central Malta (2008).
	11.	Design proposal for a boutique hotel, Xagħra, Gozo (2007).
	12.	Design proposals for the Malta Millennium Project at Mercury House and adjacent lands, Paceville, Malta (1997–1998).

With respect to the history and theory of architecture, a number of research themes are present: values in architecture [32-34], geocultural sensitive [35, 36] and sustainable [37-40] architectural design, poetics of architecture in Georgia [41-45], geocultural activity in seventeenth and eighteenth century Malta [46-49], the study of the Mosta Rotunda, one of the largest masonry domes in the nineteenth century [50-52], and the use of limestone in London [53-55]. Other themes are related to urbanism: philosophical approaches [56],

participatory engagement [57], and urban planning in Sofia [58, 59] and in Malta [60-62]. Other studies range from Roman architectural practice and the funding of public buildings [63], prefabrication, aesthetics and the welfare state in Britain [64] to housing and resettlement of young offenders [65] and the peculiarities and impacts of airports [66] in Malta. His multi-disciplinary arguments can be read in his papers on the history of architecture with topics ranging from Hegel's notion of gothic architecture [67], to Wolfflin-Frankl-Giedion tradition in the study of Baroque [68] to the use of masonry in Modernism [69] and to the architecture of Jovan Stefanovski [70]. His versatility in holistic approach to interpreting architecture spans a broad spectrum: from reading universal design as an applied science [71] to mapping out the cartography of an orphic life-in-death in Renais's film *L'Année dernière à Marienbad* [72].

Philosophical themes which Lino Bianco addresses vary from classical issues such as the notion of courage and wisdom in Plato [73] to Aquina's notion of the eternity of creation [74] to the application of Aristotelean logic to Buddhist thought [75]. He reads the creation narrative in Genesis as a case of environmental monitoring and audit [76]. Other publications relate to Newman's *The Dream of Gerontius* [77, 78], the celebration of the legacy of Cyril and Methodius under the patronage of the President of Malta [79-81], biographical notes of contemporary Thomistic scholars [82, 83] and Content and Language Integrated Learning [84-85]. Other occasional papers focused on the Mediterranean environment within a human rights context [86], the significance of gift exchange in Malta [87], pyrotechnics as sustainable intangible heritage [88] and parenthood post-marital failure [89].

The main topic of his geological research is the geology of the Maltese islands with his earliest paper dating to 1995 [90]. The principal theme is the Lower Globigerina Limestone, the main building stone of Malta since the Neolithic period. A comprehensive study was published in 2021 [91]. Other publications focused on (I) the techniques to determine the provenance of limestone [92] and the importance of optical microscopic investigation in such a study [93], (II) bio-retexturing in limestone [94] and (III) limestone replacement in architectural restoration [95]. His study addressed also the blue lenticular patches occurring within the Globigerina Limestone Formation [96, 97] and the outcropping of this formation at one of the most picturesque sites in Malta [98].

5. National and international acknowledgments

Professor Bianco holds a number of official acknowledgments: academic and political. He was a candidate for the Party of European Socialists (Malta) for the European Parliament elections of 2014. His political ideas stressed toleration in various contexts. He emphasised national unity in diversity [99, 100]. Other political themes on his agenda were irregular immigration [101] and the environment [102].

In recognition for his contributions in the development of architecture, the International Academy of Architecture elected him professor [1, 24]. The Georgian Technical University conferred upon him Professor Honoris Causa for his contributions to architectural practice and education [103]. At InterArch2012, his project "Home for an architect" in Malta, received a special prize for innovation in traditional architecture [18, 104]. Three years later, at InterArch2015, he received a special prize for his project "A Home of Time and Memory" [19, 105]. The submission for the proposed redevelopment of Via Roma, Agazzano, Italy placed third [24].

Lino Bianco's academic standing can be judged from the numerous scientific committees and advisory boards on which he sits. He is a member of the editorial board of several peer-reviewed academic journals which include *European Journal of Science and Theology*, *Open House International*, *Heritage and Sustainable Development*, *Urbanism– Architecture–Constructions*, and *Proceedings of Southwest State University* [1]. He is also a member of the advisory board of *The Journal of Baroque Studies* and of the scientific committee of International Experts for Research Enrichment and Knowledge (IEREK) [1]. Furthermore, he is a reviewer to a number of journals which include *European Journal of Science and Theology*, *Frontiers of Architectural Research*, *GeoJournal*, *Geographia Technica*, *Heritage Science*, *Landscape and Urban Planning*, *Materials*, *Minerals*, and *Space and Culture* [1]. Further to his appointment as reviewer of the doctoral education programme at Georgian Technical University [31], he was invited by the Consejo de Aseguramiento de la Calidad de la Educación Superior, a public constitutional body of the Republic of Ecuador, to review and evaluate publications relating to architectural history issued in the country [106].

The Order of the Madara Horseman (First Class) was awarded to him by the President of the Republic of Bulgaria for significant involvement in the advancement of friendly relations and bilateral cooperation between the Republic of Malta and the Republic of Bulgaria [107, 108]. This award was given in the presence of the President of Malta [109]. He was bestowed the Cross of Commander "Pro Merito Melitensi" by the Sovereign Military Order of Malta [110], and appointed Member of the Order of Merit by the President of the Republic of Malta for working towards a better environment and planning practice [16, 111, 112].

6. Concluding comment

The humanistic, multi-disciplinary education of Lino Bianco marks his approach to the work. Rather than being a monomath which is typical in contemporary society, he is a polymath with expertise in a number of disciplines. Although he did not follow a particular course in the liberal arts, he studied various subjects from a number of disciplines which he followed at pre, and during, university education. His pre-university education demonstrates that he is significantly self-taught. He has a wide-ranging knowledge in a number of disciples supported by academic degrees from recognised universities. As a Renaissance man, he bridges various professions to generate ideas and apply them, thus ideas acquired through his philosophy studies are applied to architecture and geological sciences. His varied publications demonstrate the versatility of his scientific reading, intellectual gymnastics in practice. Professor Bianco is cultured not only as a result of this education but also by means of his various commitments both at centres of learning and as a diplomat. His various professional appointments are a witness to his holistic approach which pushes the typical boundaries of the individual occupation. He bridges his occupations without any effort, intellectual gymnastics in action.

Conflicts of Interest: The author declares no conflict of interest.

References

1. University of Malta. *Prof. Lino Bianco*. Available online: https://www.um.edu.mt/profile/linobianco (accessed on 08.06.2022).
2. Presidency of the Republic of Moldova. *President of Moldova received credentials from five Ambassadors.* Available online: https://www.presedinte.md/eng/comunicate-de-presa/ presedintele-republicii-moldova-a-primit-scrisorile-de-acreditare-din-partea-a-cinci-ambasadori-agreati-44657 (accessed on 27.06.2022).
3. Ministry of Foreign Affairs of Romania. *List of Diplomatic Corps accredited in Romania*. Bucharest: Protocol Directorate, February 2022. Available online: https:// www.mae.ro/sites/default/files/file/anul_2022/2022_pdf/lista_corpului_diplomatic_(01_februarie_2022).pdf (accessed on 27.06.2022).
4. Ministry for Foreign and European Affairs and Trade. *Ambassador of Malta to Romania and Moldova, resident in Malta.* Available online: https://foreigncms.gov.mt//en/ embassies/nr/pages/lb/lino-bianco.aspx#:~:text=The%20Ambassador,at%20the%20University%20of%20Malta (accessed on 27.06.2022).
5. Lino Bianco and Associates. *Website.* (accessed on 08.06.2022). Available online: http://www.lino-bianco.com/
6. University of Malta. *Online Access Repository: Faculty for the Built Environment.* Available online: https://www.um.edu.mt/library/oar/handle/123456789/469 (accessed on 23.06.2022).
7. Sciberras, C. Perit ta' nisel Imqabbi maħtur Professur tal-Arkitettura. In *Festa Titulari Santa Marija Mqabba: Festa 2012*, Mqabba: Soċjetà Santa Marija u Banda Re Ġorġ V, 2012, pp. 152-153.
8. Malta Further & Higher Education Authority. *Malta Qualifications Database: Detailed information about all programmes referenced to the Malta Qualifications Framework (MQF).* Available online: http://qualifications.ncfhe.gov.mt/#/dashboard (accessed on 09.06.2022).
9. Students Selection Board. *Report of the Students Selection Board 1986-87*. Malta. p. 70.
10. Bianco, L. *On the Buddhist Syllogistic Theory*. B.A. Dissertation. Malta: Institute of Philosophy and Human Studies, 1987.
11. Bianco, L. *Pulverised fuel ash in pumped concrete*. B.E.&A. (Hons) Dissertation. Malta: University of Malta, 1992. Available online: https://www.um.edu.mt/library/oar/handle/123456789/79727 (accessed on 13.07.2022).
12. Bianco, L. *Some factors controlling the quality of Lower Globigerina building stone of Malta*. M.Sc. Dissertation. Leicester: Leicester University, 1993.
13. Bianco, L. *Limestone in post-war British architecture*. M.Sc. Dissertation. London: The Bartlett School of Architecture, University College London, 1995. Available online: https://ucl.primo.exlibrisgroup.com/discovery/fulldisplay?docid=alma990005682360204761&context=L&vid=44UCL_INST:UCL_VU2&lang=en&search_scope=MyInst_and_CI&adaptor=Local%20Search%20Engine&tab=Everything&query=any,contains,lino%20bianco&offset=0 (accessed on 13.07.2022).
14. Bianco, L. *Architecture and Politics: Contemporary and future planning policies for Sofia*. Ph.D. Thesis. Sofia: University of Architecture, Civil Engineering and Geodesy, 2011.
15. Department of Information. List of warranted periti. In: *The Malta Government Gazette.* 26 April 2022, p. 7403. Available online: https://kamratalperiti.org/wp-content/uploads/Periti-Warrant-list-Gaz-26.4.pdf (accessed on 29.06.2022).
16. Cabinet Office. *The National Order of Merit: Investiture Ceremony*. Valletta: Office of the Prime Minister, 2021. p. 12.
17. Academia. *Lino Bianco*. Available online: https://malta.academia.edu/LinoBianco (accessed on 08.06.2022).
18. Anon. A home for an architect. *The Architect* 2013, p. 17.
19. Anon. Maltese academics honoured. *Times of Malta.* 14 June 2015. Available online: https://timesofmalta.com/articles/view/Maltese-academics-honoured.572600 (accessed on 13.07.2022).
20. Bianco, L.; Poligioni, G.; Bondin, W.; Nectoux, P. E.; Vella, L.; Moran, W. *Redevelopment of Via Roma*. March 8, 2013. Available online: https://europaconcorsi4.rssing.com/chan-8499982/all_p3.html (accessed on 29.06.2022).
21. Bianco, L.; Poligioni, G.; Moran, W.; Vella, L. *Upgrading and restoration of the area called "Boutillière" in Cogne*. March 8, 2013. Available online: http://europaconcorsi.com/projects/225281-Upgrading-and-restoration-of-the-area-called-Boutilli-re-in-Cogne (accessed on 29.06.2022).
22. Bianco, L., Poligioni, G., Moran, W., Vella, L. *Riqualificazione di Parco della Rimembranza*. March 8, 2013. Available: https://europaconcorsi1.rssing.com/chan-8499987/all_p5.html (accessed on 29.06.2022).

23. Rapport du jury. *Station CEVA Champel-Hôpital – Aménagement D'Espaces Publics: Concurs de projet d`aménagement urbainà un degré en procédure ouverte.* Geneve, March 2013. Available online: https://issuu.com/massimovalente/docs/rapport-jury-ceva-champel-hopital-2 (accessed on 08.06.2022).

24. University of Architecture, Civil Engineering and Geodesy. *Prof. Dr Arch. Lino Bianco: Faculty of Architecture, Urban Planning - Guest lecturer.* Available online: https://uacg.bg/?p=109&l=2&id=1205 (accessed on 08.06.2022).

25. Muscat, J. An innovative approach to environment protection. *Times of Malta.* 30 August 2004. Available online: https://timesofmalta.com/articles/view/an-innovative-approach-to-environment-protection.113737 (accessed on 14.07.2022).

26. Micallef, M.; Bianco, L. In politics to bring change, not to be changed – Five minute interview. *Times of Malta.* 2 April 2014. Available online: https://timesofmalta. com/articles/view/lino-bianco-in-politics-to-bring-change-not-to-be-changed-five-minute.513267 (accessed on 14.07.2022).

27. Anon. Labour pledges to stop building of villas at Ramla l-Hamra. *Times of Malta.* 5 August 2007. Available online: https://timesofmalta.com/articles/view/labour-pledges-to-stop-building-of-villas-at-ramla-l-hamra.9076 (accessed on 14.07.2022).

28. Mallia, E. Ramla l-Hamra. Future danger? *Times of Malta.* 13 October 2007. Available online: https://timesofmalta.com/articles/view/ramla-l-hamra-future-danger.2245 (accessed on 14.07.2022).

29. Anon. Six-year Ramla Bay development saga comes to an end. *Malta Independent.* 9 June 2013. Available online: https://www.independent.com.mt/articles/2013-06-09/news/six-year-ramla-bay-%20development-saga-comes-to-an-end-1779859462/ (accessed on 14.07.2022).

30. International Academy of Architecture. *Member's Profile.* Available online: https://iaa-ngo.com/portfolio-posts/lino-bianco/ (accessed on 08.06.2022).

31. UM Newspoint. *UM Academic appointed reviewer of Architecture PhD programme in Georgia.* 14 February 2022. Available online: https://www.um.edu.mt/newspoint/news/2022/02/um-academic-appointed-reviewer-of-architecture-phd-programme-in-georgia (accessed on 26.07.2022).

32. Bianco, L. Defining Architecture: A qualitative research. *Annual of the University of Architecture, Civil Engineering and Geodesy* 2018, 51(1), pp. 59-66.

33. Bianco, L. Architecture, Values and Perception: Between rhetoric and reality. *Frontiers of Architectural Research* 2018, 7(1), pp. 92-99. https://doi.org/10.1016/j.foar.2017.11.003

34. Bianco, L. Social Media: Third-person perceptions on architecture. *Urbanism. Arhitectură. Construcţii* 2018, 9(3), pp. 265-272.

35. Bianco, L. Beyond Geodesign: The architecture of sitesynthesis. *Journal of Applied Engineering Sciences* 2017, 7(2), pp. 17-24. https://doi.org/10.1515/jaes-2017-0008

36. Bianco, L. Landscape Restoration and Rehabilitation: A Geocultural Design Approach. *International Journal of Environmental Science and Development* 2017, 8(3), pp. 174-181. https://doi.org/10.18178/ijesd.2017.8.3.942

37. Dragomirescu, H., Bianco, L. Tackling Sustainability from a Systemic Perspective: A Contextualized Approach. *Problemy Ekorozwoju - Problems of Sustainable Development* 2017, 12(1), pp. 31-39.

38. Bianco, L. Sustainable architectural design of the central Mediterranean. In *International Conference for Sustainable Design of the Built Environment (SDBE 2017)*, London, 20-21 December 2017, pp. 447-458. Available online: https://www.um.edu.mt/library/oar/bitstream/123456789/27588/1/Sustainable_Architectural_Design_of_the_Central_Mediterranean_2017.pdf (accessed on 11.05.2022).

39. Bianco, L. Rural and Urban Vernacular Architecture of the Mediterranean: A source for contemporary, contextual, architectural design solutions. In *The 5th Electronic International Interdisciplinary Conference*, [online]. 8-12 August 2016. Available online: https://www.um.edu.mt/library/oar/bitstream/123456789/17582/1/Rural%20and%20Urban%20Vernacula.pdf (accessed on 11.05.2022).

40. Bianco, L. The Urban Container. In *Shooting Society*, Edited by Carmel Borg and Raphael Vella, Malta: Midsea Books, 2012, pp. 46-47. Available online: https://www.um.edu.mt/library/oar/bitstream/123456789/16503/1/ShootingSociety_Edit.pdf (accessed on 11.05.2022).

41. Bianco, L. Shota Bostanashvili, architectural discourse and the foundation of poetics of architecture in Georgia. In *Papers of BAS: Humanities and Social Sciences*, 2017, 4(1), pp. 49-59. Available online: https://www.papersofbas.eu/images/papers/Papers-4-1-2017/Lino%20Bianko_article.pdf (accessed on 11.05.2022).

42. Bianco, L. From Poetics to Metapoetics: Architecture towards architecture. *Balkan Journal of Philosophy* 2018, 10(2), pp. 103-114. https://doi.org/10.5840/bjp201810212

43. Bianco, L. Pedagogy of poetics of architecture in contemporary Georgia. *Astra Salvensis* 2020, 8(15), pp. 61-74. Available online: https://www.um.edu.mt/library/oar/bitstream/123456789/67795/1/Pedagogy_of_poetics_of_architecture_in_contemporary_Georgia.pdf (accessed on 11.05.2022).

44. Bianco, L. Contemporary Georgian Architectural Theory and Practice: The legacy of Shota Bostanashvili. *ACE: Architecture, City and Environment* 2020, 15(43), 9019. http://dx.doi.org/10.5821/ace.15.43.9019

45. Bianco, L. Architecture and urban design in contemporary Georgia: A comprehensive review of the work of Shota Bostanashvili. *Historia Urbana* 2021, 29, pp. 177-199. Available online: https://www.ceeol.com/search/article-detail?id=1014972 (accessed on 11.05.2022).

46. Bianco, L. Architectural Ruins: A geocultural essay on the anatomy of buildings as illustrated by Casa Ippolito, Malta. *Heritage Science* 2021, 9(1), pp. 1-19. https://doi.org/10.1186/s40494-021-00500-9

47. Bianco, L., Cardona, K. Seventeenth century building engineering in the Central Mediterranean: A case study from Malta. *Terra Sebus. Acta Musei Sabesiensis* 2020, 12, pp. 329-353. Available online: https://www.cclbsebes.ro/wp-content/uploads/2021/03/Terra-Sebus-12-17_-L_Bianco_K_Cardona.pdf (accessed on 11.05.2022).

48. Bianco, L. Cartographic representations in early eighteenth century Baroque Malta. *Geographia Technica* 2017, 12(1), pp. 1-8. https://doi.org/10.21163/GT_2017.121.01

49. Bianco, L. Geocultural activity in seventeenth and eighteenth century Malta. *GeoJournal* 1999, 48(4), pp. 337-340. https://doi.org/10.1023/A:1007046023997

50. Bianco, L. The realisation of the Rotunda of Mosta, Malta: Grognet, Fergusson and the Episcopal objection. *European Journal of Science and Theology* 2018, 14(4), pp. 203-213.

51. Bianco, L. Building Science and Professional Ethics in Nineteenth Century Ecclesiastical Architecture in Malta. *Terra Sebus. Acta Musei Sabesiensis* 2018, 10, pp. 413-424.

52. Bianco, L. A geohistorical retrospective analysis of cultural heritage buildings: the case of Mosta Dome, Malta. *GeoJournal* 2019, 84(2), pp. 291-302. https://doi.org/10.1007/s10708-018-9861-8

53. Bianco, L. Limestone Cladding: The case of Modernist Architecture of London. *Historia Urbana*, 2017, 25, pp. 261-276. Available online: https://www.ceeol.com/search/article-detail?id=593255 (accessed on 11.05.2022).

54. Bianco, L. Portland Stone and the Architectural History of London: an overview. *Mesto a Dejiny - The City and History*, 2017, 6(1), pp. 33-47. Available online: http://cejsh.icm.edu.pl/cejsh/element/bwmeta1.element.cejsh-be8a588b-b24c-45d8-8d01-1591347aa603 (accessed on 11.05.2022).

55. Bianco, L. Limestone in post-war British architecture: is it a plea for a return to Pugin? *Melita Theologica* 1998, 49 (2), pp. 71-80. Available online: https://www.um.edu.mt/library/oar/bitstream/123456789/15436/1/Limestone%20in%20post%20war%20british%20architecture.pdf (accessed on 11.05.2022).

56. Bianco, L. Philosophical approaches to urbanism: A way for urban planning and design education. In: *Modern problems in construction: setting goals and ways to solve them*, Kursk, 22-24 May 2019, pp. 10-17. Available online: https://www.elibrary.ru/item.asp?id=37641169 (accessed on 11.05.2022).

57. Bianco, L. Participatory Engagement in Urban Design Process: The Case of an Urban Settlement in Malta. *Spatium* 2016, 35, pp. 71-78. https://doi.org/10.2298/SPAT1635071B

58. Bianco, L. The Contemporary Urban Planning Framework in Bulgaria. *Id-Dritt* 2011, 21, pp. 303-328. Available online: https://www.um.edu.mt/library/oar/bitstream/123456789/28116/1/The_Contemporary_Urban_Planning_Framework_in_Bulgaria_2011.pdf (accessed on 11.05.2022).

59. Bianco, L. The Ring Metaphor and the Spirit of Sofia. *Melita Theologica* 2010, 61(1), pp. 27-44.

60. Bianco, L. Development briefs and development planning in Malta. *Bank of Valletta Review* 1999, 19, pp. 68-86.

61. Bianco, L. Malta: housing and real estate, 1980-2005. *Architectural Design* 2006, 76, pp. 76-81. https://doi.org/10.1002/ad.269

62. Bianco, L. Valletta: A City in History. *Melita Theologica* 2009, 60(2), pp. 3-20.

63. Bianco, L. Roman Architectural Practice and the Funding of Public Buildings. *Malta Archaeological Review* 2007, 8, pp. 18-24. Available online: https://www.um.edu.mt/library/oar/handle/123456789/15944 (accessed on 11.05.2022).

64. Bianco, L. Prefabrication, Aesthetics and the Welfare State: The Case for the Post-War British Public Schools. *Malta Review of Educational Research* 2013, 7(1), pp. 73-88. Available online: http://www.mreronline.org/wp-content/uploads/2013/07/MRERV7I1P41.pdf (accessed on 11.05.2022).

65. Bianco, L. Housing and Resettlement of Young Offenders: The Case for a Cross-Government Action Plan for Malta. *Varstvoslovje, Journal of Criminal Justice and Security* 2016, 18(4), pp. 438-450.

66. Bianco, L. The peculiarities and impacts of airport: The case for Malta International Airport. *Journal of Engineering Science* 2021, 38(1), pp. 100-110. https://doi.org/10.52326/jes.utm.2021.28(1).10

67. Bianco, L. Hegel's notion of gothic architecture. *Melita Theologica* 1996, 47(1), pp. 3-15.

68. Bianco, L. In Defence of Baroque: The Wolfflin-Frankl-Giedion Tradition. *The Journal of Baroque Studies*, 2016, 1(4), pp. 5-20. Available online: https://www.um.edu.mt/library/oar/handle/ 123456789/15938 (accessed 10.05.2022).

69. Bianco, L. Masonry and the Modernist Ethic. *The Masonry Society Journal* 2000, 18(2), pp. 95-99.

70. Bianco, L. From idea to realisation: The architecture of Jovan Stefanovski. *Revista Arta* 2021, 30(1), pp. 87-97. https://doi.org/10.52603/arta.2021.30-1.13

71. Bianco, L. Universal design: from design philosophy to applied science. *Journal of Accessibility and Design for All* 2020, 10(1), pp. 70-97. https://doi.org/10.17411/jacces.v10i1.249

72. Catania, S., Bianco, L. L'Année dernière à Marienbad and the cartography of an orphic life-in-death: the modern Katabasis of Resnais. *Melita Theologica* 2002, 53(1), pp. 81-100.

73. Bianco, L. The unity of courage and wisdom in Plato's Protagoras. *Philosophia: E-Journal of Philosophy and Culture* 2016, 11, pp. 1-7.

74. Bianco, L. Creation, Beginning and Time in the Summa Theologiae: Why creation does not imply the beginning of the universe? *Meta* 2021, 8(1), pp. 103-120.

75. Bianco, L. Buddhist Syllogistic Theory. *Melita Theologica* 1998, 49(1), pp. 3-18.

76. Bianco, L. The creation narrative in Genesis. A case of environmental monitoring and audit. *European Journal of Science and Theology* 2021, 17(2), pp. 113-124.

77. Bianco, L. J.H. Newman's The Dream of Gerontius and Karm Scerri. *Journal of Maltese History* 2016, 5(1), pp. 83-91. Available online: https://www.um.edu.mt/library/oar/handle/123456789/18114 (accessed on 11.05.2022).

78. Bianco, L. Theology applied to music: The Dream of Gerontius revisited. *The Elgar Society Journal* 2016, 19(5), pp. 26-34.

79. Bianco, L. *Celebrating the legacy of Cyril and Methodius: Apostles of culture and education*, Office of the President, Malta, 2016. Available online: https://www.um.edu.mt/library/oar/bitstream /123456789/16766/1/Celebrating_the_legacy_of_Cyril_and_Meth.pdf (accessed on 11.05.2022).

80. Bianco, L. *Celebrating the legacy of Cyril and Methodius with special reference to historical iconography*, Office of the President, Malta, 2017. Available online: https://www.um.edu.mt/library /oar/bitstream/123456789/27530/1/Celebrating_the_legacy_of_Cyril_and_Methodius2017.pdf (accessed on 11.05.2022).

81. Bianco, L. Presentation of a Bulgarian icon to the Head of State of Malta: History in the making. *Problems of art* (Art Studies Quarterly) 2018, 51(2), pp. 55-57. Available online: https://www.um.edu.mt/library/oar/bitstream/123456789/38971/1/Presentation_of_a_Bulgarian_icon_to_the_Head_of_State_of_Malta%20_2018.pdf (accessed on 11.05.2022).

82. Bianco, L. Daniel Callus, a Maltese Dominican scholar at Oxford. *Journal of Maltese History* 2019/2020, 6(1), pp. 90-100. Available online: https://www.um.edu.mt/library/oar/handle/123456789/67877 (accessed on 09.05.2022).

83. Bianco, L. Transcending pain: A glimpse into the life of philosopher Ioan Miclea. *Astra Salvensis* 2021, 9(17), pp. 141-161.

84. Bianco, L.; Andonova, I. Content and Language Integrated Learning applied to teaching chemistry: A case study from Eastern Europe. Chemistry. *Journal of science education* 2020, 29(3), pp. 283-298.

85. Bianco, L.; Andonova, I.; Buhagiar, A. CLIL applied to pre-university chemistry teaching in English: Bulgaria as a case study. *Journal of Social Sciences* 2021, 4(3), pp. 7-17. https://doi.org/10.52326/jss.utm.2021.4(3).01

86. Aqulina, K.; Bianco, L. The Mediterranean Environment within a Human Rights Context: Towards the emergence of Mediterranean environmental human rights law and the implementation of an effective enforcement machinery therefor. *Mediterranean Journal of Human Rights* 2010, 14(1), pp. 13-33.

87. Bianco, L. The significance of gift exchange in the Maltese Islands. *Insula - The International Journal of Island Affairs* 2000, 9(1), pp. 65-67.

88. Bianco, L.; Delicata, S. Pyrotechnics as sustainable intangible heritage. In: *Proceedings of the Thirteenth International Symposium on Fireworks*, Malta, 23-27 April 2012, pp. 43-56. Available online: https://www.um.edu.mt/library/oar/handle/123456789/21204 (accessed on 11.05.2022).

89. Bianco, L. Parenthood post-marital failure is not always a gender-neutral issue: A personal recollection. *Astra Salvensis* 2021, 9(18), pp. 201-211.

90. Bianco, L. The industrial minerals of the Maltese islands: a general introduction. *Hyphen* 1995, 7(3), pp. 111-118. Available online: https://www.um.edu.mt/library/oar/handle/123456789/17583 (accessed on 11.05.2022).

91. Bianco, L. Mineralogy, geochemistry and texture of the Lower Globigerina Limestone used in the built heritage. *Minerals* 2021, 11, 740 (2021). https://doi.org/10.3390/min11070740

92. Bianco, L. Techniques to determine the provenance of limestone used in Neolithic architecture of Malta. *Romanian Journal of Physics* 2017, 62 (1-2), 901. Available online: https://rjp.nipne.ro/2017_62_1-2/RomJPhys.62.901.pdf (accessed on 11.05.2022).

93. Koleva-Rekalova, E.; Bianco, L. Importance of optical microscopic investigation in provenance studies and quality characteristics of both historical and natural Lower Globigerina building limestone of Malta. *Review of the Bulgarian Geological Society* 2018, 79(3), pp. 93-94.

94. Bianco, L. Bio-retexturing in limestone used in the built heritage of Malta. *Romanian Reports in Physics* 2017, 69(4), 802. Available online: http://www.rrp.infim.ro/IP/AN802.pdf (accessed on 11.05.2022).

95. Bianco, L. Limestone replacement in restoration: the case of the church of Santa Maria (Birkirkara, Malta). *International Journal of Conservation Science* 2017, 8(2), pp. 167-176. Available online: https://ijcs.ro/public/IJCS-17-17_Bianco.pdf (accessed on 11.05.2022).

96. Bianco, L. Mineralogy and geochemistry of blue patches occurring in the globigerina limestone formation used in the architecture of the Maltese islands. *Comptes rendus de l'Academie Bulgare des Sciences* 2017, 70(4), pp. 537-544. Available online: https://www.um.edu.mt/library/oar/handle/123456789/20509 (accessed on 11.05.2022).

97. Bianco, L. Petrological characteristics of blue lenticular patches occurring in the Lower Globigerina building limestone of Malta. *Romanian Journal of Materials* 2018, 48(1), pp. 115-120.

98. Bianco, L. Petrological, mineralogical and geochemical characteristics of the Globigerina limestone outcropping at Fomm ir-Riħ, Malta. *Comptes rendus de l'Academie Bulgare des Sciences* 2020, 73(7), pp. 985-991.

99. Bianco, L. In the European Parliament to lead. In: *Malta Independent.* 6 October 2013. Available online: https://www.independent.com.mt/articles/2013-10-06/opinions/in-the-european-parliament-to-lead-2808610827/ (accessed on 05.07.2022).

100. Bianco, L. Unity in diversity: Malta and the EU. In: *Malta Independent.* 24 November 2013. Available online: https://www.independent.com.mt/articles/2013-11-24/opinions/unity-in-diversity-malta-and-the-eu-3274637314/ (accessed on 05.07.2022).

101. Carabott, S. Europe 'needs non-EU migrants'. In: *Times of Malta.* 8 March 2014. Available online: https://timesofmalta.com/articles/view/Europe-needs-non-EU-migrants-.509696 (accessed on 05.07.2022).

102. Attard Bezzina, D. L-ispekulazzjoni u mhux l-iżvilupp, it-theddida għall-ambjent - Lino Bianco. In: *iNews Malta.* 6 October 2013. Available online: https://web.archive.org/web/20150924034609/http://www.inewsmalta.com/dart/20131006-ispekulazzjoni-mhux-vilupp-it-theddida-all-ambjent-lino-bianco (accessed on 05.07.2022).

103. Georgian, A. Technical University. *Maltese Architect Lino Bianco conferred Honoris Causa.* 19 March 2015. [online]. 2015. Available online: https://gtu.ge/Eng/News/?ELEMENT_ID=4219 (accessed on 19.07.2022).

104. International Academy of Architecture. *InterArch2012: Exhibition-competition of architectural projects and works.* 12 May 2012. Available online: https://iaa-ngo.com/2012/12/interarch-2012/ (accessed on 13.07.2022).

105. International Academy of Architecture. *InterArch2015: Exhibition-competition of architectural projects and works.* 17-20 May 2015. Available online: https://iaa-ngo.com/2015/12/interarch-2015/ (accessed on 13.07.2022).

106. UM Newspoint. *Resident academic engaged by the Council for Quality Assurance of Ecuador.* 3 December 2020. Available online: https://www.um.edu.mt/newspoint/news/2020/12/lino-bianco-new-appt (accessed on 26.07.2022).

107. Republic of Bulgaria. Decree № 20 of 31.01.2018 on awarding Mr Lino Bianco – Ambassador Extraordinary and Plenipotentiary of the Republic of Malta to the Republic of Bulgaria, with the Order "Madara Horseman" first degree. In: *State Gazette*, 2018, 12 (6). Available online: https://www.ciela.net/svobodna-zona-darjaven-vestnik/document/2137180750/issue/5675/ukaz-%E2%84%96-20--ot-31012018-g-za-nagrazhdavane-na-g-n-lino-bianko---izvanreden-i-palnomoshten-poslanik-na-republika-malta-v-republika-balgariya-s-orden- (accessed on 28.06.2022).

108. Department of Information. Government Notice No. 413: Ġieħ ir-Repubblika Act (Cap 251). In: *The Malta Government Gazette*. 10 April 2018, p. 3344. Available online: https://www.gov.mt/en/Government/DOI/Government%20Gazette/Documents/2018/04/Government%20Gazette-%2010th%20April.pdf (accessed on 28.06.2022).

109. Anon. UM academic honoured for his significant contribution to bilateral cooperation with Bulgaria. In: *Malta Independent*. 22 February 2018. Available online: https://www.independent.com.mt/articles/2018-02-22/education/UM-academic-honoured-for-his-significant-contribution-to-bilateral-cooperation-with-Bulgaria-6736185179 (accessed on 28.06.2022).

110. Department of Information. Government Notice No. 483: Ġieħ ir-Repubblika Act (Cap 251). In: *The Malta Government Gazette*. 23 April 2019, p. 3977. Available online: https://www.gov.mt/en/Government/DOI/Government%20Gazette/Documents/2019/04/Government%20Gazette%20-%2023rd%20April.pdf (accessed on 28.06.2022).

111. Department of Information. Government Notice No. 1606: Ġieħ ir-Repubblika Act (Cap 251). In: *The Malta Government Gazette*. 13 December 2021, p. 13197. Available online: https://www.gov.mt/en/Government/DOI/Government%20Gazette/Documents/2021/12/Government%20Gazette%20-%2013th%20December.pdf (accessed on 28.06.2022).

112. Sansone, K. Republic Day Honours: Malta's great decorated by President. In: *Malta Today*. 13 December 2021. Available: online https://www.maltatoday.com.mt/news/national/113787/republic_day_honours__maltas_great_decorated_by_president#.Yw3KqnZByM (accessed on 05.07.2022).

Publisher's Note: JES stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.

Submission of manuscripts: jes@meridian.utm.md

OPEN ACCESS   CC BY

CALORIFIC VALUE OF PELLETS PRODUCED FROM RAW MATERIAL COLLECTED FROM BOTH SIDES OF THE RIVER PRUT

Grigore Marian [1*], ORCID: 0000-0002-9975-2522,
Ianuș Gelu [2], ORCID: 0000-0002-2998-0992,
Andrei Gudîma [1], ORCID: 0000-0002-7250-8423,
Boris Nazar [1], ORCID: 0000-0002-9262-5061,
Bogdan Istrate [2], ORCID: 0000-0003-0817-3523,
Alexandru Banari [1*], ORCID: 0000-0001-9132-610X,
Andrei Pavlenco [1], ORCID: 0000-0002-3320-2907,
Nicolae Daraduda [1], ORCID:0000-0001-5683-0431

[1] Solid Biofuels Laboratory, Technical University of Moldova, 44 Mircesti Str., Chisinau, Republic of Moldova
[2] Faculty of Mechanical Engineering, Gheorghe Asachi Technical University of Iasi, 61-63 D. Mangeron Blvd., Iasi, Romania, 700050
*Corresponding authors: Grigore Marian, marian@uasm.md
Alexandru Banari, a.banari@uasm.md

Abstract. The production of pellets has recently experienced a sudden upward trend worldwide, including the Republic of Moldova and Romania, especially due to the energy crisis caused by the war in Ukraine and rapid depletion of fossil energy sources. The widespread use of pellets has several reasons: it is an eco-friendly fuel, it allows the automation of the combustion process, and it can be easily produced in any region that has enough raw materials. The calorific value of pellets is the main characteristic feature that determines their price and is used in combustion engineering to find out the thermal efficiency of combustion plants. This paper synthesizes the results regarding the calorific value of the pellets produced from different types of plant biomass specific to the Republic of Moldova and the counties adjacent to the river Prut in Romania. The research has been carried out within the Solid Biofuels Laboratory, SAUM using standard methods of testing densified solid biofuels. The experimental results obtained in this study showed that the pellets produced from arboriculture residues, residues generated from vine pruning, table varieties, as well as those produced from sunflower seed husk have a calorific value equal to or greater than 16.5 MJ/kg, i.e., they meet ENPlus 3 requirements.

Keywords: wood pellet, calorific value, biomass, agricultural residues, energy crops, biomass conditioning, combustion.

Rezumat. Producerea de peleți în lume, inclusiv în Republica Moldova și în România cunoaște un trend brusc ascendent în ultimii ani, în special în condițiile crizei energetice cauzate de războiul din Ucraina și epuizarea galopantă a surselor fosile de energie. Folosirea pe larg al peleților are mai multe motivații dintre care faptul că este un combustibil non ofensiv

mediului, permite automatizarea procesului de combustie, este accesibil pentru producere în orişice zonă unde există suficientă materie primă. Valoarea calorifică a peleţilor este principala caracteristică care determină preţul peleţilor şi care este folosită în ingineria arderii pentru stabilirea randamentului termic al instalaţiilor de combustie. Această lucrare prezintă o sinteză a rezultatelor referitoare la valoarea calorifică a peleţilor produşi din diferite tipuri de biomasă vegetală specifică Republicii Moldova şi judeţelor adiacente râului Prut din România. Cercetările au fost efectuate în cadrul Laboratorului de Biocombustibili Solizi, UASM cu folosirea metodelor standarde de testare a biocombustibililor solizi densificaţi. Rezultatele experimentale obţinute în acest studiu au arătat că peleţii produse din reziduuri de arboricultură, reziduuri generate de tăierea viţei-de-vie, soiuri de masă, precum şi cele produse din coajă de seminţe de floarea soarelui au o putere calorică egală sau mai mare de 16,5 MJ/kg, adică îndeplinesc cerinţele ENPlus 3.

Cuvinte cheie: *peleţi de lemn, valoare calorifică, biomasă, reziduuri agricole, culturi energetice,*
condiţionare biomasă, peletizare, combustie.

1. Introduction

This paper presents the qualitative study on determining the calorific value of the pellets produced from different types of plant biomass produced from agricultural crops and energy crop species that have achieved a more pronounced spread in agricultural households located on both banks of the river Prut, both in the Republic of Moldova and Romania.

Pellets are solid biofuels produced from plant biomass with or without additives, dehydrated and compressed to at least twice the energy density of the biomass in the chopped form with a random length of 5-40 mm and a diameter of up to 25 mm with irregular ends [1].

The demand for biomass energy, including in the form of pellets, is experiencing a rapid rise especially due to the energy crisis and increased prices of fossil fuels, mainly gas and coal. The increased consumption of pellets has led to the diversification of indigenous raw material sources recording characteristics meeting the international standard requirements. As the result, biomass from agricultural residues, wood co-products and energy crops, along with their combination, represents the most important source of raw materials used in the production of pellets in the areas adjacent to the river Prut both in the Republic of Moldova and Romania.

Qualitative assessment of pellets produced from the biomass specific to certain regions, correlated with the search for solutions for the efficient use of local energy resources, is crucial nowadays, thus contributing to the continuous development of the densified solid biofuels production with characteristics meeting the requirements of international standards.

The use of indigenous plant biomass as a raw material for pellets' production contributes to reduce the dependence of the energy sector on fossil fuels, concomitantly representing a stimulus for creating employment opportunities in rural areas [2-5].

Since the production of pellets consumes more energy than the production of split wood, wood chips or straw bales, the real benefits of pellets can be obtained when their quality characteristics correlate with the requirements of the international standards stated by the standard ISO 17225- 2 [1].

The quality of pellets depends on a number of factors that are grouped into two categories: the quality of raw material and the conditioning and pelletizing variables [3, 6].

The quality of raw material, used in the production of pellets, depends on the biomass origin, the way it is dried, the content of impurities, the collection period, the storage period, the particle sizes, etc. [6-8].

A series of procedures are used to ensure the quality of raw material, including the formation of mixtures from different biomass types with complementary characteristics, the use of different organic additives, the thermochemical treatment of biomass and finished product, etc.

Conditioning of raw material and palletization itself involve ensuring all the requirements related to the conditioning of the raw material before it is processed (moisture content, purity and biomass granulation) and the technological palletization methods (compression pressure, temperature of granule formation, roller speed), constructive parameters of the pelletizing matrix (diameter and length of the channels, the gap between the rollers and the matrix), etc.

Therefore, the purpose of this research is to fill the information gap regarding the energy potential of pellets produced from biomass specific to the regions on both banks of the river Prut by presenting a clear understanding of their calorific value.

In order to achieve the proposed goal a series of pellets, produced from different biomass types have been analysed from the point of view of their energy potential. The studies have been carried out using standard methods, applied in the field of qualitative assessment of densified solid biofuels.

2. Materials and Methods

Sampling. The biomass has been collected from various agricultural farms in the Republic of Moldova and from the counties of Iasi, Botosani, Vaslui and Galati in Romania. There have been used 56 samples of pellets produced from the biomass of distinct species of agricultural residues and energy crops. The biomass has been collected in different harvest periods and conditioned directly in the field or under the drying room conditions of the Solid Biofuels Laboratory of the State Agrarian University of Moldova.
The conditioning directly in the field has been carried out only for some types of arboreal species residues by their natural drying at the edge of the field during summer and some biomass types generated from energy crops and coarse woody debris by shredding directly in the field using Murena portable shredder (see figure 1).

Figure 1. Coarse shredding of Miscanthus biomass using Murena shredder attached to the MT 8E2 tractor.

Energy crop samples have been collected manually (see figure 2).

Figure 2. Miscanthus sample collection.

The experimental samples have been collected in accordance with the requirements of standard ISO 18135 [9], and the preparation of the samples has been carried out in accordance with standard ISO 14780 [10]. The pellets have been produced at the mini pelletizing line within the SAUM Solid Biofuels Laboratory.

Establishing the calorific value of pellets. The gross calorific value has been measured at constant volume using the IKA C6000 isoperibolic calorimeter. Measurements and calculations have been performed according to the standard EN ISO 18125 [11] (see figure 3). Five replicates have been used during this experiment, based on which the standard deviation has been determined.

Figure 3. Determining the gross calorific value by IKA C6000 isoperibolic calorimeter in the Solid Biofuels Laboratory, SAUM.

Pellet samples, before being tested, had been crushed in a Retsch SM 100 hammer mill. Sample weighing has been done using the analytical scales AS 220/C/2, RADVAG (see Figure 4). All samples used to determine the calorific value have been dehydrated by drying in the German Memmert UNBU oven. The oven is equipped with a control system and stable temperature maintenance within the limits established by the standard and with a ventilation system of the precinct depending on the granulation of the samples used for testing.

The net calorific value has been determined for dehydrated pellets, i.e., on a dry basis and for the pellets with a moisture content equal to 10% using the following formulas:

$$q_{p,net,d} = q_{V,gr,d} - 212,2 \times w(H)_d - 0,8 \times [w(O)_d + w(N)_d] , \qquad (1)$$

$$q_{p,net,m} = q_{p,net,d} \times (1 - 0,01M) - 24,43 \times M, \qquad (2)$$

where:

$q_{(p,net,d)}$ is net calorific value at constant pressure of the dry (moisture-free) fuel, J/g.

$q_{p,net,m}$ is net calorific value at constant pressure of the biofuel with moisture content M, J/g.

$q_{v,gr,d}$ is gross calorific value at constant volume of the biofuel of the dry (moisture-free) fuel (dry basis, in the dry matter), J/g.

$w(H)_d$; $w(O)_d$; $w(N)_d$ are, respectively, the hydrogen, oxygen and nitrogen content, in percentage by mass of the moisture-free biofuel, %.

3. Essential information regarding the calorific value of pellets and its role in the qualitative assessment of pellets for residential and industrial use

The main qualitative characteristic of the pellets that mostly interests the beneficiaries is the calorific value, i.e., the burning power. Historically, the calorific value is used in thermal and combustion engineering to calculate thermal efficiency of power plants and represents the amount of energy released as heat when a unit of mass of fuel burns completely and perfectly and the flue gases cool down to 25 ℃. Thus, the higher the calorific value of the pellets, the more energy we get per unit of mass. This is the main reason why the calorific value determines the price of pellets.

Figure 4. Pellet crushing and sample dosing by weighing.

The calorific value of pellets is generally expressed using several values. Thus, quality certificates, as a rule, include the gross and net calorific values on a dry basis and at the reception moisture level. Studies often specify the net calorific value at a moisture content equal to 10%, the maximum value recommended by the ENPlus 3 standards.

Gross calorific value or higher thermal value, also includes the heat obtained from the transformation phases of condensation and solidification, being determined by returning all combustion products to the reference temperature (25 °C). In other words, when the quality of pellets is assessed by means of the gross calorific value, it is also taken into account the heat released by the water vapour produced in the combustion process and transformed into a liquid.

The gross calorific value for both solid and liquid fuels is measured at constant volume, and it is measured at constant pressure for gaseous fuels.

The gross calorific value of pellets at constant volume represents the absolute value of the specific combustion energy, in joules, for a unit of mass of the pellets burned in an oxygen environment in a calorimeter bomb under special conditions. Combustion products usually consist of solid ash, gaseous oxygen, nitrogen, carbon dioxide, sulfur dioxide, liquid water (in balance with its vapor) saturated with carbon dioxide under bomb reaction conditions, all at reference temperature (25 °C) [9].

The net calorific value is determined by subtracting the amount of heat produced as a result of vaporization phase transformations from the gross calorific value. In this case, the energy consumed for water vaporization is not considered as heat-producing energy. Thus, the net calorific value assumes that the vaporization heat is latent and is not recovered in the pellet combustion process.

The net calorific value can be calculated for constant volume or pressure conditions. Standard ISO 18125 recommends using the net calorific value as operative heat for constant pressure conditions, i.e. atmospheric pressure.

	0	5	10	15	20	25	30	35	40	45	50	55	60	65	70	75
q V, gr, d =15MJ/kg	13.7	12.9	12	11.2	10.4	9.63	8.82	8.02	7.21	6.41	5.6	4.8	3.99	3.19	2.39	1.58
qV, gr, d = 18MJ/kg	16.7	15.7	14.7	13.8	12.8	11.9	10.9	9.97	9.01	8.06	7.1	6.15	5.19	4.24	3.29	2.33
qV, gr, d = 20MJ/kg	18.7	17.6	16.5	15.5	14.4	13.4	12.3	11.3	10.2	9.16	8.1	7.05	5.99	4.94	3.89	2.83
qV, gr, d = 22MJ/kg	20.7	19.5	18.3	17.2	16	14.9	13.7	12.6	11.4	10.3	9.1	7.95	6.79	5.64	4.49	3.33
qV, gr, d = 25MJ/kg	23.7	22.4	21	19.7	18.4	17.1	15.8	14.5	13.2	11.9	10.6	9.3	7.99	6.69	5.39	4.08

Moisture content (%)

Figure 5. Dependence of net calorific values of pellets on the moisture content. *Data are presented for the following conditions: H = 6.2%, N = 0.5%, S = 0.05%, O = 42 %.*

This value is calculated by subtracting the losses of latent vaporization heat associated with the pellet moisture and the water content formed during combustion from the gross calorific value (see formulas 1 and 2 in section 2).

The net calorific value at reception represents a specific feature meeting the ENPlus requirements, as it expresses the real amount of heat obtained from the complete and perfect combustion of a pellet unit without the evidence of vaporization heat, i.e., the amount of heat obtained from the combustion of pellets in thermal power plants used nowadays for residential and industrial purposes. According to standard ISO 17225-2 [2], the net calorific value at reception of pellets for all ENPlus property classes both for residential and industrial uses should not be less than 16.5 MJ/kg.

It should be noted that the net calorific value is greatly influenced by the moisture content of pellets. This can be seen in Figure 5, which shows the dynamics of the net calorific value depending on the moisture content for pellets with different gross calorific values on a dry basis. It is also mentioned that the percentages in which the moisture content is expressed refer to the mass participation. Thus, the pellets include 0.1 kg of water per kg of fuel at a moisture content of 10%.

The second factor that greatly influences the calorific value of pellets is the quality of the raw material. This aspect is studied in the following chapter.

4. Results and Discussion

As mentioned above, the calorific value of pellets is greatly influenced by the raw material from which they are made. Organic substances such as cellulose, hemicellulose and lignin vary quite significantly depending on the type of plant biomass and on the plant parts from which they are taken [5, 10].

Table 1 presents the net calorific value of the pellets produced from various types of plant biomass collected from different regions in the Republic of Moldova and the counties of Iasi, Botosani, Vaslui and Galait, Romania. The calorific value is provided for a moisture content equal to 10%. The data represent the average ± standard deviation determined for 5 parallel measurements carried out in the same laboratory by the same operator for the same analysed samples.

It is known that agricultural residues represent the most accessible source for pellet production both in the Republic of Moldova and Romania [3, 13 - 17]. However, not all agricultural residues can be used directly as raw material, because some of their characteristics do not meet the requirements of the international standards, especially the calorific value.

The results presented in this paper show that the pellets produced from biomass derived from herbaceous agricultural residues have a net calorific value at 10% moisture lower than 16.5 MJ/kg (except those produced from sunflower seed husk). At the same time, tree biomass pellets have a much higher calorific value, varying within the limits of (16.76 ± 0.22) MJ/kg for the pellets produced from quince residues and 18.40 ± 0.18 for the ones produced from cherry residues. Pellets produced from vine residues, sampled table varieties, both in the Republic of Moldova and Romania also have recorded a calorific value within the limits stipulated by the ENPlus3 requirements. The burning power of pellets produced from technical varieties of vine residues, is somewhat lower; their calorific value is close to the one recommended by the ENPlus 3 standards.

The residues generated from sea buckthorn bushes have good prospects to be used as a raw material for the production of pellets; their calorific value is (16.62 ± 0.15) MJ/kg. The pellets produced from the residues of blackberry and blackcurrant bushes marked values lower than 16.5 MJ/kg, so this biomass can only be used in combination with other types of biomass with higher calorific values, for example residues of fruit bushes.

An important source of raw material for the production of pellets is the one generated by energy crops. A vast variety of energy crops requires more thorough research regarding the possibility of using the biomass resulting from their cultivation as a raw material for the production of densified solid biofuels.

This study focuses on determining the calorific value of pellets produced from plant biomass generated from several species of energy crops with a real perspective of use as raw material under the conditions of the Republic of Moldova and the counties in Romania adjacent to the river Prut.

The analysis of values of the measured parameters of pellets produced from energy crops biomass, presented in Table 1, made it possible to determine the difference in the net calorific value at 10% moisture of 1.84 MJ/kg between the pellets with the highest calorific value (produced from energy willow with $q_{p,\ net,\ m=10\%}$.= 17.45 ± 0.17 MJ/kg) and those with the lowest calorific value (produced from the reed sampled from the Danube delta with $q_{p,\ net,\ m=10\%}$.=15.61 ± 0.23 MJ/kg).

The results presented above show that the energy willow biomass can be recommended to be used as a raw material in the production of solid densified biofuels from the point of view of the energy potential.

Table 1

Calorific value of pellets produced from biomass collected in the regions adjacent to the river Prut

Raw material used	Biomass type	$q_{V,\ gr,\ d}$	$q_{p,\ net,\ d}$	$q_{p,\ net,\ m=10\%}$
		MJ/kg		
Sunflower seed husk	Seed husk	20.24 ± 0.19	18.91 ± 0.19	16.78 ± 0.17
Winter and spring wheat	Straw	18.48 ± 0.18	17.15 ± 0,18	15.19 ± 0,16
Winter and spring barley	Straw	18.47 ± 0.22	17.14 ± 0.22	15.18 ± 0.21
Oats	Straw	18.31 ± 0.17	17.04 ± 0.17	15.09 ± 0.16
Corn stalks	Stalks	17.92 ± 0.22	16.72 ± 0.21	14.80 ± 0.19
Sunflower stalks and leaves	Stalks and leaves	16.59 ± 0.31	15.47 ± 0.31	13.68 ± 0.28
Average		*18.34*	*17.07*	*15.12*
Apricots		20.81 ± 0.24	19.50 ± 0.24	17.31 ± 0.21
Sweet cherries		22.06 ± 0.19	20.71 ± 0.19	18.40 ± 0.18
Quinces		20.22 ± 0.24	18.89 ± 0.24	16.76 ± 0.22
Apples	Cuttings	20.26 ± 0.33	18.94 ± 0.33	16.80 ± 0.31
Pears		20.72 ± 0.21	19.39 ± 0.21	17.21 ± 0.20
Peaches and nectarines		21.40 ± 0.19	20.07 ± 0.19	17.82 ± 0,18
Plums		21.40 ± 0.18	20.08 ± 0.19	17.82 ± 0.18
Cherries		20.81 ± 0.19	19.46 ± 0.19	17.27 ± 0.18
Average		*20.96*	*19.63*	*17.42*
Alb de Suruceni	Cuttings	20.19 ± 0.22	18.84 ± 0.22	16.71 ± 0.21
Apiren roz		20.19 ± 0.21	18.84 ± 0.21	16.71 ± 0.20

Continuation Table 1

Cardinal		20.44 ± 0.24	19.09 ± 0.24	16.94 ± 0.22
Chismis moldovenesc		20.13 ± 0.20	18.78 ± 0.20	16.44 ± 0.19
Codreanca (Blac Magic)		20.39 ± 0.26	19.04 ± 0.26	16.89 ± 0.24
Italia		20.22 ± 0.23	18.87 ± 0.23	16.74 ± 0.22
Moldova		20.29 ± 0.24	18.94 ± 0.24	16.80 ± 0.23
Muscat de Hamburg		20.19 ± 0.22	18.84 ± 0.22	16.71 ± 0.21
Muscat timpuriu		20.21 ± 0.19	18.86 ± 0.19	16.73 ± 0.19
Prezentabil		20.30 ± 0.28	18.95 ± 0.28	16.81 ± 0.26
Average		*20.25*	*18.90*	*16.75*
Chasselas Baneasa		19.99 ± 0.14	18.64 ± 0.14	16.53 ± 0.13
Coarna alba		20.12 ± 0.13	18.77 ± 0.13	16.65 ± 0.11
Coarna neagra		20.00 ± 0.14	18.65 ± 0.14	16.54 ± 0.14
Gelu		20.00 ± 0.16	18.65 ± 0.16	16.54 ± 0.15
Greaca	Cuttings	19.99 ± 0.15	18.64 ± 0.15	16.53 ± 0.14
Otilia		19.99 ± 0.14	18.64 ± 0.14	16.53 ± 0.12
Roz Romanesc		20.10 ± 0.16	18.75 ± 0.16	16.63 ± 0.15
Select		19.99 ± 0.14	18.64 ± 0.14	16.53 ± 0.14
Victoria		20.00 ± 0.15	18.65 ± 0.15	16.54 ± 0.12
Average		*19.14*	*17.90*	*15.87*
Aligote		19.66 ± 0.14	18.31 ± 0.14	16.24 ± 0.13
Cabernet		20.00 ± 0.16	18.65 ± 0.16	16.54 ± 0.15
Chardonnay		19.87 ± 0.14	18.52 ± 0.14	16.43 ± 0.13
Feteasca alba		19.72 ± 0.21	18.38 ± 0.21	16.29 ± 0.19
Izabela		19.40 ± 0.14	18.05 ± 0.14	16.00 ± 0.13
Merlot		19.54 ± 0.13	18.19 ± 0.13	16.13 ± 0.12
Muscat Ottonel		19.67 ± 0.15	18.32 ± 0.15	16.24 ± 0.14
Pinot noir	Cuttings	19.90 ± 0.16	18.55 ± 0.16	16.45 ± 0.15
Rara neagra		19.97 ± 0.14	18.62 ± 0.14	16.51 ± 0.13
Rcatsiteli		20.01 ± 0.12	18.66 ± 0.12	16.55 ± 0.12
Saperavi		19.88 ± 0.13	18.54 ± 0.13	16.44 ± 0.12
Sauvignon		19.43 ± 0.14	18.08 ± 0.14	16.03 ± 0.13
Savinion blanc		19.53 ± 0.15	18.18 ± 0.15	16.12 ± 0.14
Traminer		19.32 ± 0.16	17.97 ± 0.16	15.93 ± 0.15
Average		*19.71*	*18.36*	*16.28*
Sea buckthorn		20.00 ± 0.16	18.73 ± 0.16	16.62 ± 0.15
Blackberry	Cuttings	18.54 ± 0.26	17.30 ± 0.26	15.32 ± 0.24
Blackcurrant		18.73 ± 0.28	17.39 ± 0.28	15.40 ± 0.26
Average		*19.09*	*17.81*	*15.78*
Miscanthus giganteus		19.87 ± 0.13	18.57 ± 0.13	16.47 ± 0.12
Silphium		19.00 ± 0.12	17.68 ± 0.12	15.66 ± 0.11
Sakhalin buckwheat	Cuttings	19.27 ± 0.28	17.96 ± 0.28	15.92 ± 0.26
Energy willow		20.99 ± 0.18	19.66 ± 0.18	17.45 ± 0.17
Energy poplar		19.40 ± 0.21	18.11 ± 0.21	16.06 ± 0.19
Reed from the Danube Delta		18.84 ± 0.26	17.62 ± 0.26	15.61 ± 0.23
Average		*19.56*	*18.27*	*16.19*

Miscanthus giganteus pellets produced from the biomass harvested in 2022 showed good burning power indices ($q_{p, net, m=10\%}$= 16.47 ± 0.12 MJ/kg). These data correlate with the results presented by us before regarding the calorific value of the Miscanthus biomass

collected in the experimental fields of the Alexandru Ciubotaru National Botanical Garden (Institute) from the Republic of Moldova in 2020 and 2021 [18].

Basically, the net calorific value of the pellets produced from Miscanthus giganteus complies with the values required for all quality classes of ENPlus certified pellets. These findings show that Miscanthus giganteus is a promising industrial crop to be used as a feedstock in the production of densified solid biofuels.

To support the recorded findings, it can be added that this crop can be cultivated on marginal agricultural areas and has an impressive productivity - of about 15 ... 20 t/ha [19, 20]. At the same time, it is known that Miscanthus inflorescences do not provide nectar or pollen, so it is recommended to provide measures in order to increase biodiversity in Miscanthus growing regions. Co-cultivation of honey plants with high energy potential would be a perfect solution for this purpose.

The studies carried out at Alexandru Ciubotaru National Botanical Garden (Institute) in the experimental sector of the Vegetal Resources Laboratory, highlighted the following plants with high melliferous potential: phacelia (*Phacelia tanacetifolia Benth., Hydrophyllaceae R. Br. family*); alfalfa, clover (*Medicago sativa L., Trifolium sp., Fabaceae Lindl. family*); sylphia, sunflower and Jerusalem artichoke (*Silphium perfoliatum L., Helianthus annuus L., H. tuberosus L., Asteraceae Bercht. & J. Presl family*), etc. [21].

5. Conclusions

Based on the research carried out in this study, it can be concluded that the pellets produced from plant biomass generated from agricultural residues and a series of energy crops have a sufficient burning power to be used for residential and industrial purposes. The biomass generated from tree residues, sunflower seed husks and vine residues, table varieties ensure the production of pellets with a calorific value equal to or greater than 16.5 MJ/kg, i.e., are in compliance with the ENPlus 3 requirements. Other types of biomass studied in this research from the point of view of their calorific value, can be used in mixtures along with other types of biomass.

Thus, the average calorific value of the pellets produced from arboreal agricultural residues is 17.42 MJ/kg, the highest calorific value being recorded by the pellets produced from cherry arboreal residues (18.4±0.18) MJ/kg. The average calorific value of the pellets produced from agricultural vine residues is 16.75 MJ/kg, the best results being recorded by the pellets produced from Cardinal vine variety residues (16.94±0.22) MJ/ kg. The calorific value of the pellets produced from sunflower husk is (16.78±0.171) MJ/kg.

Data presented in this study can be used both by producers of densified solid biofuels and specialists that deal with the development and design of technologies for the production of pellets from plant biomass.

Conflicts of Interest: The authors declare that they have no conflict of interest.

References

1. Institute for Standardization of Moldova. SM SR EN ISO 17225-2:2021. Fuel specifications and classes – Part 2. Grade wood pellets: General. Effective date: 25.06.2021.
2. Alakangas, E. Biomass and agricultural residues for energy generation. *Fuel Flexible Energy Generation* 2016, pp. 59-96. https://doi.org/10.1016/B978-1-78242-378-2.00003-1.
3. Marian, G.; Gelu, I.; Istrati, B.; Gudîma, A.; Nazar, B.; Pavlenco, A.; Banari, A.; Daraduda, N. Quality of pellets produced from agricultural wood residues specific to the Prut river basin. *Herald of Agrarian Science of the Black Sea Region МНАУ* 2021, 109 (1), pp. 84-93. https:// https:/doi: 10.31521/2313-092x/2021-1(109).
4. Portugal-Pereira, J.; Soria, R.; Rathmann, R.; Schaeffer, R.; Szklo, A. Agricultural and agro-industrial residues-to-energy: Technoeconomic and environmental assessment in Brazil. In: *Biomass and Bioenergy*. Elsevier, UK, 2015, 81, p. 521-533. https://doi.org/10.1016/j.biombioe.2015.08.010
5. Paiano, A.; Lagioia, G. Energy potential from residual biomass towards meeting the EU renewable energy and climate targets. The Italian case. *Energy Policy*, 2016, 91, pp. 161-173. https://doi.org/10.1016/j.enpol.2015.12.039.
6. Gudîma, A.; Marian, G.; Pavlenco, A. Current state of research on the influence of production variables on the quality of densified biofuels in pellet form. *Meridian Ingineresc* 2017, 1, pp. 51-60 [in Romanian].
7. Miranda, T.; Montero, I. Review of Pellets from Different Sources. *Materials* 2015, 8, pp. 1413-1427. doi:10.3390/ma8041413.
8. Albashabsheh, N.T.; Stamm, J.L. Optimization of lignocellulosic biomass-to-biofuel supply chains with densification: Literature review. *Biomass and Bioenergy* 2021, 144, pp. 1-10.
9. Institute for Standardization of Moldova. SM EN ISO 18135:2017. Solid biofuels. Sampling. Effective date: 25.09.2017.
10. Institute for Standardization of Moldova. SM EN ISO 14780:2017. Solid biofuels. Sample preparation. Effective date: 25.06.2021. Effective date: 26.09.2017.
11. Institute for Standardization of Moldova. SM EN ISO 18125:2017. Solid biofuels - Determination of calorific value. Effective date: 27.09.2017.
12. Bonassa, G.; Schneider, L.T.; Canevera, V.B.; Cremoneza, P.A.; Frigob, E.P.; Dieterb, J.; Telekenb, J.G. Scenarios and prospects of solid biofuel use in Brazil. *Renewable and Sustainable Energy Reviews* 2018, 82(3), pp. 2365-2378. https://doi.org/10.1016/j.rser.2017.08.075.
13. Scarlat, N.; Blujdea, V.; Dallemand, J.F. Assessment of the availability of agricultural and forest residues for bioenergy production in Romania. *Biomass and Bioenergy* 2011, 35 (5), pp. 1995-2005. https://doi.org/10.1016/j.biombioe.2011.01.057.
14. Hăbăşescu, I.; Cerempei, V. Energetic potential of vegetable mass in agriculture of the Republic of Moldova. In: Energy of Moldova. Regional aspects of development, Proceedings of the international conference "Energy of Moldova - 2012", Chisinau, Republic of Moldova, Edition II, Energy Institute of the Academy of Sciences of Moldova, 4-6 October 2012, p. 355-357. [in Romanian].
15. Gudîma, A. Evaluation of the use of agricultural residues for energy purposes. Case study for Soroca district, Republic of Moldova. *Meridian Ingineresc* 2017, 1 (64), pp. 26-29 [in Romanian].
16. Pavlenco, A. The quality of solid biofuels produced from agricultural residues. Article synthesis. *Agricultural Science* 2018, 2, pp. 128-140 [in Romanian].
17. Țenu, I.; Roman, C.; Senila, L.; Roşca, R.; Cârlescu, P.; Băetu, M.; Arsenoaia, V.; Dumitrachi, E.P.; Corduneanu, O.R. Valorization of Vine Tendrils Resulted from Pruning as Densified Solid Biomass Fuel (Briquettes). *Processes* 2021, 9 (8), 1409.
18. Daraduda, N.; Marian, G. Perspectives for the use of biomass generated by some Miscanthus genotypes in the production of densified solid biofuels. *Journal of Engineering Science* 2022, 2 (29), pp. 133-143.
19. Bajura, T.; Gandacova, S. New bioenergy sources for national economy: miscanthus. *Annals of the National Institute of Economic Research* 2020, 10, pp. 16 - 20 [in Romanian].
20. Tilvikiene, V.; Kadziuliene, Z.; Liaudanskienea, I.; Zviceviciusb, E.; Cerniauskieneb, Z.; Algirdas, A.C.; Railab, J.; Baltrusaitisc, J. The quality and energy potential of introduced energy crops in northern part of temperate climate zone. *Renewable Energy* 2020, 151, pp. 887-895. https://doi.org/10.1016/j.renene.2019.11.080.
21. Cîrlig N.; Lupan A.; Ţiţei, V.; Guţu, A.; Iurcu-Străistaru, E.; Initiation of the foundation of the honey plant collection within the National Botanical Garden (Institute) „A. Cibotaru". *Jurnal of Botany* 2020, 2 (21), pp. 169-170. DOI:10.52240/1857-2367.2020.2(21).41

22. Marian, Gr. Solid biofuels, production and properties. Bons Offices, Chisinau, 2016. 172 p.
23. Niedziółka I.; Szpryngiel M.; Kachel-Jakubowskaa M.; Kraszkiewicza A.; Zawiślakb K.; Sobczakb P.; Nadulskib R. Assessment of the energetic and mechanical properties of pellets produced from agricultural biomass. *Renewable Energy* 2015, 76, pp. 312 - 317. https://doi.org/10.1016/j.renene.2014.11.040
24. Ciolacu, F.; Ianuș, G.; Marian, G.; Munteanu, C.; Paleu, V.; Nazar, B.; Istrate, B.; Gudîma, A.; Daraduda, N. A Qualitative Assessment of the Specific Woody Biomass of Fruit Trees. *Forests* 2022, 13(3), 405. https://doi.org/10.3390/f13030405.
25. Marian, G.; Ianuș, G.; Istrate, B.; Banari, A.; Nazar, B.; Munteanu, C.; Măluţan, T.; Gudîma, A.; Ciolacu,F.; Daraduda, N.; Paleu, V.. Evaluation of Agricultural Residues as Organic Green Energy Source Based on Seabuckthorn, Blackberry, and Straw Blends. *Agronomy* 2022, 12(9). https://doi.org/10.3390/agronomy12092018.

Publisher's Note: JES stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.

Submission of manuscripts: jes@meridian.utm.md

OPEN ACCESS

MICROENCAPSULATION OF ANTHOCYANINS FROM CORNELIAN CHERRY FRUITS IN WHEY PROTEIN ISOLATE AND PECTIN

Iuliana-Maria Enache [1], ORCID: 0000-0002-4691-7311,
Liliana Lucescu Ciurlă [1], ORCID: 0000-0003-2874-9825,
Nicoleta Stănciuc [2], ORCID: 0000-0002-4763-8656,
Liviu Mihai Irimia [1], ORCID: 0000-0003-1356-1774,
Antoanela Patraș [1*], ORCID: 0000-0002-4054-4884,
Camelia Vizireanu [2], ORCID: 0000-0002-8704-2662

[1]*"Ion Ionescu de la Brad" Iasi University of Life Sciences", Department of Sciences, Iasi, Romania*
[2]*"Dunărea de Jos" University of Galați, Department of Food Science, Food Engineering, Applied Biotechnology, Galati, Romania*
*Corresponding author: Antoanela Patraș, apatras@uaiasi.ro

Abstract. Cornelian cherry (*Cornus mas* L.) is one of the most important forest fruits, considered as a valuable horticultural resource of bioactives, such as anthocyanins - cyanidin-3-glucoside, flavonoids, vitamins (e.g. vitamin C), carotenoids (e.g. β-carotene). The aim of this study was to obtain designed delivery systems of bioactive from cornelian cherry, as microencapsulated powders in order to assure their controlled release and to develop stable and natural additives for different application. Anthocyanin's (concentrated extract) from cornelian cherry fruits were microencapsulated in a complex, biopolymeric matrice, formed by whey protein isolate (WPI) and pectin (PT). Two experimental variants were obtained by varying the ratio between WPI and PT, such as 1:1 (PT1) and 1:2 (PT2). The powders were tested for encapsulation efficiency of the anthocyanins, phytochemical profile of the extract and freeze-dried powders, as well as colorimetric analysis. Encapsulation efficiency of the anthocyanins varied between 80.04 and 82.11% with an important level of biologically active compounds (total polyphenols, total flavonoids) and remarkable antioxidant activity. Colorimetric analysis reveals a red colour of the powders, associated with their anthocyanin content. Both experimental variants proposed in this study protected the anthocyanins from cornelian cherry fruits. Moreover, microencapsulated powders can be used as natural food additives due to their red colour and phytochemical profile.

Keywords: *complex biopolymeric matrice, Cornus mas L., encapsulation efficiency, colour.*

Rezumat. Cornul de pădure (*Cornus mas* L.) este unul dintre cele mai importante fructe de pădure, considerat o resursă horticolă valoroasă de compuşi bioactivi, cum ar fi antociani - cianidin-3-glucozidă, flavonoide, vitamine (de exemplu, vitamina C), carotenoide (de exemplu, β-caroten). Scopul acestui studiu a fost de a obţine sisteme de eliberare a substanţelor bioactive din fructele de corn, sub formă de pulberi microîncapsulate, pentru a

asigura eliberarea lor controlată și pentru a dezvolta aditivi naturali și stabili pentru diferite aplicații. Antocianii (extract concentrat) din fructele de corn au fost microîncapsulați într-o matrice biopolimerică complexă, formată din izolat de proteic din zer (IPZ) și pectină (PT). Două variante experimentale au fost obținute prin variația raportului dintre IPZ și PT, cum ar fi 1:1 (PT1) și 1:2 (PT2). Au fost testate pudrele pentru eficiența de încapsulare a antocianilor, profilul fitochimic al extractului și al pulberilor liofilizate, precum și din punct de vedere colorimetric. Eficiența de încapsulare a antocianilor a variat între 80.04 și 82.11%, cu un nivel important de compuși biologic activi (polifenoli totali, flavonoide totale) și o activitate antioxidantă remarcabilă. Analiza colorimetrică relevă o culoare roșie a pudrelor, asociată cu conținutul de antociani. Ambele variante experimentale propuse în acest studiu au protejat antocianii din fructele de corn. În plus, pulberile microîncapsulate pot fi utilizate ca aditivi alimentari naturali datorită culorii roșii și profilului lor fitochimic.

Cuvinte cheie: *matrice biopolimerică complexă, Cornus mas L., eficiența încapsulării, culoare.*

1. Introduction

Cornelian cherry (*Cornus mas* L) is one of the forest fruits found in 38 spontaneous crop in Romania, considered as a rich resource of biologically active compounds (polyphenols: anthocyanins, flavonoids, vitamins and carotenoids etc) [1]. According to a study conducted by Yilmaz, (2009) [2], cornelian cherry, known also as *dogwood*, only 4 of the 65 species are edible.

Anthocyanins represent an important subgroup of flavonoids, perhaps the most important of them. Over time studies have revealed a number of essential characteristics of flavonoids, of which the best known can be considered: antioxidant, antimicrobial, antibacterial, antifungal and antiviral activity [3]. A study conducted by Manganaris [4] concluded that in the *Plantae* kingdom the most popular compounds with antioxidant properties are: ascorbic acid, carotenoids, tocopherol and phenolic compounds.

To the best of our knowledge, one of the most important categories of biologically active compounds found naturally in fresh fruits and vegetables are anthocyanins. The 700 anthocyanins identified since now are associated with some specific colour of leaves, flowers and fruits, such as orange, red, blue and purple colours and the shades between them. The compounds above-mentioned are derived from aglycons, being known as *anthocyanidins*. Each of six more commonly anthocyanidins found in nature are correlated with a maximum absorbance, as well as with a specific colour, respectivelly: pelargonidin (494 nm, identified by orange colour), cyanidin and peonidin (506 nm, correlated with orange-red colour), delphinidin and petunidin (red colour, 508 nm) and malvinidin (510 nm, bluish-red colour components) [5, 6].

A remarkable property of anthocyanins is their antioxidant capacity. A study conducted by Einbond (2004) [7] suggests that the most common anthocyanin is thought to be cyanidin. According to the same study, cyanidin-3-glucoside is considered to be the most active anthocyanin associated with a high level of antioxidants. Therefore, based on the above-mentioned information, cyanidin-3-glucoside is a natural compound considered one of the most popular anthocyanins correlated with an important antioxidant capacity.

Moreover, the literature highlights [8] that anthocyanin extraction is strongly influenced by the solvent or solvents used, including the ratio between them, table 1 shown the most commonly of them.

Table 1

The most commonly solvents used for anthocyanins extraction

Anthocyanins	Method of extraction/ Solvent(s)
Cyanidin-3-glucoside	Maceration/Methanol [7]
Delphinidin-3-glucoside	Maceration/Methanol [7]
Cyanidin-3-glucoside	Conventional extraction/Ethanol 60-100% [1]
Cyanidin-3-rutinoside	Combined method (hydroalcoholic solvent and ultrasound extraction)/ Ethanol 70% [9]

On the another hand, the anthocyanins extraction yield can be influenced by several factors. The influencing factors of the anthocyanins extraction yield and recommended variant can be considered: type of solvent (e.g. organic/inorganic solvent), quantity of solvent (expressed as µL/mL), type of extraction (e.g. maceration/ conventional extraction/ ultrasound extraction), temperature extraction (e.g. 30 – 50 $^{\circ}$C), duration of the extraction (e.g. 15-45 min) [1, 9].

In order to obtain a functional extract of phenolic compounds from black rice, as well as for anthocyanins extraction, previous studies used random parameters (extraction time and temperature, concentration of solvent), as well as a mathematical method (Response Surface Methodology) for the optimization phenolic compounds extraction yield [10 -15]. The most widely used method for determining anthocyanins is the differential pH method [16].

As for the health benefits of anthocyanins, the most common examples can be considered the relief or treatment of conditions such as cancer, allergies, viral infections, low immunity, inflammation, diabetes [17].

In terms of determination and quantification methods, antioxidant activity can be achieved after microwave-assisted extraction, ultrasound-assisted extraction, maceration or conventional extraction using various methods, shown in table 2:

Table 2

Analytical methods used for the quantification of antioxidant activity [17, 18].

Method	Reagent/ Method explication
DPPH	2,2-diphenyl-1-picrylhydrazyl
ORAC	Oxygen Radical Absorbance Capacity
TEAC	Trolox Equivalent Antioxidant Capacity
FRAP	Ferric Radical Absorbance Capacity
CUPRAC	Cupric Radical Absorbance Capacity
ABTS	2,2-azino-bis-3-ethylbenzothiazoline-6-sulfonic acid

It can be considered that anthocyanins are the most common biologically active compounds in nature, but they degrade very quickly.

In order to protect anthocyanins against external factors (pH, light, storage temperature, storage time, etc.) it is necessary to find viable, efficient and cost-effective alternatives. The objectives listed above can be achieved by applying microencapsulation, and a number of technologies can be used, including spray drying, ionic gelation, freeze drying, etc. [17-19].

Freeze-drying involves removing water from the sample by freezing. This technique consists of two stages: (1) sublimation in the primary drying stage *and* (2) desorption in the secondary drying stage. Among the advantages of using freeze-drying are: applicability in

various fields (e.g. pharmaceuticals, biological products, flowers, cultured micro-organisms, bulk pharmaceuticals, medical and cosmetic devices, chemicals, pigments, enzymes etc.), thermolabile products (e.g. heat-sensitive foods, products unstable in water, high storage stability over time. The extraction of water from food by freeze-drying involves the following steps: freezing the food → sublimation of the ice formed directly into water vapor → removal of the water vapor. Freeze-drying is considered to be complete when the sublimation process is completed. The quality of the final product obtained by freeze-drying is dependent on three essential parameters that must be set very correctly: the pressure of the equipment (e.g., 10 Pa), the working time (e.g., 48 hours) and the operating temperature of the equipment (e.g., -42 °C) [9, 20, 21]. An important role in the freeze-drying process is played by the excipients used (bulking agents, antimicrobial agents, surfactants, cosolvents) [22].

Several studies showed the importance of the practical applications of microencapsulation in the food industry and biotechnology. In terms of specific applications for the food industry, a wide range of lactic acid bacteria, biologically active compounds extracted from fruits and vegetables, can be subjected to the microencapsulation process, including *Bifidobacterium lactis* [23], pineapple, acerola cherries, guava, papaya, mango [24], pomegranate [25], cherries [26], bananas [27], black rice [28], grape peel [29], strawberry [30], tomato peel [31], eggplant peel [32], yellow onion peel [33], black beans [34], blackcurrants [35], cornelian cherry fruit [9, 36].

Nowadays, a negative impact on human health is given by the consumption of food synthetic additives, associated with many diseases, such as: obesity, cancer, diabetes, hypertension, dementia or eye diseases [37].

Wall materials (including those used in this work) can be correlated with many health benefits. Whey protein isolate is associated with antimicrobial and antiviral effect, anticancer effect, cardiovascular health, immunological activity, improves cognitive performance, physical performance, and weight management [38]. The pectin's effects for human health include maintenance of low level of cholesterol, prevention of colon and prostate cancer; remove heavy metals from body [39].

The main aim of this study was to obtain and characterise two complex biopolymeric matrices of cornelian cherry extract (performed with whey protein isolate and pectin) by the variation of their ratio (1:1, 1:2). The final goal was to demonstrate their benefits for future applications as natural food additives due to their important level of anthocyanins, polyphenols and flavonoids content, as well as antioxidant capacity.

2. Materials and Methods

Reagents used in this study (extraction: ethanol; total antioxidant activity: methanol, 2,2-Diphenyl-1-picrylhydrazyl (DDPH), 6-Hydroxy-2.5.7.8-tetramethylchroman-2-carboxylic acid (Trolox); total polyphenols content: Folin–Ciocalteu reagent, sodium carbonate, gallic acid; total flavonoids content: sodium nitrate, aluminium chloride, sodium hydroxide, catechin; total anthocyanins content: cyanidin-3-glucoside, sodium acetate, potassium chloride; encapsulation efficiency: acetic acid) were purchased from Sigma-Aldrich (Steinheim, Germany).

The cornelian cherry fruits (CCF) were purchased from a local market (Galati, Romania) in 2018. Fruit washed with distilled water and dried with paper towels had the stones removed.

CCF extract was obtained by hydroalcoholic extraction (70% ethanol) combined with ultrasound extraction (30 min, 40°C, 100 W and 40 kHz - MRC Scientific Instrument), followed

by filtration. Finally, the liquid extract was centrifuged at 5000 rpm for 30 min at 4°C (Universal 320R, Tuttlingen, Germany). Microencapsulation of anthocyanins from CCF was achieved by mixing the concentrated extract (2.5 g) with wall materials: whey protein isolate (WPI) and pectin (PT). Encapsulation of the bioactive compounds from *Cornus mas* fruits was achieved by mixing the concentrated extracts fruits with whey protein isolate and pectin as follows: variant 1: 2.5 g concentrate extract of cornelian cherry fruits, 1 g whey protein isolate, 1 g pectin were mixed and brought to the mark with ultrapure water using a 100 mL volumetric flask. For the development of the second experimental variant, the same protocol was used, except that 2 g of pectin was added. Two microencapsulated powders were obtained, respectively: WPI:PT=1:1 (PT1) and WPI:PT=1:2 (PT2). Both experimental variants were left for 4 hours on a magnetic stirrer (650 rpm) in order to completely homogenize the ingredients of the mixture. Subsequently, the samples were kept for 24 hours at -4 °C in order to hydrate the mixture, and then freeze-dried (-42°C, for 48 hours at a pressure of 10 Pa - CHRIST Alpha 1-4 LD plus, Osterode am Harz, Germany).

Phytochemical profile and antioxidant capacity of the extract and microencapsulated powders was performed by: total anthocyanin content (TAC) – pH differential method (results expressed as mg cyanidine-3-glucoside per gram of dry weight (C3G/g DW), total polyphenols content (TPC) – Folin-Ciocalteu method (results expressed as mg gallic acid equivalent (GAE)/g DW) total flavonoid content (TFC) – colorimetric method (results expressed as mg catechin equivalent (CE)/g DW), while total antioxidant activity (T-AA) was studied by DDPH radical scavenging method (mMol Trolox Equivalent (TE)/g DW) [9, 35, 36]. The spectrophotometer UV-Vis Jenway was used to read to absorbance for each sample (OSA, UK).

Encapsulation efficiency is the percentage of encapsulated anthocyanins in the wall materials used, by measuring the surface anthocyanin content (SAC), by extracting the anthocyanins in a mixture of methanol and ethanol in a ratio of 1:1, (v/v) and the total anthocyanin content (TAC), by extracting in a mixture of methanol: acetic acid: ultrapure water (25:4:21, v/v/v), according to equation 1:

$$EE(\%) = \frac{TAC-SAC}{TAC} x\ 100 \qquad (1)$$

The colorimetric analysis was performed by determining the colour parameters (L*, a*, b*) using the colorimeter. According to the CIEL*a*b* system protocol, the L* parameter indicates the brightness of the sample (0 - for black, 100 - for white), the a* parameter provides information about the green/red spectrum of the sample (positive values are associated with red shades, while negative values are associated with green shades), and the b* blue/yellow parameter indicates the tendency of the analysed sample to have yellow (positive values) or blue (negative values) colour [40].

$$C* = \sqrt{(a*^2 + b*^2)} \qquad (2)$$

$$h* = arctang\left(\frac{b*}{a*}\right) \qquad (3)$$

$$\Delta E* = \sqrt{(\Delta L*)^2 + (\Delta a*)^2 + (\Delta b*)^2} \qquad (4)$$

The overall colour difference or total colour difference (ΔE*) was obtained as follows: for the experimental variants of microencapsulated powders, the difference between the initial L*, a*, b* values obtained for the concentrated extract of cornelian cherry fruit and the obtained powders (PT1, PT2) was taken into account, while the ΔE* for the concentrated

extract was calculated as the difference between the L*, a*, and respectively, b* values of the cornelian cherry fruit and the values of the concentrated extract.

All the analysis of this study were performed in triplicate and the results were expressed as mean ± standard deviation. Microsoft Excel Package Tools and IBM SPSS Statistics 21 were used to perform statistical analysis. Tukey test with 95% confidence interval was applied.

3. Results and Discussion

The anthocyanins content, polyphenolic content and flavonoids content, as well as the antioxidant capacity of the cornelian cherry extract and microencapsulated powder are showed in table 3.

Table 3

Bioactive compounds, antioxidant activity and encapsulation efficiency of concentrated extract and microencapsulated powders

Tested parameter	Value
Concentrated extract	
Total antioxidant activity (mMol TE/g DW)	185.15 ± 1.15
Total polyphenolic content (mg GAE/g DW)	17.41 ± 0.67
Total anthocyanins content (mg C3G/g DW)	12.86 ± 0.80
Total flavonoid content (mg CE/g DW)	5.08 ± 0.13
Microencapsulated powders	
PT1	
Encapsulation efficiency (%)	80.04 ± 0.81[a]
Total antioxidant activity (mMol TE/g DW)	53.54 ± 0.27[a]
Total polyphenolic content (mg GAE/g DW)	10.45 ± 0.06[a]
Total anthocyanins content (mg C3G/g DW)	5.15 ± 0.12[a]
Total flavonoid content (mg CE/g DW)	3.76 ± 0.13[a]
PT2	
Encapsulation efficiency (%)	82.11 ± 0.14[b]
Total antioxidant activity (mMol TE/g DW)	53.59 ± 0.11[a]
Total polyphenolic content (mg GAE/g DW)	10.79 ± 0.67[a]
Total anthocyanins content (mg C3G/g DW)	5.71 ± 0.06[b]
Total flavonoid content (mg CE/g DW)	3.69 ± 0.18[a]

Note. For each powders parameter tested, values that are for the same parameter (e.g. total antioxidant activity) that do not have the same lowercase letters ((a) or (b)) are statistically different at $p < 0.05$ based on the Tukey method with 95% confidence interval

In table 3 can be observed that the characteristics of the concentrated extract were well preserved in the two microencapsulated powders with the exception of the total antioxidant activity, which decreased with 71%. Generally, the results of the PT2 variant were slightly better compared to PT1 (excepting the total flavonoid content), but the differences

are extremely small. The encapsulation efficiency of the anthocyanins was higher than 80%, highlighting the efficiency of the biopolymeric combination used in this study to retain the bioactive compounds. The obtained results are in accordance with previous researches [36], which reported an encapsulation efficiency of 95.46±1.30 % for anthocyanins extracted from black currant fruits microencapsulated in WPI, chitosan and inulin. In another study [9], were microencapsulated cornelian cherry fruits extracts in WPI and casein or inulin. In this case, encapsulation efficiency varied between 77.97±0.57 % (variant with casein) and 79.03 ± 0.72% (variant with inulin).

Table 4

Comparative analysis of the cornelian cherry parameters previously published

Determined parameter	Solvent used	Method of determination (λ, nm)	Value	Unit of measurement	References
Antioxidant activity	Ethanol 80%	DPPH (515 nm)	0.43-1.32	$(1/IC_{50})\cdot100$	[41]
	Methanol	DPPH (515 nm)	38.98-82.37	%	[42]
Anthocyanins content	Ethanol 80%	pH differential method (510 nm, 700 nm)	0.06-3.03	mg C3G/ g	[41]
	Methanol; Hydrochloric acid (1%, v/v)	pH differential method (510 nm, 700 nm)	106.89-442.11	mg C3G/ 100 g	[42]
Flavonoids content	Methanol; Hydrochloric acid (1%, v/v)	Colorimetric method (NaOH) (510 nm)	321.71-669.00	mg CE/100 g fresh fruit	[42]
	Methanol Water Ethyl acetate Acetone Petroleum ether	Colorimetric method–AlCl$_3$ (415 nm)	7.18 ± 0.10 3.53 ± 0.39 41.49 ± 0.57 8.05 ± 0.76 6.91 ± 0.09	mg RE /g extract	[43]
Polyphenols content	Hydrochloric acid: methanol: water (2:80:18)	Folin Ciocâlteu method (765 nm)	2.61±0.21 8.11±0.40	g GAE/ kg fresh fruit	[44]
	Methanol Water Ethyl acetate Acetone Petroleum ether	Folin Ciocâlteu method (765 nm)	31.36±0.34 12.77±0.81 179.05±0.53 55.38±0.86 27.14±0.30	mg GAE/g extract	[43]

Comparing data with the literature is difficult due to the different extraction techniques used and the results expression, but the results of present study are close to the previously published values, if converted to similar units.

Another concern of the food industry is the colour of food. One of the major objectives of this study was to demonstrate that the phytochemical composition of our samples change in a similar manner to the change of their red colour (a* parameter), and the obtained results prove it.

It is obvious that the phenolic compounds, and especially the anthocyanin pigments due to their red colour in acidic media, influence the chromatic parameter a*. Table 5 presents the chromatic parameters of the two microencapsulated powders, compared to the concentrated extract. The values obtained for a* parameter prove that all the analysed samples are red coloured, as the value of a* is positive (table 5). The red colour is able to create a positive psychological impact on the consumer, who is very attracted to brightly coloured foods [45].

Table 5

Chromatic analysis of the concentrated extract and microencapsulated powders

Chromatic parameter tested	Value
Concentrated extract	
Luminosity (L*)	53.72±0.83
Green/red colour component (a*)	20.22±0.32
Blue/yellow colour component (b*)	7.04±0.03
Tone (hue angle, h*)	21.41±0.15
Chroma (colour intensity, C*)	0.33±0.01
Total colour difference (ΔE*)	57.82±0.63
Microencapsulated powders	
PT1	
Luminosity (L*)	36.79±0.59[a]
Green/red colour component (a*)	10.95±0.74[a]
Blue/yellow colour component (b*)	5.85±0.45[a]
Tone (hue angle, h*)	12.42±0.79[a]
Chroma (colour intensity, C*)	0.49±0.03[a]
Total colour difference (ΔE*)	38.83±0.79[a]
PT2	
Luminosity (L*)	35.04±0.68[b]
Green/red colour component (a*)	11.65±0.42[b]
Blue/yellow colour component (b*)	5.52±0.14[a]
Tone (hue angle, h*)	12.89±0.43[a]
Chroma (colour intensity, C*)	0.44±0.01[a]
Total colour difference (ΔE*)	37.34±0.51[b]

Note. For each powders parameter tested, values that are for the same parameter (e.g. luminosity) that do not have the same lowercase letters ((a) and (b)) are statistically different at $p < 0.05$ based on the Tukey method and the 95% confidence interval.

The colour parameters (table 5), as well as the previously measured parameters (table 3), were well preserved in the microencapsulated powders, with very closed values between the 2 variants (PT1 and PT2). The total colour difference of powders (compared to the concentrated extract) proved that PT2 was closer to the initial extract, compared to PT1

(smaller ΔE* value) and this confirms, also from the chromatic point of view, that PT2 is a better encapsulating solution than PT1 (but the difference is small). Double amount of pectin than the amount of whey protein isolate has been shown to slightly improve the properties of the microencapsulated powder, but an economic analysis would be required to verify whether this small improvement is worth the cost difference.

4. Conclusions

In order to protect the anthocyanins from cornelian cherry fruits against environmental factors (light, storage, temperature etc.), two walls materials were used (whey protein isolate and pectin). The scientific novelty of present research comes from the development, integration and implementation of two variants of microencapsulations with complex biopolymeric matrices that have not been previously applied for the protection of the bioactive compounds from cornelian cherry fruits, according to our knowledge. The biopolymeric wall materials used in this study for the microencapsulation of the cornelian cherry fruit anthocyanins allowed obtaining two powders with high encapsulation efficiency and satisfactory phytochemical profile. Colorimetric analysis highlighted a light red colour with yellow shades in both experimental variants. Further studies are needed to test the *in vitro* digestion of these anthocyanins and also, to test cornelian cherry fruits as source of natural food additives, researches which are currently on-going in our laboratories.

Conflicts of Interest: The authors declare no conflict of interest

References

1. Enache, I.M.; Coman, G.; Rosca, S.; Vizireanu, C.; Mihalcea, L. Optimization conventional extraction of bioactive compounds from *Cornus mas* by RSM and determination of favorability factors by GIS technique. *Not. Bot. HortiAgrobot. Cluj-Napoca.* 2021, 49 (2), 12307.DOI: https://doi.org/10.15835/nbha49212307 (a)
2. Yilmaz, K.U.; Ercisli, S.; Zengin, Y.; Sengul, M.; Kafkas, E.Y. Preliminary characterisation of cornelian cherry *(Cornus mas* L.) genotypes for their physico-chemical properties. *Food Chem.* 2009, 114 (2), pp. 408–412.
3. Tapas, A.R.; Sakarkar, D.M.; Kakde, R.B. Flavonoids as nutraceuticals: a review. *Trop. J. Pharm. Res.* 2008, 7 (3), pp. 1089–1099. https://doi.org/10.4314/tjpr.v7i3.14693.
4. Manganaris, G.A.; Goulas, V.; Vicente, A.R.; Terry, L.A. Berry antioxidants: Small fruits providing large benefits. *J. Sci. Food Agric.* 2013, 94 (5), pp. 825–833. https://doi.org/10.1002/jsfa.6432
5. de Pascual-Teresa, S.; Sanchez-Ballesta, M.T. Anthocyanins: from plant to health. *Phytochem. Rev* 2008, 7, pp. 281–299. https://doi.org/10.1007/s11101-007-9074-0
6. Wallace, T.C.; Giusti, M.M. Anthocyanins. *Adv. Nutr.* 2015, 6 (5), pp. 620–622. doi:10.3945/an.115.009233
7. Einbond, L.S.; Reynertson, K.A.; Luo, X.D.; Basile, M.J.; Kennelly, E.J. Anthocyanin antioxidants from edible fruits. Food Chem. 2014, 84 (1), pp. 23–28. https://doi.org/10.1016/S0308-8146(03)00162-6
8. Escribano-Bailón, M.T.; Santos-Buelga, C.; Rivas-Gonzalo, J.C. Anthocyanins in cereals. *J. Chromatogr. A.* 2004, 1054 (1–2), pp. 129–141. https://doi.org/10.1016/j.chroma.2004.08.152
9. Enache, I.M.; Vasile, M.A.; Crăciunescu, O.; Prelipcean, A.M.; Oancea, A.; Enachi, E.; Barbu, V.V.; Stănciuc, N.; Vizireanu C. Co-microencapsulation of anthocyanins from cornelian cherry (*Cornus mas* l.) fruits and lactic acid bacteria into antioxidant and anti-proliferative derivative powders. *Nutrients* 2022. 14, 3458. https://doi.org/10.3390/nu14173458

10. Phetpornpaisan, P.; Tippayawat, P.; Jay, M., Sutthanut, K. A local Thai cultivar glutinous black rice bran: A source of functional compounds in immunomodulation, cell viability and collagen synthesis, and matrix metalloproteinase-2 and -9 inhibition. *J. Funct. Foods.* 2014, 7, pp. 650–661. https://doi.org/10.1016/j.jff.2013.12.020

11. Shao, Y.; Xu, F.; Sun, X.; Bao, J.; Beta, T. Identification and quantification of phenolic acids and anthocyanins as antioxidants in bran, embryo and endosperm of white, red and black rice kernels (*Oryza sativa* L.). *J. Cereal Sci.* 2014, 59 (2), pp. 211–218. https://doi.org/10.1016/j.jcs.2014.01.004

12. Bassani, D.C.; Nunes, D.S.; Granato, D. Optimization of phenolics and flavonoids extraction conditions and antioxidant activity of roasted yerba-mate leaves (*Ilex paraguariensis* A. St.-Hil., Aquifoliaceae) using response surface methodology. *An. Acad. Bras. Cienc.* 2014, 86 (2), pp. 923–934. https://doi.org/10.1590/0001-3765201420130019

13. Granato, D.; de Araújo Calado; V.Ô. M.; Jarvis, B. Observations on the use of statistical methods in Food Science and Technology. *Food Res. Int.* 2014, 55, pp. 137–149.

14. Granato, D.; Grevink, R.; Zielinski, A.A.F.; Nunes, D.S.; Van Ruth, S. M. Analytical strategy coupled with response surface methodology to maximize the extraction of antioxidants from ternary mixtures of green, yellow, and red teas (*Camellia sinensis* var. Sinensis). *J. Agric. Food Chem.* 2014, 62 (42), pp. 10283–10296.

15. Pedro, A.C.; Granato, D.; Rosso, N.D. Extraction of anthocyanins and polyphenols from black rice (*Oryza sativa* L.) by modeling and assessing their reversibility and stability. *Food Chem.* 2016, 191, pp. 12–20. https://doi.org/10.1016/j.foodchem.2015.02.045

16. Turturică, M.; Stănciuc, N.; Bahrim, G.; Râpeanu, G. Effect of thermal treatment on phenolic compounds from plum (*Prunus domestica*) extracts – A Kinetic study. *J. Food Eng.* 2016, 171, pp. 200–207. https://doi.org/10.1016/j.jfoodeng.2015.10.024.

17. Veberic, R.; Jakopic, J.; Stampar, F.; Schmitzer, V. European elderberry (*Sambucus nigra* L.) rich in sugars, organic acids, anthocyanins and selected polyphenols. *Food Chem.* 2009, 114 (2), pp. 511–515. https://doi.org/10.1016/j.foodchem.2008.09.080

18. Altemimi, A.; Lakhssassi, N.; Baharlouei, A.; Watson, D.G.; Lightfoot, D.A. Phytochemicals: Extraction, isolation, and identification of bioactive compounds from plant extracts. *Plants* 2017, 6 (4), 23. https://doi.org/10.3390/plants6040042

19. Dinda, B.; Kyriakopoulos, A.M.; Dinda, S.; Zoumpourlis, V.; Thomaidis, N.S.; Velegraki, A.; Markopoulos, C.; Dinda, M. *Cornus mas* L. (cornelian cherry), an important European and Asian traditional food and medicine: Ethnomedicine, phytochemistry and pharmacology for its commercial utilization in drug industry. *J. Ethnopharmacol.* 2016, 193, pp. 670–690. https://doi.org/10.1016/j.jep.2016.09.042

20. Nireesha, Gr. L.; Divya, C.; Sowmya, N.; Venkateshan, M.; Niranjan Babu, V.L. Lyophilization/freeze drying - an review. *Int. J. Novel Trends Pharm. Sci.* 2013, 3 (4), pp. 87-98.

21. Ciurzyńska, A.; Lenart, A. Freeze-drying - application in food processing and biotechnology - a review. *Polish J. Food Nutr. Sci.* 2011, 61 (3), pp. 165–171, https://doi.org/10.2478/v10222-011-0017-5

22. Baheti, A.; Kumar, L.; Bansal, A.K. Excipients used in lyophilization of small molecules. *J. Excip. Food Chem.* 2010, 1 (1), pp. 41–54.

23. Saarela, M.; Virkajärvi, I.; Alakomi, H.L.; Sigvart-Mattila, P.; Mättö, J. Stability and functionality of freeze-dried probiotic *Bifidobacterium* cells during storage in juice and milk. *Int. Dairy J.* 2006, 16 (12), pp. 1477–1482. https://doi.org/10.1016/j.idairyj.2005.12.007

24. Marques, L.G.; Silveira, A.M.; Freire, J.T. Freeze-drying characteristics of tropical fruits. *Dry. Technol.* 2007, 24 (4), pp. 457–463. https://doi.org/10.1080/07373930600611919

25. de Ciriano, M.G.I.; Rehecho, S.; Calvo, M.I.; Cavero, R.Y.; Navarro, Í.; Astiasarán, I.; Ansorena, D. Effect of lyophilized water extracts of *Melissa officinalis* on the stability of algae and linseed oil-in-water emulsion to be used as a functional ingredient in meat products. *Meat Sci.* 2010, 85 (2), pp. 373–377. https://doi.org/10.1016/j.meatsci.2010.01.007

26. Zorić, Z.; Dragović-Uzelac, V.; Pedisić, S.; Kurtanjek, Ž.; Garofulić, I.E. Kinetics of the degradation of anthocyanins, phenolic acids and flavonols during heat treatments of freeze-dried sour cherry *Marasca paste*. *Food Technol. Biotechnol.* 2014, 52 (1), pp. 101–108.

27. Jiang, H.; Zhang, M.; Mujumdar, A.S.; Lim, R.X. Drying uniformity analysis of pulse-spouted microwave–freeze drying of banana cubes. *Dry. Technol.* 2015, 34 (5), pp. 539–546. https://doi.org/10.1080/07373937.2015.1061000

28. Bulatao, R.M.; Samin, J.P.A.; Salazar, J.R.; Monserate, J.J. Encapsulation of anthocyanins from black rice (*Oryza sativa* L.) bran extract using chitosan-alginate nanoparticles. *J. Food Res.* 2017, 6 (3), pp. 40–47. https://doi.org/10.5539/jfr.v6n3p40

29. Stănciuc, N.; Turturică, M.; Oancea, A.M.; Barbu, V.; Ioniţă, E.; Aprodu, I.; Râpeanu, G. Microencapsulation of anthocyanins from grape skins by whey protein isolates and different polymers. *Food Bioproc. Tech.* 2017, 10 (9), pp. 1715–1726. https://doi.org/10.1007/s11947-017-1938-8

30. Ursache, F.M.; Andronoiu, D.G.; Ghinea, I.O.; Barbu, V.; Ioniţă, E.; Cotârleţ, M.; Dumitraşcu, L.; Botez, E.; Râpeanu, G.; Stănciuc, N. Valorizations of carotenoids from sea buckthorn extract by microencapsulation and formulation of value-added food products. *J. Food Eng.* 2018, 219, pp. 16–24, https://doi.org/10.1016/j.jfoodeng.2017.09.015

31. Gheonea (Dima), I.; Aprodu, I.; Cîrciumaru, A.; Râpeanu, G.; Bahrim, G.E.; Stănciuc, N. Microencapsulation of lycopene from tomatoes peels by complex coacervation and freeze-drying: Evidences on phytochemical profile, stability and food applications, *J. Food Eng.* 2021, 288, 110166, pp. 1-8, https://doi.org/10.1016/j.jfoodeng.2020.110166

32. Condurache, N.N., Aprodu, I., Crăciunescu, O.; Tatia, R.; Horincar, G.; Barbu, V.; Enachi, E.; Râpeanu, G.; Bahrim, G.E.; Oancea, A.; Stănciuc, N. Probing the functionality of bioactives from eggplant peel extracts through extraction and microencapsulation in different polymers and whey protein hydrolysates. *Food Bioproc. Tech.* 2019, 12, pp. 1316–1329. https://doi.org/10.1007/s11947-019-02302-1

33. Milea, Ş.A.; Aprodu, I.; Vasile, A.M.; Barbu, V.; Râpeanu, G.; Bahrim, G.E.; Stănciuc, N. Widen the functionality of flavonoids from yellow onion skins through extraction and microencapsulation in whey proteins hydrolysates and different polymers. *J. Food Eng.* 2019, 251, pp. 29-35. https://doi.org/10.1016/j.jfoodeng.2019.02.003

34. Vasile, M.A.; Milea, Ş.A.; Enachi, E.; Barbu, V.; Cîrciumaru, A.; Bahrim, G.E.; Râpeanu, G.; Stănciuc, N. Functional enhancement of bioactives from black beans and lactic acid bacteria into an innovative food ingredient by comicroencapsulation. *Food Bioproc. Tech.* 2020, 13, pp. 978–987. https://doi.org/10.1007/s11947-020-02451-8

35. Enache, I.M.; Vasile, A.M.; Enachi, E.; Barbu, V.; Stănciuc, N.; Vizireanu, C. Co-microencapsulation of anthocyanins from black currant extract and lactic acid bacteria in biopolymeric matrices. *Molecules* 2020, 25, 1700, pp. 1-12. doi: 10.3390/molecules25071700

36. Enache, I.M.; Vasile, A.M.; Enachi, E.; Barbu, V.; Stănciuc, N.; Vizireanu, C. Co-microencapsulation of anthocyanins from cornelian cherry fruits and lactic acid bacteria in biopolymeric matrices by freeze-drying: evidences on functional properties and applications in food. *Polymers* 2020, 12 (906), pp. 1–12. https://doi.org/10.3390/polym12040906.

37. Boeing, H.; Bechthold, A.; Bub, A.; Ellinger, S.; Haller, D.; Kroke, A.; Leschik-Bonnet, E.; Müller, M. J.; Oberritter, H.; Schulze, M.; Stehle, P.; Watzl, B. Critical review: Vegetables and fruit in the prevention of chronic diseases. *Eur. J. Nutr.* 2012, 51, pp. 637–663. https://doi.org/10.1007/s00394-012-0380-y

38. Solak, B.B.; Akin, N. Health benefits of whey protein: a review. *J. Food Sci. Eng.* 2012, 2, pp. 129-137.

39. Ghai, K.; Gupta, A.K.; Gupta, P.K. Pectin: A versatile biopolymer with numerous health benefits and medical uses. *J. Biol. Act. Prod. Nat.* 2012, 2 (4), pp. 250–255. https://doi.org/10.1080/22311866.2012.10719132

40. Enache, I.M.; Benito-Román, Ó.; Coman, G.; Vizireanu, C.; Stănciuc, N.; Andronoiu, D.G.; Mihalcea, L.; Sanz, M.T. Extraction optimization and valorization of the cornelian cherry fruits extracts: evidence on antioxidant activity and food applications. *Appl. Sci.* 2021, 11, p. 0729. https://doi.org/10.3390/app112210729

41. Popović, B.M.; Štajner, D.; Slavko, K.; Sandra, B. Antioxidant capacity of cornelian cherry (*Cornus mas* L.) – Comparison between permanganate reducing antioxidant capacity and other antioxidant methods. *Food Chem.* 2012, 134 (2), pp. 734–741. https://doi.org/DOI: 10.1016/j.foodchem.2012.02.170

42. Hassanpour, H., Yousef, H., Jafar, H., Mohammad, A. Antioxidant capacity and phytochemical properties of cornelian cherry (*Cornus mas* L.) genotypes in Iran. *Sci. Hortic.* 2011, 129 (3), pp. 459–463. https://doi.org/10.1016/j.scienta.2011.04.017

43. Stankovic, M.S.; Zia-Ul-Haq, M.; Bojovic, B.M.; Topuzovic, M.D. Total phenolics, flavonoid content and antioxidant power of leaf, flower and fruits from cornelian cherry (*Cornus mas* L.). *Bulg. J. Agric. Sci.* 2014, 20, pp. 358–363.

44. Rop, O.; Mlcek, J.; Kramarova, D.; Jurikova, T. Selected cultivars of cornelian cherry (*Cornus mas* L.) as a new food source for human nutrition. *Afr. J. Biotechnol.* 2010, 9 (8), pp. 1205–1210. https://doi.org/DOI: 10.5897/AJB09.1722

45. Spence, C. On the psychological impact of food colour. *Flavour* 2015, 4 (21), pp. 1-16. https://doi.org/10.1186/s13411-015-0031-3

Publisher's Note: JES stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.

Submission of manuscripts: jes@meridian.utm.md

THE EFFECT OF REDUCING THE QUANTITY OF SALT ON THE QUALITY AND ACCEPTABILITY OF GRISSINI

Natalia Suhodol *, ORCID: 0000-0002-5609-5139,
Eugenia Covaliov, ORCID: 0000-0003-4574-2959,
Olga Deseatnicova, ORCID: 0000-0003-4801-8173,
Aurica Chirsanova, ORCID: 0000-0002-1172-9900,
Vladislav Resitca, ORCID: 0000-0002-6063-1731,
Tatiana Capcanari, ORCID: 0000-0002-0056-5939,
Alina Boiștean, ORCID: 0000-0002-5374-5853

Technical University of Moldova, 168 Stefan cel Mare Blvd., Chisinau, Republic of Moldova
*Corresponding author: Natalia Suhodol, natalia suhodol@toap.utm.md

Abstract. The article proposes the technology of obtaining grissini type salt-free bakery products. Research has been carried out on reducing the salt content and replacing the water with fermentation products as kefir, whey, and sour borscht, in order to reduce the effect of the lack of salt on the product quality. The results showed that the complete exclusion of salt from the recipe had a negative effect on the dough stability, physicochemical properties and organoleptic characteristics of the baked products. Partial or total omission of salt resulted in higher dough rise ability and lower moisture content in the final product. Grissini made according to the classic recipe with salt, registered high appearance and sensory properties, while reducing the salt content results in products with a less pronounced crust and taste. The use of kefir, whey and sour borscht as water substitutes in the recipe contributed to the improvement of the chromatic parameters, as well as the physicochemical indicators of the baked grissini. However, consumers appreciated less the samples with the addition of kefir and whey. These samples being distinguished by a specific taste and smell, in some extent unusual for panelists. At the same time, grissini based on sour borscht were highly appreciated. According to the results, the sample with sour borscht without added salt recorded similar characteristics to the control sample, but in order to align it with national standards in terms of physicochemical indicators, the dough fermentation period needs to be revised.

Keywords: *acidity, grissini, kefir, sour borscht, whey.*

Rezumat. Articolul propune tehnologia de obținere a produselor de panificație de tip grissini fără sare. Au fost efectuate cercetări privind reducerea conținutului de sare și înlocuirea apei cu produse de fermentare, precum chefirul, zerul și borșul acru pentru a reduce impactul negativ al lipsei de sare asupra calității produsului. Rezultatele au arătat că excluderea

completă a sării din rețetă influențează negativ atât proprietățile stabilitatea aluatului, cât și proprietățile fizico-chimice și calitățile organoleptice ale produsului copt. Omiterea parțială sau totală a sării a dus la o capacitate de creștere a aluatului mai mare și un conținut mai scăzut de umiditate în produsul final. În cazul grissinilor fabricate după rețeta clasică cu sare, aspectul și caracteristicile senzoriale au fost apreciate la cel mai înalt nivel, în timp ce diminuarea conținutului de sare duce la obținerea produselor cu crustă mai slab pronunțată și gust mai puțin evidențiat. Utilizarea chefirului, zerului și borșului acru ca înlocuitori ai apei în rețetă a contribuit la îmbunătățirea parametrilor cromatici ai grissinelor. Cu toate acestea, probele cu adaos de chefir și zer au fost mai puțin apreciate de consumatori, remarcandu-se printr-un gust și un miros specific, într-o oarecare măsură neobișnuit pentru paneliști. În același timp, grissini pe bază de borș acru au înregistrat valori similare apreciere înaltă chiar și în comparație cu cele clasice. Conform rezultatelor analizei senzoriale, proba cu borș acru fără adaos de sare a înregistrat caracteristici similare cu cele pentru proba de control, dar pentru a o alinia standardelor naționale în ceea ce ține de indicatorii fizico-chimici, perioada de fermentare a aluatului necesită a fi revizuită.

Cuvinte cheie: *aciditate, grissini, chefir, borș acru, zer.*

1. Introduction

Currently, it is relevant to expand the range of food products for preventive and therapeutic nutrition, which help to increase the duration and improve the human life quality. In ancient times, before access to salt sources, the daily consumption level was low (0.1 - 1.0 g/day). Nowadays, eating habits have changed substantially. Salt consumption differs from region to region and averages between 8.75 and 10.5 g/day [1]. The sodium content of processed foods is far above the norm, and the consumer cannot control them. Approximately 15% of dietary sodium is found in foods, and ~71% is added during food processing. Dietary sodium exists in a variety of chemical forms, such as sodium chloride (table salt), sodium bicarbonate and sodium glutamate that is a flavor enhancer found in processed foods. The human body, predetermined by evolution to conserve salt, could not adapt to increased salt intake. This is reflected in the growing number of people with hypertension and cardiovascular disease (CVD) and is putting pressure on the health care system [2].

Increased salt intake causes significant long-term complications that have physical, mental, social and economic consequences [3]. It causes and develops a range of noncommunicable diseases, including high blood pressure. The restriction of sodium in many treatments has shown beneficial effects, for these reasons it has become an adjunct therapy in several diseases involving high blood pressure, kidney disease, CVD, liver cirrhosis, etc. [4, 5].

According to World Health Organization (WHO) recommendations (5 g/day), adult salt intake in the Republic of Moldova is twice the maximum dose [6]. WHO target in Global Plan of Action to Support Government Efforts to Eliminate Noncommunicable Diseases is to reduce global salt consumption by 30% by 2025 [7]. For these reasons, the WHO has created several recommendations for reducing salt consumption among the population, which could lead to an additional year of healthy living for lower costs than the average annual income in low- and middle-income countries. One solution in this direction could be cooperation with industrial sectors to improve the quality and affordability of low-salt foods [8].

Bakery products are an essential source of salt for the human body. According the food pyramid, bread is consumed in large quantities, most often even 2-3 times a day,

especially among Moldovans. The traditional custom of greeting guests with bread and salt demonstrates that the Moldovan people consume these two products in excessive quantities. According to an annual publication by the National Bureau of Statistics of the Republic of Moldova, the energy value of a person's average daily food consumption in 2016 was 2441.7 kcal, the share of energy of bread and bakery products being 41.0%. The annual consumption of bread and bakery products was on average 116.8 kg/person, with significant variations from 126.8 kg/person, which is equal to 320 g/day [9].

Considering the high bread and respectively high salt consumption of the population of the Republic of Moldova, a prospective solution would be to create bakery products with low salt content. However, salt reduction has a significant effect on dough formation, for example dough rheological properties, and final product quality. Several studies mention that the optimal salt content in the dough composition of a bakery product is 1 – 2 % related to flour weight [10 – 12].

However, according to Carcea et al. (2020), quantities of salt above 1.5% related to flour weight are unnecessary to improve breadmaking quality of flours [13].

Adding salt to the dough decreases the flour's ability to absorb water, due to its dehydrating action on gluten. On one hand, this fact increases the dough development time. On the other hand, the addition of salt enhances the dough resistance, elasticity and extensibility.

McCann & Day (2013) explain this fact by the ability of salt of increasing the non-covalent interactions of the gluten, which results in the formation of a fibrous network structure [14]. Having a strong gluten network in bakery products, especially in the layered or leavened dough, is imperious because it ensures the entrainment of gas bubbles in the dough and their retention during fermentation and baking [11].

Thus, reducing or eliminating salt content from bakery products recipes can cause negative effects on the gluten network formation and dough rheological properties, influencing this way and some physical characteristics of the final product (volume, crumb structure, texture, etc.).

In a dough composition, salt inhibits yeast growth causing a decrease in their activity, leading to lower CO_2 production [15]. Salt also inhibits the activity of flour amylolytic enzymes that liberate maltose (a fermentable sugar) outside their optimal pH range [13, 16]. In the dough without salt, the yeasts activate intensely, consuming a large amount of sugars, and when placed in the oven, the dough no longer contains sufficient amounts of reducing carbohydrates to form sufficient melanoidins to confer a ruddy color to the final product's crust [17].

The aim of the research was to develop the technology of a salt-free bakery product and at the same time to replace the liquid phase with other products, which would reduce the negative effect of lack of salt on the product quality.

2. Materials and Methods
2.1. Materials
The raw materials used to prepare salt-free grissini were taken from the classic recipe [18], which involves the use of premium wheat flour [19], water [20], salt [21], sugar [22], bakery yeast (*Saccharomyces Cerevisiae*, press form) [23], sunflower oil [24], eggs [25] and sunflower seeds [26] were used for decoration. Kefir (2.5 % fat) [27], whey and sour borscht [28] were used in order to replace the water in the experimental samples.

2.2. Methods
Samples preparation

In this research the salt amount in the grissini's formulation was reduced to 50, 25 and 0 %. In the salt-free grissini recipe, the water was totally replaced with fermentation products such as kefir, whey and sour borscht. The purpose of replacing the water with the respective products is to provide an optimal salty taste and the appropriate texture. According to Plessas et al. (2005), kefir grains, besides providing nutritional value, contribute also to the extension of bakery products shelf life [29].

The main ingredients and the used amounts are presented in table 1. For each of the basic and auxiliary raw materials, preparatory operations were: sifting (for flour) in order to remove impurities but also for flour aeration, dosing (for salt, sugar, yeast), washing and disinfection with the required solutions (for eggs) in order to prevent infection with Salmonella or other pathogenic microorganisms. It was prepared the 20±1 % salt solution and 50±2 % sugar solution. The pressed bakery yeast was dissolved in water in a 1:3 ratio. The dough with a moisture content of 37.0 ±1 % was prepared applying the straight dough method. In order to ferment, the dough was left for 120±10 minutes at a temperature of 31±1 °C, rolled out and molded in the form of sticks (length -150 mm; width -10-15 mm), left for 25±2 minutes for proofing at a temperature of 41±1 °C and relative humidity 83±2 %. On the surface, the grissini were covered with eggwash and sprinkled with sunflower seeds that naturally have a certain sodium content, thus highlighting the taste of the products and at the same time compensating for the lack of salt. The products were baked in a preheated oven at 220±2 °C for 10±1 minutes.

Table 1

Grissini formulas

Raw material	Control	$G_{50\%salt}$	$G_{25\%salt}$	$G_{0\%salt}$	KG	WG	SBG
Wheat flour, g	100	100	100	100	100	100	100
Water, g	38.3	37.9	37.7	37.4	-	-	-
Kefir, g	-	-	-	-	45	-	-
Whey, g	-	-	-	-	-	40	-
Sour borscht, g	-	-	-	-	-	-	39
Salt, g	1.5	0.75	0.37	0	0	0	0
Bakery yeast (pressed form), g	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Sugar, g	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Sunflower oil, g	5.0	5.0	5.0	5.0	5.0	5.0	5.0
Sunflower seeds, g	50	50	50	50	50	50	50
Eggs, g	20	20	20	20	20	20	20

Note. $G_{50\%salt}$ – grissini with 50% salt; $G_{25\%salt}$ – grissini with 25% salt; $G_{0\%salt}$ – grissini with no added salt; KG - kefir grissini (no added salt); WG - whey grissini (no added salt); SBG – sour borscht grissini (no added salt).

Dough Raising Capacity (DRC) was evaluated acording to Hamad et al. (2005), by assessing the volume of the dough during the fermentation process, using Eq. (1). The grissini dough was transferred to a 100 mL graduated cylinder and initial volume of the dough was noted down. The dough volume rise was noted after 40 minutes [30].

$$DRC\ (\%) = \frac{V_1 - V_0}{V_0} \times 100, \tag{1}$$

where: V_0 - volume of the dough before fermentation.

V_1 - volume of the dough after 40 minutes of fermentation.

Determination of grissini physicochemical indicators

Moisture content was determined by drying the samples in an oven at 130±2 ° C during 45 minutes, according to ISO 24557:2009 [31].

Total titratable acidity was determined according to AACC 02-31.01 [32].

Salt content was estimated by measuring chloride ion concentration, according to ISO 9297:1989 (Mohr's method) and mentioned by Jafri et al. (2017) [33].

Color evaluation. Grissini's crust color was analyzed by using a Minolta Chromameter as in Giannone et al. (2018). Color parameters L^* (*lightness*), a^**(redness)* and b^**(yellowness)* were determined [34]. In order to describe the color change, in addition to the L^*, a^* and b^* values, according to the equations (2) and (3), the following indicators were calculated [34, 35].

✓ brown index − *BI* (the higher it is, the more intense is the crust's brown color)*:*

$$BI = 100 - L \tag{2}$$

✓ total color difference − Δ*E*

$$\Delta E = \sqrt{(L_0^* - L^*)^2 + (a_0^* - a^*)^2 + (b_0^* - b^*)^2}, \tag{3}$$

where: L_0^*, a_0^*, b_0^* − color parameters of control sample.

L^*, a^*, b^* − color parameters of reduced salt and salt free grissini.

Sensory analysis. Food sensory evaluation measures and interprets people's reactions based on sight, smell, touch, taste and hearing. 15 specialists in the food industry field aged 22-60 years, were selected as panelists that performed sensory assessment of grissini sticks, using a 5-point scale ranging from 0 ("dislike extremely") to 5 ("like extremely") [36].

Consumer test was performed according to Pasqualone et al. (2019). A test involving 50 habitual bakery products consumers (25 male and 25 females, enrolled among students and employees of Technical University of Moldova, aged from 20 to 60 years old) was performed in order to rank preferences concerning the salt content and taste of grissini. Each consumer was offered samples of all types of prepared grissini, a glass of water and a tasting sheet. Thus, each consumer had to make a top of the 7 samples of grissini, with the qualifications: the most appreciated (score = 1), to the least appreciated (score = 7) [35].

Statistical analysis. All experiments were performed in triplicates. The results are given as mean ± standard deviation (SD). The data were statistically analyzed by ANOVA and Tukey tests (α = 0.05).

3. Results and Discussion

Effect of salt reduction on dough raising capacity

In this research, the dough samples were prepared with water, kefir, whey, and sour borscht. Each of these raw materials has a different chemical composition and nutritional value, so the fermentation process took place differently in each of them.

The table below shows the results of the dough rising (%) capacity at different time periods (table 2).

Table 2

Grissini dough rising capacity, %

Sample	Time, min				
	0	10	20	30	40
Control	n.d.	36.2 ± 0.2^{ab}	54.5 ± 0.6^{ab}	78.2 ± 0.6^{b}	96.4 ± 1.4^{a}
$G_{50\%salt}$	n.d.	30.4 ± 0.6^{a}	50.6 ± 1.0^{a}	70.8 ± 1.4^{a}	100.2 ± 2.2^{a}
$G_{25\%salt}$	n.d.	40.4 ± 0.2^{b}	60.8 ± 0.8^{b}	92.7 ± 2.0^{c}	106.2 ± 1.4^{b}
$G_{0\%salt}$	n.d.	52.2 ± 0.6^{c}	80.2 ± 1.2^{d}	110.2 ± 2.1^{e}	140.6 ± 3.1^{e}
KG	n.d.	40.6 ± 0.1^{b}	70.6 ± 0.6^{c}	94.3 ± 1.3^{c}	122.8 ± 2.6^{c}
WG	n.d.	42.4 ± 0.3^{b}	60.8 ± 1.2^{b}	100.5 ± 1.7^{d}	126.3 ± 2.4^{d}
SBG	n.d.	46.8 ± 0.5^{bc}	70.4 ± 0.7^{c}	104.8 ± 1.9^{de}	121.5 ± 2.5^{c}

Note: $G_{50\%salt}$ – grissini with 50% salt; $G_{25\%salt}$ – grissini with 25% salt; $G_{0\%salt}$ – grissini with no added salt; KG - kefir grissini (no added salt); WG - whey grissini (no added salt); SBG – sour borscht grissini (no added salt). n.d. – not determined. Results are expressed as mean ± standard deviation, insignificant ($p > 0.05$), in each column different letters ([a-e]) mean significant differences ($p < 0.001$).

The largest increase in volume was recorded for the grissini sample prepared on water, without added salt. In the first 10 minutes, the volume of the sample increased by 52%. The samples prepared on water with a reduced salt amount ($G_{50\%salt}$ and $G_{25\%salt}$) reached these values only after 20 minutes.

The presented data show that the dough samples prepared with salt addition started to rise more slowly in volume. This can be explained by the fact that salt slows down the activity of yeast in bread dough, by diverting the water from yeast cells. Struyf et al. (2017) which states that salt's presence in wheat flour dough induces both osmotic and ionic stress to the yeast cells reported similar results [39]. At the end of fermentation, the dough samples prepared on kefir, sour borscht, and whey basis, reached similar volumes to the salt free sample. It should be noted that after more than 40 minutes of fermentation, the KG, WG and SBG samples recorded large volumes with very large bubbles of gas, also having a specific sour smell of leavened dough. Similarly, Lynch et al. (2009) reported a significant increase in the maximum dough volume as the salt level decreases from 1.2% to 0.0%. The study also reported the negative effect of the reduced salt amount on dough gas holding capacity. The authors mention that at salt concentration below 0.3 % (w/w) the gluten network is weak and the formed CO_2 escapes from the dough [38].

Effect of salt reduction on baked grissini's sensory scores

The sensory attributes of grissini samples in terms of crust surface, crust color, taste and flavor, shape, crumb texture and crunchiness were evaluated after 12 hours from preparation. The results of the organoleptic examination of grissini samples with different salt concentrations are presented in the table 3.

Table 3

The results of the sensory evaluation for grissini

	Control	$G_{50\%salt}$	$G_{25\%salt}$	$G_{0\%salt}$	KG	WG	SBG
Crust surface	5.00 ± 0.00^{a}	4.80 ± 0.00^{b}	4.30 ± 0.00^{c}	3.55 ± 0.00^{b}	3.90 ± 0.00^{b}	4.10 ± 0.02^{b}	4.80 ± 0.02^{b}
Crust color	5.00 ± 0.00^{a}	4.90 ± 0.03^{a}	4.50 ± 0.00^{b}	3.50 ± 0.00^{b}	4.20 ± 0.01^{a}	4.10 ± 0.02^{b}	5.00 ± 0.00^{a}
Taste and flavor	4.80 ± 0.00^{b}	4.80 ± 0.01^{b}	4.60 ± 0.00^{b}	3.50 ± 0.00^{b}	3.90 ± 0.00^{b}	4.00 ± 0.02^{c}	5.00 ± 0.00^{a}

Continuation Table 3

Shape	5.00±0.00[a]	5.00±0.02[a]	4.80±0.00[a]	3.90±0.00[a]	4.20±0.05[a]	4.50±0.00[a]	4.95±0.02[a]
Crumb texture	4.80± 0.00[b]	4.90± 0.00[a]	4.80±0.00[a]	3.90±0.00[a]	4.25±0.05[a]	4.15±0.00[b]	5.00±0.00[a]
Crunchiness	4.80±0.00[b]	4.60±0.00[c]	4.60±0.00[b]	4.00±0.00[a]	4.20±0.02[a]	4.35±0.01[a]	4.90±0.02[a]
Average score	4.90±0.00[ab]	4.83±0.03[b]	4.60 ±0.04[b]	3.72±0.02[b]	4.10±0.02[b]	4.20±0.02[b]	4.94±0.04[a]

Note. Results are expressed as mean ± standard deviation, insignificant ($p > 0.05$), in each line different letters [a-c] mean significant differences ($p < 0.001$). $G_{50\%salt}$ – grissini with 50% salt; $G_{25\%salt}$ – grissini with 25% salt; $G_{0\%salt}$ – grissini with no added salt; KG - kefir grissini (no added salt); WG - whey grissini (no added salt); SBG – sour borscht grissini (no added salt).

The trends in consumer liking scores for grissini samples, were that the grissini based on sour borscht with no added salt had the highest average score of 4.94 (figure 1). This is probably due to the salt that is naturally found in sour borsch (fact proved and by the determined salt amount in table 4). From the panelists'point of view, SBG had a pleasant taste and flavor, a little sour, the lack of salt was not perceived. In contrast, the liking scores for KG and WG were lower, being characterized by an unusual taste and flavor, also the crust surface and color was less uniform due to the milk proteins that are still contained in these fermentative products and probably to the length of dough fermentation period.

In the case of all water based samples, a direct relation was established between the salt amount and taste score: once one decreases, so does the other. We did not detect a statistically significant difference among the control and $G_{50\%salt}$ and $G_{25\%salt}$ samples, but did detect a major one for the $G_{0\%salt}$ sample. The average score of the $G_{0\%salt}$ sample was 3,72. The sample received low acceptability scores for all the analysed characteristics, mainly for crust surface, crust color and taste and flavor.
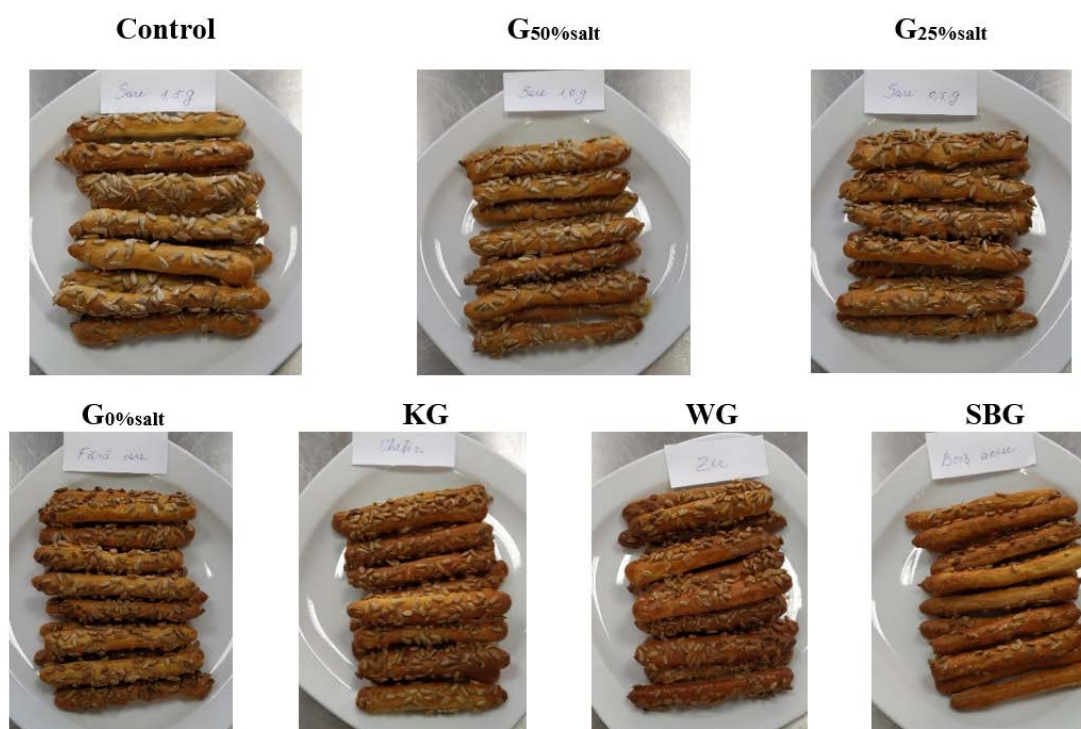
Figure 1. Sensory evaluation of grissini samples.

$G_{50\%salt}$ – grissini with 50% salt; $G_{25\%salt}$ – grissini with 25% salt; $G_{0\%salt}$ – grissini with no added salt; KG - kefir grissini (no added salt); WG - whey grissini (no added salt); SBG – sour borscht grissini (no added salt).

On one hand, in the view of some panelist, $G_{0\%salt}$ sample was dry and had no taste.

On the other hand, there were panelists that mentioned that the lack of salt is compensated by the taste of the sunflower seeds, confering to products a good and balanced taste. In their research Li et al. (2021) managed to reduce the salt content in bread by 20% (from 1.5 % to 1.2 % (w/w)) without impacting the consumer salt perception [37].

Also, in the study performed by Lynch et al., the panelists mentioned that salt free bread was yeasty, acidic and sour. The authors managed to reduce the salt content in bread from 1.2% to 0.3% (w/w) without significantly affecting the wheat dough rheological properties and bread-making performances [38].

Salt and moisture content of baked grissini

Determining the moisture content (W, %) in bakery and confectionery products is very important because it allows to establish the duration and storage conditions of the products, the low moisture content being associated with a longer shelf life [40]. According to the regulated physicochemical indicators established for grissini, the moisture content must not exceed 12.00% [43]. The moisture content as well as the salt content in grissini sticks are shown in table 4.

Table 4

Moisture and salt content in salt-free grissini

Sample	Moisture, %	Salt content NaCl, %
Control	11.80 ± 0.21^a	1.63 ± 0.10^a
$G_{50\%salt}$	11.66 ± 0.08^{ab}	0.88 ± 0.09^b
$G_{25\%salt}$	11.58 ± 0.06^b	0.50 ± 0.05^c
$G_{0\%salt}$	11.45 ± 0.08^c	0.12 ± 0.01^e
KG	11.73 ± 0.11^a	0.70 ± 0.04^b
WG	11.63 ± 0.14^{ab}	0.38 ± 0.01^d
SBG	11.57 ± 0.07^b	0.53 ± 0.02^c

Note. Results are expressed as mean ± standard deviation, insignificant (p > 0.05), in each line different letters ([a-e]) mean significant differences (p < 0.001). $G_{50\%salt}$ – grissini with 50% salt; $G_{25\%salt}$ – grissini with 25% salt; $G_{0\%salt}$ – grissini with no added salt; KG - kefir grissini (no added salt); WG - whey grissini (no added salt); SBG – sour borscht grissini (no added salt).

Regarding moisture content, all the researched samples fall within the limits established by the technical regulations for grissini, ranging from 11.45 to 11.80 %, the highest value being obtained for the control sample. However, taking into account that the dough moisture content was the same in all cases, and that the differences among the moisture content values in grissini are not significant, these can be considered experiment errors.

The highest NaCl content (1.63 %) was found in the control sample. The lowest value of NaCl concentration was registered in the $G_{0\%salt}$ sample, in which no salt was added, with a value of 0.12 %. This is due to the fact that a small amount of salt is also found in the eggwash, as well as in the sunflower seeds. In fact, according to national and european legislative regulations this is the only sample that can be called "product with a reduced salt content" [41, 42].

In the samples prepared on the kefir, whey and sour borscht basis with no added salt, the NaCl content recorded values between 0.40 and 0.70%, which shows that the used

fermentation products naturally contain salt. The highest NaCl content (0.70 %) was recorded for the KG sample, and the lowest (0.38 %) for the WG sample. The SBG sample, which was the most apreciated from sensory point of view, had a 0.53 % salt contet.

Effect of salt reduction on baked grissini's acidity

According to the national legislative regulations, the maximum allowable acidity for grissini-type bakery products made from premium wheat flour is 2.5 degrees [43], value marked with the horizontal red line in figure 2. The presented data indicate that the products prepared on water basis have the acidity that falls within the permissible limits. However, the salt content influenced the fermentation process, respectively in the control sample the acidity is 2.19 degrees, and in the salt free sample it reaches the value of 2.49 degrees. In the case of the KG, WG and SBG samples, the acidity recorded values above the admissible limit, ranging from 2.84 to 2.89 degrees, the highest value being obtained for WG.
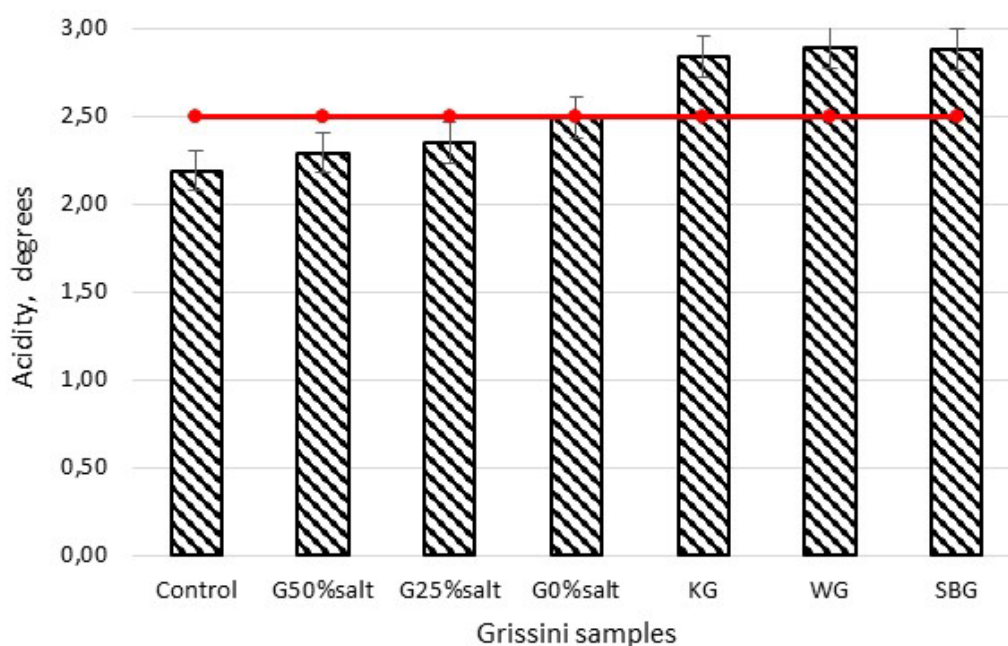
Figure 2. Effect of salt reduction on grissini's acidity.
$G_{50\%salt}$ – grissini with 50% salt; $G_{25\%salt}$ – grissini with 25% salt; $G_{0\%salt}$ – grissini with no added salt; KG - kefir grissini (no added salt); WG - whey grissini (no added salt); SBG – sour borscht grissini (no added salt).

On one hand, this can be explained by the natural presence of acids in whey, kefir or sour borscht [44]. On the other hand, the exceeding acidity may be caused by the fact that the fermentation period was the same for all samples, so a greater amount of acids was formed.

Thus, in the fermentation process the components of the kefir, whey and sour borscht would also have been involved. Probably shortening the fermentation period for this samples' dough would ensure acidity values of the baked products below the maximum admissible values, fact that will be taken into account in further research.

A greater acidity can influence to some extent the organoleptic qualities, namely the sour taste and the specific, less pleasant smell.

Contrastingly, numerous studies have shown that lactic acid produced by lactic acid bacteria found in whey, kefir or sour borscht has a positive effect on the inhibition of pathogenic microorganisms [45-47].

Effect of salt reduction on baked grissini's color parameters

Grissini with a lower salt content showed a less colored crust. Chromatic parameters, including total color difference and brown index of grissini samples are shown in table 5.

Table 5

Color parameters of grissini

Sample	L^*	a^*	b^*	ΔE	BI
Control	60.32 ± 1.07^a	5.82 ± 0.11^a	32.28 ± 0.45^a	-	39.68 ± 1.14^a
$G_{50\%salt}$	62.87 ± 1.13^a	5.48 ± 0.08^a	32.02 ± 0.56^b	2.59 ± 0.02^a	37.13 ± 0.98^{ab}
$G_{25\%salt}$	65.40 ± 0.94^c	4.94 ± 0.17^c	31.59 ± 0.37^c	5.20 ± 0.08^b	34.60 ± 1.05^b
$G_{0\%salt}$	68.54 ± 0.74^d	4.04 ± 0.14^d	30.85 ± 0.78^e	8.53 ± 0.34^e	31.46 ± 1.65^d
KG	63.75 ± 1.26^{ab}	4.72 ± 0.07^b	30.35 ± 0.65^b	4.09 ± 0.02^a	36.25 ± 0.76^{ab}
WG	63.88 ± 1.08^c	4.22 ± 0.09^c	28.38 ± 1.02^d	5.52 ± 0.46^d	36.12 ± 1.68^c
SBG	63.04 ± 0.82^b	4.43 ± 0.21^d	30.88 ± 0.69^d	3.36 ± 0.58^c	36.96 ± 1.32^{bc}

Note. $G_{50\%salt}$ – grissini with 50% salt; $G_{25\%salt}$ – grissini with 25% salt; $G_{0\%salt}$ – grissini with no added salt; KG - kefir grissini (no added salt); WG - whey grissini (no added salt); SBG – sour borscht grissini (no added salt). Results are expressed as average ± standard deviation, insignificant ($p > 0.05$), in each column different letters ([a-e]) mean significant differences ($p < 0.001$).

The color parameters (a^*, b^* and L^*) significantly changed as salt level decreased, due to a lower Maillard reaction intensity [48]. This can be explained by the fact that salt reduction increases yeast's activity, leading to a decrease of the amount of free reducing sugars involved in Maillard Reaction. Belz et al (2012) listed numerous positive effects of salt on bakery products, including bread color (more intense), delayed fermentation and the availability of a higher amount of sugars that may be involved in Maillard and caramelization reactions [49].

The data from table 5 clearly show that with salt reduction the L^* value significantly increased ($p > 0.05$) from control (60.32) to $G_{0\%salt}$ (68.54). In case of the samples with water substituents (KG, WG, SBG) same tendency was observed, but the L^* uptrends were not as sharp as in the case of water-based samples. On the contrary, the values of redness parameter (a^*) showed a slight decrease with the decrease of salt concentration, ranging within the limits 5.82 for the control and 4.04 for $G_{0\%salt}$. A similar decreasing trend was observed and for the b^* values, minimum value was recorded for the WG sample (28.38), while the maximum was in the case of control sample (32.28). The decrease of a^* and b^* values could be caused by the decrease of the Maillard reaction intensity between reducing sugars with the amino acids.

The values obtained for ΔE and BI, parameters that actually emphasize the effect of the salt content on the grissini's color changes to some extent are consistent with the data presented for the salt content. With the decrease of the salt content in the grissini's formulation, an increase of the total color difference is observed, which reaches values of 8.53 in case of total exclusion of salt. An opposite trend is observed in the case of BI evolution that reaches minimum values when salt was totally excluded.

Consumer test

The mean appreciation scores of reserched grisini samples depending on their salt content are reported in figure 3. The KG and WG samples were not subjected to this test because their assessment would have been influenced by the "unusual" taste and flavor mentioned by the panelists. From the ranking created by consumers, regarding the "favorite salty" taste, the SBG sample took the leading place. On the 2nd place was ranked the control

sample, the accumulated score being without significant differences compared to SBG sample. Next to this samples were ranked $G_{50\%salt}$ ang $G_{25\%salt}$.
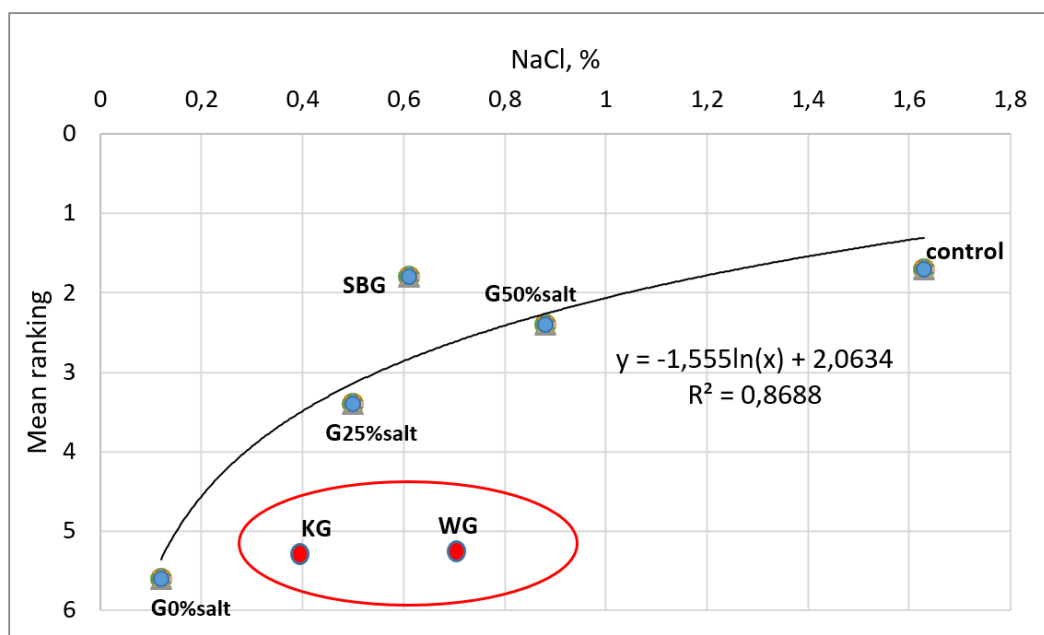
Figure 3. Mean ranking of grissini with different salt level.

$G_{50\%salt}$ – grissini with 50% salt; $G_{25\%salt}$ – grissini with 25% salt; $G_{0\%salt}$ – grissini with no added salt; KG - kefir grissini (no added salt); WG - whey grissini (no added salt); SBG – sour borscht grissini (no added salt).

Based on these, the SBG sample can be succesfully implemented in production, as long as the technology of production will allow to obtain grissini with acidity values within the admisible limits. Also, it can be concluded that salt intake can be reduced by up to 25 % without any changes in consumer preferences. Samples in which the salt was completely excluded obtained the lowest scores. As a result of processing the obtained scores, an exponential relationship was obtained that would allow the prediction of consumers' preferences regarding the acceptable salt level.

4. Conclusions

In this work, a simple method to reduce the salt level in grissini was reported. The results show that sour borscht, can be successfully used to replace the water in low or salt-free grissini in order to compensate the lack of salt and its impact on product quality. However, an aditional study is required in order to establish the optimal technological parameters for dough fermentation so as to maintain the acidity below 2.5 degrees.

The determination of the NaCl content in the grissini showed that for the samples based on kefir the salt content was similar to the sample with only 50% of salt amount. Regarding the same indicator, the whey based sample was closer to the grissini with 25% salt. Thus, to completely reduce the salt content of a complex product, the salt content of all ingredients must be taken into account.

Reducing the salt content negatively influenced the taste and flavor profile, moisture retention during baking and color parameters of grissini, leading to a lower sensorial score and a less intensely colored crust (weaker Maillrd reaction), but had a positive effect on dough raising capacity. Based on the values obtained as a result of the sensory evaluation, but also of the consumer test, the salt level can be reduced in the grissini formulation up to 25%.

Concerning the water substituents in the recipe, all the grissini samples had a higher titratable acidity than the admissible value, fact due to their richiness in acids. Additional studies are underway to evaluate the optimal fermentation period and optimal water replacement ratio with fermentation products to maintain this index within the admissible limits.

Conflicts of Interest: The authors declare no conflict of interest.

References
1. Powles, J.; Fahimi, S.; Micha, R.; Khatibzadeh, S.; Shi, P.; Ezzati, M.; Engell, R. E.; Lim, S. S.; Danaei, G.; Mozaffarian, D. Global, Regional and National Sodium Intakes in 1990 and 2010: A Systematic Analysis of 24 h Urinary Sodium Excretion and Dietary Surveys Worldwide. *BMJ Open* 2013, 3 (12), e003733. https://doi.org/10.1136/bmjopen-2013-003733.
2. Harnack, L. J.; Cogswell, M. E.; Shikany, J. M.; Gardner, C. D.; Gillespie, C.; Loria, C. M.; Zhou, X.; Yuan, K.; Steffen, L. M. Sources of Sodium in US Adults From 3 Geographic Regions. *Circulation* 2017, 135 (19), pp. 1775–1783. https://doi.org/10.1161/CIRCULATIONAHA.116.024446.
3. Loughlin, E. A.; Judge, C. S.; Gorey, S. E.; Costello, M. M.; Murphy, R. P.; Waters, R. F.; Hughes, D. S.; Kenny, R. A.; O'Donnell, M. J.; Canavan, M. D. Increased Salt Intake for Orthostatic Intolerance Syndromes: A Systematic Review and Meta-Analysis. *The American Journal of Medicine* 2020, 133 (12), pp. 1471-1478.e4. https://doi.org/10.1016/j.amjmed.2020.05.028.
4. Ha, S. K. Dietary Salt Intake and Hypertension. *Electrolyte Blood Press* 2014, 12 (1), 7. https://doi.org/10.5049/EBP.2014.12.1.7.
5. Alonso, S.; Tan, M.; Wang, C.; Kent, S.; Cobiac, L.; MacGregor, G. A.; He, F. J.; Mihaylova, B. Impact of the 2003 to 2018 Population Salt Intake Reduction Program in England: A Modeling Study. *Hypertension* 2021, 77 (4), pp. 1086–1094. https://doi.org/10.1161/HYPERTENSIONAHA.120.16649.
6. Chirsanova, A.; Covaliov, E.; Capcanari, T.; Suhodol, N.; Deseatnicova, O.; Boistean, A.; Resitca, V.; Sturza, R. Consumer Behavior Related to Salt Intake in the Republic of Moldova. *Journal of Social Sciences* 2020, 3 (4), pp. 101-110. https://doi.org/10.5281/ZENODO.4296387.
7. Santos, J. A.; Tekle, D.; Rosewarne, E.; Flexner, N.; Cobb, L.; Al-Jawaldeh, A.; Kim, W. J.; Breda, J.; Whiting, S.; Campbell, N.; Neal, B.; Webster, J.; Trieu, K. A Systematic Review of Salt Reduction Initiatives Around the World: A Midterm Evaluation of Progress Towards the 2025 Global Non-Communicable Diseases Salt Reduction Target. *Advances in Nutrition* 2021, 12 (5), pp. 1768–1780. https://doi.org/10.1093/advances/nmab008.
8. Al-Jawaldeh, A.; Taktouk, M.; Chatila, A.; Naalbandian, S.; Al-Thani, A.-A. M.; Alkhalaf, M. M.; Almamary, S.; Barham, R.; Baqadir, N. M.; Binsunaid, F. F.; Fouad, G.; Nasreddine, L. Salt Reduction Initiatives in the Eastern Mediterranean Region and Evaluation of Progress towards the 2025 Global Target: A Systematic Review. *Nutrients* 2021, 13 (8), 2676. https://doi.org/10.3390/nu13082676.
9. Boaghi, E.; Capcanari, T.; Mija, N.; Deseatnicova, O.; Opopol, N. The Evolution of Food Products Consumption in the Republic of Moldova in the Demogrphic Transition Period. *Journal of Engineering Sciences* 2018, 4, pp. 74-81. https://doi.org/10.5281/ZENODO.2576744.
10. Collando-Fernandez, M. Breadmaking Processes. In *B. Caballero, Encyclopedia of Food Science and Nutrition Second Edition*; Academic Press: San Diego, CA, 2003, pp 627–634.
11. Cauvain, S. P. Reduced Salt in Bread and Other Baked Products. In *Reducing Salt in Foods*; *Elsevier* 2007, pp 283–295. https://doi.org/10.1533/9781845693046.3.283.
12. Arena, E.; Muccilli, S.; Mazzaglia, A.; Giannone, V.; Brighina, S.; Rapisarda, P.; Fallico, B.; Allegra, M.; Spina, A. Development of Durum Wheat Breads Low in Sodium Using a Natural Low-Sodium Sea Salt. *Foods* 2020, 9 (6), 752. https://doi.org/10.3390/foods9060752.
13. Carcea, M.; Narducci, V.; Turfani, V.; Mellara, F. A Comprehensive Study on the Influence of Sodium Chloride on the Technological Quality Parameters of Soft Wheat Dough. *Foods* 2020, 9 (7), 952. https://doi.org/10.3390/foods9070952.

14. McCann, T. H.; Day, L. Effect of Sodium Chloride on Gluten Network Formation, Dough Microstructure and Rheology in Relation to Breadmaking. *Journal of Cereal Science* 2013, 57 (3), pp. 444–452. https://doi.org/10.1016/j.jcs.2013.01.011.

15. Voinea, A.; Stroe, S.-G.; Codină, G. G. The Effect of Sodium Reduction by Sea Salt and Dry Sourdough Addition on the Wheat Flour Dough Rheological Properties. *Foods* 2020, *9* (5), 610. https://doi.org/10.3390/foods9050610.

16. Vihinen, M.; Mantsiila, P. Microbial Amylolytic Enzyme. *Critical Reviews in Biochemistry and Molecular Biology* 1989, 24 (4), pp. 329–418. https://doi.org/10.3109/10409238909082556.

17. Avramenko, N. A.; Tyler, R. T.; Scanlon, M. G.; Hucl, P.; Nickerson, M. T. The Chemistry of Bread Making: The Role of Salt to Ensure Optimal Functionality of Its Constituents. *Food Reviews International* 2018, 34 (3), pp. 204–225. https://doi.org/10.1080/87559129.2016.1261296.

18. Sbornik Receptur i Tehnologičeskih Instrukcij Po Proizvodstvu Dietičeskih Hlebobuločnyh Izdelij, Vyrabatyvaemyh Po Nacional'nym Standartam, 2012. M.: GOSNIIHP, 2012, 72p.

19. Decision of the Government of the Republic of Moldova No. 68 of 29.01.2009. Regarding the Approval of the Technical Regulations "Flour, Semolina and Cereal Bran". 2009. Available online: https://www.legis.md/cautare/getResults?doc_id=22139&lang=ro (accessed on 30 July 2022).

20. Low of the Parliament of the Republic of Moldova No. 182 of 19.12.2019. The Quality of Drinking Water. 2019. Available online: https://www.legis.md/cautare/getResults?doc_id=119769&lang=ro (accessed on 30 July 2022).

21. Decision of the Government of the Republic of Moldova No. 596 of 3.08.2011 (2nd appendix). Sanitary Regulation on Food Salt. (2011). Available online: https://www.legis.md/cautare/getResults?doc_id=110144&lang=ro (accessed on 30 July 2022).

22. Decision of the Government of the Republic of Moldova No. 774 of 03.07.2007. Regarding the Approval of the Technical Regulations "Sugar. Production and Marketing". 2007. Available https://www.legis.md/cautare/getResults?doc_id=114180&lang=ro (accessed on 30 July 2022).

23. Decision of the Government of the Republic of Moldova No. 229 of 29.03.2013. Regarding the Approval of the Sanitary Regulation on Food Additives. 2013. Available online: https://www.legis.md/cautare/getResults?doc_id=109707&lang=ro (accessed on 30 July 2022).

24. Decision of the Government of the Republic of Moldova No. 434 of 27.05.2010. Regarding the Approval of the Technical Regulations "Edible Vegetable Oils". 2010. Available online: https://www.legis.md/cautare/getResults?doc_id=86300&lang=ro (accessed on 30 July 2022).

25. Decision of the Government of the Republic of Moldova No. 1208 of 27.10.2008. Regarding the Approval of the Sanitary-Veterinary Rules Concerning the Marketing of Eggs for Human Consumption. 2008. Available online: https://www.legis.md/cautare/getResults?doc_id=111684&lang=ro (accessed on 30 July 2022).

26. Firm Standard No. 40383910-001:2008. Sunflower Seeds. [Product Label].

27. Decision of the Government of the Republic of Moldova No. 158 of 07.03.2019 Regarding the Approval of the Quality Requirements for Milk and Dairy Products. 2019. Available online: https://www.legis.md/cautare/getResults?doc_id=113282&lang=ro (accessed on 30 July 2022).

28. Firm Standard No. 40188568-001:2010. Sour Borscht. [Product Label].

29. Plessas, S.; Pherson, L.; Bekatorou, A.; Nigam, P.; Koutinas, A. Bread Making Using Kefir Grains as Baker's Yeast. *Food Chemistry* 2005, *93* (4), pp. 585–589. https://doi.org/10.1016/j.foodchem.2004.10.034.

30. Hamad, S.; Al-Eid, S. A Study on the Quality of Some Baker's Yeast Brands Collected from the Markets of the Eastern Region of Saudi Arabia. *Journal of Agricultural Science* 2005, 13 (1), pp. 85–95.

31. Nielsen, S. S. Determination of Moisture Content. In *Food Analysis Laboratory Manual*; Nielsen, S. S., Ed.; Food Science Texts Series; Springer US: Boston, MA, 2010; pp. 17–27. https://doi.org/10.1007/978-1-4419-1463-7_3.

32. AACC (Cereals & Grains Association) 02-31.01 Total Titratable Acidity.

33. Jafri, A.; El Kardi, Y.; Derouiche, A. Sodium Chloride Composition of Commercial White Bread in Morocco. *East Mediterr Health J* 2017, 23 (10), pp. 708–710. https://doi.org/10.26719/2017.23.10.708.

34. Giannone, V.; Giarnetti, M.; Spina, A.; Todaro, A.; Pecorino, B.; Summo, C.; Caponio, F.; Paradiso, V. M.; Pasqualone, A. Physico-Chemical Properties and Sensory Profile of Durum Wheat Dittaino PDO (Protected Designation of Origin) Bread and Quality of Re-Milled Semolina Used for Its Production. *Food Chemistry* 2018, 241, pp. 242–249. https://doi.org/10.1016/j.foodchem.2017.08.096.

N. Suhodol, E. Covaliov, O. Deseatnicova, A. Chirsanova, Vl. Resitca, T. Capcanari, A. Boiștean   *163*

35. Pasqualone, A.; Caponio, F.; Pagani, M. A.; Summo, C.; Paradiso, V. M. Effect of Salt Reduction on Quality and Acceptability of Durum Wheat Bread. *Food Chemistry* 2019, 289, pp. 575–581. https://doi.org/10.1016/j.foodchem.2019.03.098.

36. ISO 6658:2017. Sensory Analysis. Methodology. General Guidance; International Organization for Standardization: Geneva, Switzerland, 2017

37. Li, Y.-L.; Han, K.-N.; Feng, G.-X.; Wan, Z.-L.; Wang, G.-S.; Yang, X.-Q. Salt Reduction in Bread via Enrichment of Dietary Fiber Containing Sodium and Calcium. *Food Funct.* 2021, 12 (6), pp. 2660–2671. https://doi.org/10.1039/D0FO03126G.

38. Lynch, E. J.; Dal Bello, F.; Sheehan, E. M.; Cashman, K. D.; Arendt, E. K. Fundamental Studies on the Reduction of Salt on Dough and Bread Characteristics. *Food Research International* 2009, 42 (7), pp. 885–891. https://doi.org/10.1016/j.foodres.2009.03.014.

39. Struyf, N.; Van der Maelen, E.; Hemdane, S.; Verspreet, J.; Verstrepen, K. J.; Courtin, C. M. Bread Dough and Baker's Yeast: An Uplifting Synergy. *Comprehensive Reviews in Food Science and Food Safety* 2017, 16 (5), pp. 850–867. https://doi.org/10.1111/1541-4337.12282.

40. Smith, J. P.; Daifas, D. P.; El-Khoury, W.; Koukoutsis, J.; El-Khoury, A. Shelf Life and Safety Concerns of Bakery Products—A Review. *Critical Reviews in Food Science and Nutrition* 2004, 44 (1), pp. 19–55. https://doi.org/10.1080/10408690490263774.

41. The Approval of the Sanitary Regulation Regarding Nutritional and Health Claims Written on Food Products. GD No. 196 from 25-03-2011. Access Link: Https://Www.Legis.Md [in Romanian].

42. Commission Regulation (EU) No. 1047/2012 of 8 November 2012 Amending Regulation (EC) No 1924/2006with Regard to the List of Nutrition Claims. Off. J. Eur. Union (OJEU) 2012, L310,36–37.

43. Decision of the Government of the Republic of Moldova No. 775 of 03.07.2007. Bakery Products and Pasta. 2007. Available online: https://www.legis.md/cautare/getResults?doc_id=97589&lang=ro (accessed on 30 July 2022)

44. Pourbaba, H.; Anvar, A. A.; Pourahmad, R.; Ahari, H. Changes on Acidic Parameters and Probiotic Survival in the Kefir Using Lactobacillus Acidophilus and Lactobacillus Paracasei Complementary Probiotics during Cold Preservation. *Iran J Vet Med* 2021, No. Online First. https://doi.org/10.22059/ijvm.2020.313626.1005141.

45. Perczak, A.; Goliński, P.; Bryła, M.; Waśkiewicz, A. The Efficiency of Lactic Acid Bacteria against Pathogenic Fungi and Mycotoxins. *Archives of Industrial Hygiene and Toxicology* 2018, 69 (1), pp. 32–45. https://doi.org/10.2478/aiht-2018-69-3051.

46. Gao, Z.; Daliri, E. B.-M.; Wang, J.; Liu, D.; Chen, S.; Ye, X.; Ding, T. Inhibitory Effect of Lactic Acid Bacteria on Foodborne Pathogens: A Review. *Journal of Food Protection* 2019, 82 (3), pp. 441–453. https://doi.org/10.4315/0362-028X.JFP-18-303.

47. Bungenstock, L.; Abdulmawjood, A.; Reich, F. Evaluation of Antibacterial Properties of Lactic Acid Bacteria from Traditionally and Industrially Produced Fermented Sausages from Germany. *PLoS ONE* 2020, 15 (3), e0230345. https://doi.org/10.1371/journal.pone.0230345.

48. Silow, C.; Axel, C.; Zannini, E.; Arendt, E. K. Current Status of Salt Reduction in Bread and Bakery Products – A Review. *Journal of Cereal Science* 2016, 72, pp. 135–145. https://doi.org/10.1016/j.jcs.2016.10.010.

49. Belz, M. C. E.; Ryan, L. A. M.; Arendt, E. K. The Impact of Salt Reduction in Bread: A Review. *Critical Reviews in Food Science and Nutrition* 2012, 52 (6), pp. 514–524. https://doi.org/10.1080/10408398.2010.502265.

Publisher's Note: JES stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.

Submission of manuscripts: jes@meridian.utm.md

EFFECTS OF LACTOSE HYDROLYSIS AND MILK TYPE ON THE QUALITY OF LACTOSE-FREE YOGHURT

Liliana Popescu, ORCID: 0000-0003-3381-7511,
Viorica Bulgaru *, ORCID: 0000-0002-1921-2009,
Rodica Siminiuc, ORCID: 0000-0003-4257-1840

Technical University of Moldova, 168 Stefan cel Mare blvd., Chisinau, Republic of Moldova
*Corresponding author: Viorica Bulgaru, *viorica.bulgaru@tpa.utm.md*

Abstract. The purpose of the work was to investigate the influence of different lactose hydrolysis processes, the contribution of the enzyme and the milk type on the characteristics of the obtained lactose-free yogurt. The analysis was performed on non-hydrolyzed yogurt (control sample), the pre-hydrolyzed yogurt (that was hydrolyzed before fermentation), and the co-hydrolyzed yogurt (concurrent addition of β-galactosidase and starter culture). According to the obtained results, at the end of the fermentation time, an advanced hydrolysis degree was reached (over 80%) both for yogurt samples obtained from pre-hydrolyzed milk and obtained by co-hydrolysis. The optimal method from the economic point of view is to obtain yogurt by co-hydrolysis. The sensory quality of the yogurt samples obtained from hydrolyzed milk by co-hydrolysis was characterized by a better flavor than the control sample, for yogurt from both types of milk. This may be due to the availability of a greater amount of glucose for the production of aromatic compounds, a sweeter taste than natural yogurt, with a light caramel flavor, a firm coagulum, a porcelain appearance, without whey removal. Cow's milk yogurt showed higher viscosity values to goat's milk yogurt for both pre-hydrolyzed and co-hydrolyzed milk. Lactose hydrolysis determined the reduction of the syneresis index of the yogurt compared to the control samples. The studies led to the development of lactose-free yogurt with improved sensory and rheological properties recommended for people with lactose intolerance.

Keywords: *co-hydrolysis, pre-hydrolysis, β-galactosidase, fermentation, lactose, yogurt.*

Rezumat. În articol a fost investigată influenţa diferitelor procese de hidroliză a lactozei, tipul enzimei şi a laptelui asupra caracteristicilor iaurtului fără lactoză. Analiza a fost efectuată pe iaurt nehidrolizat (probă martor), iaurt pre-hidrolizat (care a fost hidrolizat înainte de fermentare) şi iaurt co-hidrolizat (adăugarea concomitentă de β-galactozidază şi cultura starter). Conform rezultatelor obţinute, la sfarşitul timpului de fermentaţie s-a atins un grad avansat de hidroliză (peste 80%) atît în cazul probelor de iaurt obţinute din lapte pre-hidrolizat cît şi în cazul celui obţinut prin co-hidroliză. Metoda optima, din punct de vedere economic, este obţinerea iaurtului delactozat prin co-hidroliză. Calitatea senzorială a

probelor de iaurt obținute din laptele hidrolizat prin co-hidroliză a fost caracterizată printr-o aromă mai bună decât proba martor. Acest lucru se poate datora disponibilității unei cantități mai mari de glucoză pentru producerea de compuși aromatici, un gust mai dulce decât iaurtul natural, cu o aromă ușoară de caramel, un coagul ferm, un aspect de porțelan, fără separare de zer. Iaurtul din lapte de vacă a prezentat valori mai mari ale vâscozității în comparație cu iaurtul din lapte de capră atât pentru laptele pre-hidrolizat, cât și pentru cel co-hidrolizat. Hidroliza lactozei a determinat reducerea indicelui de sineresă al iaurtului comparativ cu probele martor. Studiile au condus la dezvoltarea iaurtului fără lactoză, cu proprietăți senzoriale și reologice îmbunătățite, recomandat persoanelor cu intoleranță la lactoză.

Cuvinte cheie: *co-hidroliză, pre-hidroliză, β-galactozidază, fermentație, lactoză, iaurt.*

1. Introduction

Yogurt is a dairy product obtained by fermenting milk under the action of specific microorganisms from the *Streptococcus thermophilus* and *Lactobacillus delbrueckii subsp. bulgaricus* starter culture which can be supplemented with other probiotic cultures such as *Lactobacillus acidophilus* and *Bifidobacterium bifidus* [1]. Probiotics are live microorganisms that, administered in adequate amounts, confer health benefits to the consumer [2]. Consummation of yogurt and other fermented dairy products is considered to be beneficial for the digestive and general health of consumers [3]. Yogurt contains important amounts of protein, calcium, iodine and vitamin B_{12}, and can be recommended for people with high risk of obesity and cardiovascular diseases, both in children and adults. [4]. In addition, several regulatory bodies, including the European Food Safety Authority, have approved health claims related to the consumption of yogurt and the reduction of symptoms caused by inadequate lactose digestion [5]. Two different theories have been put forward to explain this phenomenon [6, 7].

It has been suggested that lactobacillus in fermented milk that synthesize lactase continue metabolic activity in the gastrointestinal tract, participating in (total or partial) lactose digestion. Monosaccharides are both consumed by bacteria and absorbed in the small intestine, so the symptoms of lactose intolerance are reduced. This hypothesis is valid only if the lactic acid bacteria in the yogurt and the lactase enzyme synthesized by them will pass the gastric juice barrier [7].

A second explanation that has been put forward suggests that the lactose in yogurt is better digested. This fact is due to the slower intestinal transit of viscous yogurt compared to liquid milk. For this reason, lactase synthesized in the small intestine will have longer time to digest lactose and therefore reduce the symptoms of intolerance [8].

Several bacterial strains have been tested in people with lactose intolerance. Lactose intolerance was measured based on increased hydrogen concentrations in exhaled air after lactose ingestion. Hydrogen accumulates as a result of the fermentation of unhydrolyzed lactose under the colon microflora action of people with lactose intolerance [9]. Short-term benefits have been demonstrated for *Lactobacillus reuteri* administration through respiratory hydrogen reduction and improved symptom score after 10 days [10]. Malolepszy et al., 2006 [11] show that after 43 days of eight lactic acid probiotics administration did not reduce the level of hydrogen in the exhaled air and respectively the lactose intolerance symptoms. Yet another study using two probiotics, *Lactobacillus casei strain Shirota* and *Bifidobacterium breve* showed a short-term benefit after four weeks and even after three months, when the probiotics had already been stopped [12]. A recent review of 15 studies on the use of 8

different probiotics to reduce symptoms of lactose intolerance led the authors to demonstrate that although efficacy varied, there was an overall positive benefit for this type of treatment. The rate of lactose hydrolysis in fermented dairy products is influenced by the microbial cells number or lactase activity as well as other factors, such as intracellular substrate transport, etc. [9, 13].

Therefore, the lactose in yogurt is better digested than that in milk, as a result of the microorganism's ability to synthesize lactase. However, a low lactose content in yogurt is not suitable for people with lactose intolerance [14, 15] Thus, the most reliable remedy seems to be complete lactose enzymatic digestion in yogurt. This can be done by adding β-galactosidase to milk before pasteurization (pre-hydrolysis) as well as adding β-galactosidase along with the starter culture after pasteurization of milk (co-hydrolysis).

Only a few studies refer to the lactose hydrolysis influence on the fermented milk characteristics. Some studies reported a reduction in fermentation time in the case of free lactose fermented milk [16], while others [17] observed an increase in fermentation time or lactose hydrolysis did not influence the fermentation process period.

Yogurt is most often produced from cow's milk, less often from goat's, sheep's, buffalo's milk, etc. Goat's milk is appreciated for its nutritional and hypoallergenic properties, and indicated in the diet of children allergic to bovine milk [18, 19]. Between 40-100% of patients allergic to cow's milk proteins tolerate goat's milk [20]. The importance of goat's milk consists in its high digestibility and nutritional value, as well as its dietary and nutraceutical characteristics [21]. Goat milk is an excellent source of high quality proteins and peptides [21], with important biological activity such as antimicrobial, antioxidant, immunomodulatory, antihypertensive, hypocholesterolemic and activities [22]. Recent research has also shown that goat milk has better digestibility, higher iron and magnesium bioavailability, and higher calcium and copper content than bovine milk [20, 23] and it is rich in niacin, thiamin, riboflavin and pantothenate [24]. Goat milk, due to its health benefits, can be the basis for the creation of new dairy products through enzymatic processes and membrane separation [25, 26]. The aim of the study was to investigate the different enzymes influence, lactose hydrolysis methods and the milk type on the obtained yogurt characteristics. For a systematic approach, non-hydrolyzed yogurt (control sample), pre-hydrolyzed yogurt (which was hydrolyzed before fermentation) and co-hydrolyzed yogurt (concurrent addition of β-galactosidase and starter culture) were performed simultaneously.

2. Materials and Methods

Materials. Commercial enzymes β-galactosidase obtained from *Bacillus licheniformis*, activity 5500 BLU·g/1 NOLA Fit 5500 (Chr. Hansen, Denmark) and β-galactosidase obtained from *Kluyveromyces lactis*, activity 5000 NLU·g/1 Maxilact LGi 5000 (Sedim Cedex – France), according to information provided by the manufacturer. Cow's milk with a 2.5% fat content, proteins 3.2% and lactose 4.75% produced by "Lapmol" S.R.L., Republic of Moldova. Goat's milk with a 5.5% fat content, protein 3.0% and lactose 4.45% produced by "Vilador" S.R.L., Republic of Moldova. Yogurt freeze drying starter cultures containing *Streptococcus thermophilus*, *Lactobacillus delsbrueckii subsp. bulgaricus, Lactobacillus acidophilus, Bifidobacterium* (YAB 352B, Sacco, Italy).

Lactose hydrolysis. In the case of lactose pre-hydrolysis, β-galactosidase enzymes were added before pasteurization. The pre-hydrolysis regime was carried out according to the following regimes: enzymes from *B. licheniformis* Nola Fit 5500 in 0.3% proportion, hydrolysis

time 4 hours at a temperature of 4-6°C and enzymes from *K. lactis* Maxilact LGi 5000 in 0.3% proportion, hydrolysis time 12 hours at temperature 4-6 °C [27]. The hydrolyzed milk was pasteurized at a temperature of 85°C for 10 minutes. In the case of lactose co-hydrolysis, the enzyme NOLA Fit 5500 and Maxilact LGi 5000, in 0.15% proportion, were added together with the starter culture to the pasteurized milk cooled to the temperature of 39 °C [27].

Milk fermentation. Fermentation of the control, pre-hydrolyzed and co-hydrolyzed sample was performed simultaneously at the fermentation temperature of 39°C in the same thermostat until pH 4.6. After fermentation, the yogurt samples were aged at 4°C for 24 hours. During the yogurt samples fermentation, the evolution of the pH and the degree of lactose hydrolysis was monitored. Yogurt samples were evaluated on the 1st and 14th days of storage to determine the evolution of sensory and rheological characteristics. All samples were done in triplicate and the analysis of the samples was repeated three times.

Analytical determination of a lactose. The free D-glucose concentration, as well as the D-glucose component of lactose was determined by glucose spectrophotometric method using the lactose test kit (k-LOLAC, Megazyme). The method includes pre-treatment steps to clarify and deproteinate samples and also to remove the high levels of free D-glucose in the samples. The determinations were carried out in accordance with the method for the measurement of lactose in low-lactose and lactose-free products under method [28]. The reading of the samples absorbance was carried out at a wavelength of 340 nm using UV Vis spectrophotometer Shimadzu UV-1900.

The yogurt samples **sensory analysis** was determined using the 5-point scoring scale according to ISO 22935-3:2009 [29]. The sensory properties of the individual yogurt samples were analyzed by a group of nine-member evaluators, who were selected in accordance with ISO 8586:2012 [30].

Syneresis. Syneresis was determined by centrifuging 10g of yogurt sample at 1600 rpm for 10 min. After centrifugation, the supernatant was decanted and weighed on an analytical laboratory balance [31].

Viscosity was determined with rotational viscometer Brookfield DV-III Ultra at the storage temperature of yogurt samples according to the method presented by Özge Dönmez et al. 2017 [32] with some modifications. Yogurt samples were mixed gently 30 times for homogenization. Viscosity was determined using spridler 04 at shear rate (75 rpm).

The **pH** was measured with a digital pH-meter (Si Analytics TitroLine 5000) at 20°C.

Statistical analysis. The variance analysis of the results was carried out by least square method with application of Student test and Microsoft Office Excel program version 2010. All assays were performed in triplicate. The experimental results are expressed as average ± SD.

3. Results and Discussion
Degree of hydrolysis of lactose in yogurt

The lactose hydrolysis degree of non-hydrolyzed yogurt samples (control sample), pre-hydrolyzed yogurt, and yogurt obtained by co-hydrolyzation are shown in table 1.

Table 1

Evolution of the lactose hydrolysis degree during milk fermentation

Sample code	Lactose hydrolysis degree, %	
	until fermentation	after fermentation
Cow milk		
C1 Control	0	12.57±0.65

Continuation Table 1

C2	Pre-hydrolyzate with NF	93.80±0.28	98.56±1.01
C3	Pre-hydrolyzate with M	89.76±1.95	97.03±0.37
C4	Co-hydrolyzed with NF	0	84.25±1.44
C5	Co-hydrolyzed with M	0	81.83±1.29
Goat milk			
G1	Control	0	13.98±0.65
G2	Pre-hydrolyzate with NF	92.15±1.09	96.47±0.94
G3	Pre-hydrolyzate with M	87.40±0.42	92.61±0.27
G4	Co-hydrolyzed with NF	0	82.77±1.11
G5	Co-hydrolyzed with M	0	80.02±0.72

Note. NF – Nola Fit 5500, M - Maxilact LGi 5000. Values in the table represent means of three replicated trials ± standard deviation.

According to the data presented in table 1, it was note that in the cow and goat milk control samples, the lactose hydrolysis was achieved only under the action of β-galactosidase synthesized by the starter culture lactic bacteria, the degree of hydrolysis reaching values of 12.57 and 13.98% respectively. At the end of the fermentation time, an advanced hydrolysis degree was reached (over 80%) both in the case of yogurt samples obtained from pre-hydrolyzed milk and the one obtained by co-hydrolysis. Therefore, the process of obtaining yogurt by co-hydrolysis reduced the production time by excluding the lactose pre-hydrolysis technological stage and reduced the production costs. The optimal method from the economic point of view is to obtain yogurt by co-hydrolysis.

Evolution of pH during milk fermentation. Establishing the fermentation time.

The influence of lactose hydrolysis (before or during fermentation) on fermentation time, yogurt sensory and rheological properties was investigated using two types of β-galactosidase enzymes and two milk types (cow's milk and goat's milk). In each trial (control, pre-hydrolyzed and co-hydrolyzed milk), fermentations were performed simultaneously with the same starter culture. The pH value evolution was determined by the lactic acid bacteria development from the starter culture, as well as by the lactose hydrolysis method (figure 1).

a)   b)
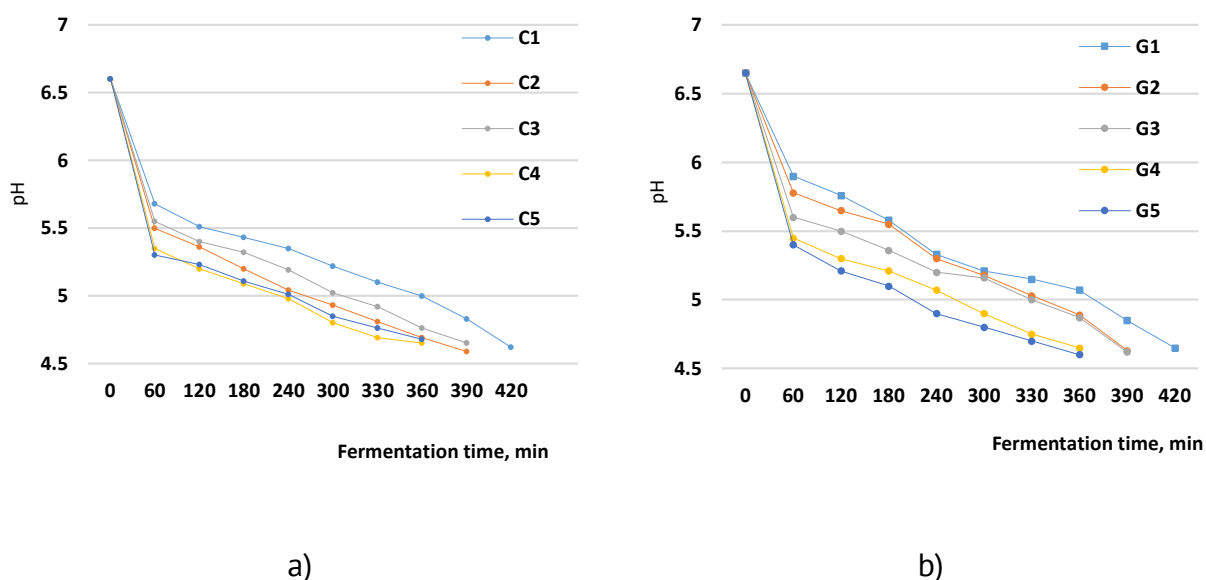
Figure 1. pH evolution during milk fermentation: a) cow's milk; b) goat's milk.

The increase in fermented milk acidity depends mainly on the type and amount of carbohydrates. To reach pH 4.60 the yogurt samples fermentation time generally varied between 5 and 7 hours. In yogurt samples obtained by milk fermentation in which the prior milk lactose hydrolysis was performed, the fermentation was accelerated by 30 minutes compared to the respective references, suggesting that the lactose hydrolisis improved the fermentation process.

The process of obtaining yogurt by co-hydrolysis further accelerated the fermentation, reducing the fermentation time from 420 to 360 minutes, during which the lactose hydrolysis and fermentation took place simultaneously, which led to an increase in the available glucose amount to the rapid multiplication of the starter microorganisms. It should be noted that upon simultaneous addition of β-galactosidase and starter culture, only a limited time remains for lactose hydrolysis. The enzymes used in the study are completely inactivated at a pH < 5.5 [27], which is reached after 2.5-3.0 hours of yogurt fermentation.

The decrease in the yogurt samples fermentation time obtained from pre-hydrolyzed milk or by co-hydrolysis is probably determined by the rapid lactic acid production from the first hours of fermentation. The goat's milk fermentation time was longer, while the gelation pH values were lower, compared to those of cow's milk, because of low levels of α_{s1}-casein and a higher level of β-casein in goat milk compared to cow's milk. This aspect characterizes the lower coagulation capacity of goat's milk, but which gives it a higher digestibility [33].

No significant differences between the used enzymes type were identified.

Schmidt et al. (2016) [34] published similar results in obtaining low-lactose yoghurt with five thermophilic yoghurt starters (mixtures of *Streptococcus thermophilus* and *Lactobacillus delbrueckii ssp. Bulgaricus)*. They demonstrated that the complete lactose hydrolysis before inoculation accelerated fermentation compared to the references. Sfakianakis et al. (2014) [35] presented the cooperation between *Streptococcus thermophilus* and *Lactobacillus bulgaricus*, namely the intensive production of lactase enzyme by *Streptococcus thermophilus* is necessary for the lactose hydrolysis and then *Lactobacillus bulgaricus* is capable of rapidly producing lactic acid from glucose as a result of the monosaccharides accumulation in milk from the lactose hydrolysis. The studies presented by Kárnyáczki et al. (2017) [36] claim the opposite, that pre-hydrolysis inhibits the activity of some yogurt cultures probably due to the transition from lactose to glucose as the main carbon source or due to the increase in osmotic pressure in lactase hydrolyzed milk.

Sensory analysis of yogurt samples

The yogurt sensory quality, after production and storage period, is one of the determining factors in consumer choice. To evaluate the acceptability of the lactose-free yogurt, sensory analysis was performed. The results of sensory analysis and overall acceptability were presented in table 2.

Yogurt samples are characterized by firm consistency curd, with a porcelain appearance, without removal of whey. Color was yellowish-white, uniform. The odor of yogurt is due to the lactic acid production, acetaldehyde and other carbonyl compounds during fermentation by the starter microorganisms.

Yogurt samples obtained from both pre-hydrolyzed and co-hydrolyzed milk are characterized by a better taste than the control sample, for cow's milk yogurt, the score increased from 4.24 (C1) to 5.00 for all lactose-free yogurt samples. The same thing is observed in the case of goat's milk yogurt samples This may be due to the availability of more glucose for the aromatic compounds production.

Evolution of yogurt samples sensory properties

Sensory properties	Yogurt samples	Cow milk		Goat milk	
		Storage time, days / Sensory scores on 5-point scale			
		1	14	1	14
Appearance and consistency	Control	4.05±0.07	4.25±0.10	3.92±0.02	4.05±0.05
	Pre-hydrolyzate with NF	4.62±0.19	4.76±0.19	4.42±0.03	4.63±0.08
	Pre-hydrolyzate with M	4.68±0.06	4.72±0.16	4.65±0.01	4.84±0.03
	Co-hydrolyzed with NF	5.00±0.00	5.00±0.00	5.00±0.00	5.00±0.00
	Co-hydrolyzed with M	5.00±0.00	5.00±0.00	5.00±0.00	5.00±0.00
Color	Control	5.00±0.00	5.00±0.00	5.00±0.00	5.00±0.00
	Pre-hydrolyzate with NF	5.00±0.00	5.00±0.00	5.00±0.00	5.00±0.00
	Pre-hydrolyzate with M	5.00±0.00	5.00±0.00	5.00±0.00	5.00±0.00
	Co-hydrolyzed with NF	5.00±0.00	5.00±0.00	5.00±0.00	5.00±0.00
	Co-hydrolyzed with M	5.00±0.00	5.00±0.00	5.00±0.00	5.00±0.00
Odor	Control	5.00±0.00	5.00±0.00	5.00±0.00	5.00±0.00
	Pre-hydrolyzate with NF	5.00±0.00	5.00±0.00	5.00±0.00	5.00±0.00
	Pre-hydrolyzate with M	5.00±0.00	5.00±0.00	5.00±0.00	5.00±0.00
	Co-hydrolyzed with NF	5.00±0.00	5.00±0.00	5.00±0.00	5.00±0.00
	Co-hydrolyzed with M	5.00±0.00	5.00±0.00	5.00±0.00	5.00±0.00
Taste	Control	4.24±0.02	4.47±0.02	4.18±0.06	4.35±0.03
	Pre-hydrolyzate with NF	5.00±0.00	5.00±0.00	5.00±0.00	5.00±0.00
	Pre-hydrolyzate with M	5.00±0.00	5.00±0.00	5.00±0.00	5.00±0.00
	Co-hydrolyzed with NF	5.00±0.00	5.00±0.00	5.00±0.00	5.00±0.00
	Co-hydrolyzed with M	5.00±0.00	5.00±0.00	5.00±0.00	5.00±0.00
Overall acceptability	Control	4.57±0.09	4.68±0.13	4.53±0.02	4.60±0.04
	Pre-hydrolyzate with NF	4.91±0.06	4.94±0.06	4.86±0.05	4.91±0.03
	Pre-hydrolyzate with M	4.92±0.10	4.93±0.07	4.91±0.02	4.96±0.03
	Co-hydrolyzed with NF	5.00±0.00	5.00±0.00	5.00±0.00	5.00±0.00
	Co-hydrolyzed with M	5.00±0.00	5.00±0.00	5.00±0.00	5.00±0.00

Note. NF – Nola Fit 5500, M - Maxilact LGi 5000. Values in the table represent means of three replicated trials ± standard deviation.

Also, the yogurt samples obtained from both pre-hydrolyzed and co-hydrolyzed milk are characterized by a considerably sweeter taste than natural yogurt, with a light caramel flavor. Regardless of the milk type, not detected obvious differences in sensory properties between yogurt from non-hydrolyzed and co-hydrolyzed milk.

Similar results were reported by Vènica et al. (2013) [37] who did not detect differences in sensory properties between drinking yogurt from non-hydrolyzed and co-hydrolyzed milk, while the experimental results reported by Ibarra et al. (2012) [17] demonstrated that the sensory quality of yogurt decreased with increasing degree of lactose hydrolysis. Yogurt obtained from milk with a lactose hydrolysis degree of 50 and 70% respectively (hydrolysis was carried out before fermentation) presented a creamier texture and a better flavor than yogurt from non-hydrolyzed milk, while lactose hydrolysis of 90% resulted in products with a softer consistency and an overly sweet flavor. Therefore, yogurt

samples obtained by lactose pre-hydrolysis or co-hydrolysis are characterized by a lactose hydrolysis high degree as well as a sweeter taste as a result of the accumulation of reducing monosaccharides with a higher sweet taste compared to that of lactose. In lactose-free or partially lactose-free yogurt, the amount of added sugar can be reduced, resulting in a product with a lower energy value.

The yogurt samples sensory analysis showed insignificant variations for color, odor, taste scores depending on the milk type and significant variations for appearance and consistency scores. Compared to cow's milk yogurts, goat's milk yogurt had a weaker consistency, ranging from 3.92 (G1) to 4.05 (C1), which also determined a decrease in the overall acceptability value. Lactose hydrolysis led to an increase in the score for the appearance and consistency of the yogurt samples, especially those obtained from goat's milk.

During the storage period, the sensory quality of the yogurt samples obtained by lactose pre-hydrolysis or co-hydrolysis did not change. So post-acidification during shelf life can be reduced as a result of lactose hydrolysis in yogurt. This can be explained by the fact that yogurt bacteria are less active in the absence of lactose or have difficulty switching from one carbon source (lactose) to another (glucose), which leads to a better sensory stability of the product [38].

Rheological properties of yogurt samples

When evaluating the yogurt quality, one of the most important aspects to consider is the rheological properties. Fermented dairy products have a highly sensitive gel-like protein matrix, which is highly dependent on the milk composition, but is also affected by the technological operations of homogenization, pasteurization, the microorganisms species in the starter culture and the fermentation temperature [34]. Two rheological parameters were measured in this study: viscosity and syneresis index (table 3).

Table 3

Evolution of yogurt samples rheological properties

Sample code		Storage time, days			
		Viscosity, Pa·s		Syneresis index, %	
		1	14	1	14
Cow milk					
C1	Control	0.69±0.02	0.51±0.01	41.65±0.78	40.24±1.49
C2	Pre-hydrolyzate with NF	0.76±0.02	0.73±0.01	37.89±0.73	36.77±0.51
C3	Pre-hydrolyzate with M	0.80±0.01	0.78±0.01	38.76±0.73	37.36±0.45
C4	Co-hydrolyzed with NF	0.92±0.01	0.86±0.02	36.67±0.23	36.03±0.68
C5	Co-hydrolyzed with M	0.96±0.01	0.90±0.01	36.41±0.98	35.76±0.45
Goat milk					
G1	Control	0.58±0.01	0.48±0.01	45.62±0.26	43.38±0.43
G2	Pre-hydrolyzate with NF	0.64±0.01	0.58±0.03	44.76±0.31	43.31±0.48
G3	Pre-hydrolyzate with M	0.71±0.01	0.70±0.02	44.37±0.44	43.37±0.44
G4	Co-hydrolyzed with NF	0.77±0.01	0.72±0.01	43.61±0.27	42.01±0.70
G5	Co-hydrolyzed with M	0.85±0.01	0.80±0.01	43.73±0.19	42.27±0.51

Note. NF – Nola Fit 5500, M - Maxilact LGi 5000. Values in the table represent means of three replicated trials ± standard deviation.

Differences in apparent viscosity at a shear rate of 75 rpm between the yogurt samples were more pronounced, while the storage-induced decrease in viscosity was negligible.

Sensory scores of the appearance and consistency correlated with the rheological properties expressed by the apparent viscosity and the syneresis index.The apparent viscosity of low-lactose yogurt (co-hydrolyzed with the Nola Fit enzyme from cow's and goat's milk) showed a significantly higher viscosity (by 33 and 23%, respectively) than the respective control samples. The control yogurt sample showed a significantly lower apparent viscosity value than the corresponding yogurt from pre-hydrolyzed and co-hydrolyzed milk, for the cow's milk yogurt from 0.69 Pa·s (C1) to 0.96 Pa·s (C5) and for the yogurt from goat's milk 0.58 Pa·s (G1) to 0.85 Pa·s (G5). This results may be due to the increased monosaccharides content that are more soluble and have a softer body and creamier texture or the exopolysaccharides concentration synthesized in a higher amount in yogurt samples from hydrolyzed milk compared to non-hydrolyzed milk [34].

Cow milk yogurt showed significantly higher apparent viscosity values compared to regular goat milk yogurt for both pre-hydrolyzed and co-hydrolyzed milk. This is mainly due to the higher content of α-casein in cow's milk compared to goat's milk, which allows the formation of a larger number of structure-relevant bonds in milk gels. The given results are in correlation with other authors [33, 39]. The syneresis index determines the susceptibility of lactose-free yogurt to water-binding capacity, which is an important sensory attribute from the consumer's point of view. Regardless of the milk type and the lactose hydrolysis process, the yogurt syneresis index varied between 36.41% and 44.76%. Lactose hydrolysis determined the reduction of the yogurt syneresis index compared to the control samples. The improved water-binding capacity caused by the lactose hydrolysis is a clear advantage in the yogurt manufacture, especially that obtained from goat's milk, it can increase the consumer acceptance of the final product and can also lead to the finished product shelf life extension.

5. Conclusions

Lactose hydrolysis in milk (before or during fermentation) influences the fermentation time, textural properties and sensory attributes of yogurt. In yogurt samples obtained from pre-hydrolyzed and co-hydrolyzed milk fermentation process was accelerated compared to the control samples, suggesting that lactose hydrolisis improves the fermentation process. At the end of the fermentation time, an advanced hydrolysis degree is reached (over 80%) both in yogurt samples obtained from pre-hydrolyzed milk and the one obtained by co-hydrolysis, in the case of yogurt samples from cow's milk and from goat's milk. Obtaining yogurt by co-hydrolysis reduces the yogurt production time by excluding the lactose pre-hydrolysis technological stage and, respectively, production costs. The economic effect is also confirmed by the results of the yogurt samples sensory and rheological analysis. Sensory properties of lactose-free yogurt are superior to those of natural yogurts. The yogurt samples obtained from both pre-hydrolyzed milk and co-hydrolyzed milk were characterized by a light caramel, sweeter taste than the control sample, which also led to an increase in the score from 4.24 (C1) to 5.00 for all samples of lactose-free cow's milk yogurt. The same is observed in goat milk yogurt samples.Consequently, in yogurt obtained by lactose co-hydrolysis, the amount of added sugar can be reduced, resulting in a product with a lower energy value. Lactose hydrolysis also caused an increase in yogurt viscosity compared to control samples, for the cow's milk yogurt from 0.69 Pa·s (C1) to 0.96 Pa·s (C5) and for the yogurt from goat's milk 0.58 Pa·s (G1) to 0.85 Pa·s (G5)Cow's milk yogurt showed significantly higher

apparentviscosity values compared to regular goat's milk yogurt. The improved water-binding capacity caused by the lactose hydrolysis is a clear advantage in the yogurt manufacture, especially that obtained from goat's milk, it lead to the increase the consumer acceptance of the final product from 4.53 in the case of yogurt sample G1 until 5.00 in the case of yogurt samples G4 and G5.

Conflicts of Interest: The authors declare no conflict of interest.

References
1. Codex Alimentarius, Milk and Milk Products. 2nd. Food and Agriculture Organization of the United Nations. Rome, 2011. Available online: https://www.fao.org/3/i2085e/i2085e00.pdf (accessed on 20 October 2022).
2. Hill, C.; Guarner, F.; Reid, G.; Gibson, G.; Merenstein, D.; Pot, B.; Morelli, L.; Canani, R.; Flint, H., Salminen, S. Expert consensus document. The International Scientific Association for Probiotics and Prebiotics consensus statement on the scope and appropriate use of the term probiotic. *Nat. Rev. Gastroenterol. Hepatol.* 2014, 11, pp. 506–514.
3. Ashraf, R.; Shah, N.P. Immune system stimulation by probiotic microorganisms. *Crit Rev Food Sci Nutr* 2014, 54, pp. 938–956.
4. Marette, A.; Picard-Deland, E. Yogurt consumption and impact on health: focus on children and cardiometabolic risk. *Am J Clin Nutr* 2014, 99, pp. 1243S–1247S.
5. European Food Safety Authority. Scientific Opinion on the substantiation of health claims related to live yoghurt cultures and improved lactose digestion. *EFSA Journal* 2010, 8, 1763.
6. Kies, A.K. Authorised EU health claims related to the management of lactose intolerance: Reduced lactose content, dietary lactase supplements and live yoghurt cultures. In: Sadler MJ., editor. *Foods, Nutrients and Food Ingredients with Authorised EU Health Claims.* Woodhead Publishing; Cambridge, UK 2014, pp. 177–211.
7. Savaiano, D.A. Lactose digestion from yogurt: Mechanism and relevance. *Am. J. Clin. Nutr.* 99, 2014, pp. 1251S–1255S.
8. Eri, Y.; Reiko, W.; Takefum, I.; Tatsuya, I.; Katsunori, K. Effect of lactose hydrolysis on the milk-fermenting properties of Lactobacillus delbrueckii ssp. bulgaricus 2038 and Streptococcus thermophilus 1131. J Dairy Sci 2021, 104 (2), pp. 1454-1464. doi: 10.3168/jds.2020-19244.
9. Szilagyi, A.; Ishayek, N. Lactose Intolerance, Dairy Avoidance, and Treatment Options. *Nutrients 2018,* 10 (12), 1994.
10. Ojetti, V.; Gigante, G.; Gabrielli, M.; Ainora, M.; Mannocci, A.; Lauritano, E.; Gasbarrini, G.; Gasbarrini A. The effect of oral supplementation with Lactobacillus reuteri or tilactase in lactose intolerant patients: Randomized trial. *Eur. Rev. Med. Pharmacol. Sci.* 2010, 14, pp. 163–170.
11. Malolepszy, P.; Shrier, I.; Szilagyi, A. Adaptation to lactose may not be achieved by long term ingetsion of a multi-species containing probiotic: An extended study. *Int. J. Probiotics Prebiotics* 2006, 1, pp. 113–120.
12. Almeida, C.C.; Lorena, S.L.; Pavan, C.R.; Akasaka, H.M.; Mesquita, M.A. Beneficial effects of long-term consumption of a probiotic combination of Lactobacillus casei Shirota and Bifidobacterium breve Yakult may persist after suspension of therapy in lactose-intolerant patients. *Nutr. Clin. Pract.* 2012, 27, pp. 247–251.
13. Oak, S.J.; Jha, R. The effects of probiotics in lactose intolerance: A systematic review. *Crit. Rev. Food Sci. Nutr.* 2018, 57, pp. 1–9.
14. Silanikove, N.; Leitner, G.; Merin, U. The Interrelationships between lactose intolerance and the Modern Dairy Industry: Global Perspectives in Evolutional and Historical Backgrounds. *Nutrients* 2015, 7, pp. 7312–7331.
15. Pachekrepapol, U.; Somboonchai, N.; Krimjai, W. Physicochemical, rheological, and microbiological properties of lactose-free functional yogurt supplemented with fructooligosaccharides. *Journal of food processing and preservation* 2021, 45(1), e15017.
16. Matijević, B.; Božanić, R.; Tratnik, Lj. Optimizing enzymatic hydrolysis of lactose in a sweet whey. In *4th IDF Dairy Science and Technology Week*, Reen., 2009, 65 p.

17. Ibarra, A.; Acha, R.; Calleja, M.-T.; Chiralt-Boix, A.; Wittig, E. Optimization and shelf life of a low-lactose yogurt with Lactobacillus rhamnosus HN001. *Journal of Dairy Science* 2012, 95(7), pp. 3536-3548.

18. Pradeep-Prasanna, P. H.; Charalampopoulos, D. Encapsulation in an alginate goats' milk inulin matrix improves survival of probiotic Bifidobacterium in simulated gastrointestinal conditions and goats' milk yoghurt. *International Journal of Dairy Technology* 2019, 72(1), pp. 132-141.

19. Beltrán, M. C.; Morari-Pirlog, A.; Quintanilla, P.; Escriche, I.; Molina, M. Influence of enrofloxacin on the coagulation time and the quality parameters of goat's milk yoghurt. *International Journal of Dairy Technology*, 2017, 71(1), pp. 105-111.

20. Gashaw Getaneh, D.; Mebrat, A.; Wubie, A.; Kendie, H. Review on Goat Milk Composition and its Nutritive Value. *Journal of Nutrition and Health Sciences* 2016, 3(4), 10 p.

21. López-Aliaga, I., Díaz-Castro, J.; Alférez, M.J.M.; Barrionuevo, M.; Campos, M. A review of the nutritional and health aspects of goat milk in cases of intestinal resection. *Dairy Sci. Technol.* 2010, 90, pp. 611–622.

22. Medeiros, G. K. V. V.; Queiroga, R. C. R. E.; Costa, W. K. A.; Gadelha, C.A.A; Lacerda, R.R.E; Lacerda, J.T.J.G; Pinto, L.S.; Braganhol, E., Teixeira, F.C.; Barbosa, P.P.; Campos, M.I.F.; Goncalves, G.F.; Pessoa, L.F.; Gadelha, T.S. Proteomic of goat milk whey and its bacteriostatic and antitumour potential. *International Journal of Biological Macromolecules* 2018, 113(1), pp. 116-123.

23. Fangmeier, M.; Kemerich, G. T.; Machado, B. L.; Maciel, M. J.; Souza, C. F. V. Effects of cow, goat, and buffalo milk on the characteristics of cream cheese with whey retention. *Food Science and Technology (Campinas)* 2019, 39(1), pp. 122-128

24. Ranadheera, C. S.; Evans, C. A.; Baines, S. K.; Balthazar, C.F.; Cruz, A.G.; Esmerini, E.A.; Freitas, M.Q.; Pimentel, T.C.; Wittwer, A.E.; Naumovski, N.; Graça, J.S.; Sant'Ana, A.S.; Ajlouni, S.; Vasiljevic, T. Probiotics in goat milk products: Delivery capacity and ability to improve sensory atributes. *Comprehensive Reviews in Food Science and Food Safety* 2019, 18, pp. 867-882.

25. Araújo, N.G.; da Silva, J.B.; Moreira, R.T.; Cardarelli, H.R. Effect of temperature and concentration of βeta-galactosidase on the composition of reduced lactose pasteurized goat milk. *Food Science and Technology Campinas* 2021, 41(2), pp. 432-438.

26. Cais-Sokolińska, D.; Wójtowski, J.; Pikul, J.; Dankow, R.; Majcher, M.; Teichert, J.; Bagnicka, E. Formation of volatile compounds in kefir made of goat and sheep milk with high polyunsaturated fatty acid content. *J Dairy Sci.* 2015, 98(10), pp. 6692-705.

27. Popescu, L.; Bulgaru, V.; Siminiuc, R. Effect of Temperature, pH and Amount of Enzyme Used in the Lactose Hydrolysis of Milk. *Food and Nutrition Sciences* 2021, 12, pp. 1243-1254.

28. Ivory R.; Delaney, E.; Mangan, D.; McCleary, B.V. Determination of lactose concentration in Low-Lactose and Lactose-Free Milk, Milk Products, and Products Containing Dairy Ingredients, Enzymatic Method: Single-Laboratory Validation First Action Method 2020.08. *Journal of AOAC International* 2021, 104 (5), pp. 1308–1322.

29. ISO 22935-3:2009 Milk and milk products. Sensory analysis. Part 3: Guidances on a method for evaluation of compliance with product specifications for sensory properties by scoring. International Organization for Standardization: Genova, Switzerland, 2009.

30. ISO 8586:2012 Sensory analysis. General guidelines for the selection, training and monitoring of selected assessors and expert sensory assessors. International Organization for Standardization: Genova, Switzerland, 2012.

31. Varnaite, L.; Kersǐene, M.; Sǐpailiene, A.; Kazernavičiūtė, R.; Venskutonis, P.; Leskauskaitė, D . Fiber-Rich Cranberry Pomace as Food Ingredient with Functional Activity for Yogurt Production. *Foods* 2022, 11, 758.

32. Özge Dönmez, B.; Ataç, M.; Vural, G. Syneresis and rheological behaviors of set yogurt containing green tea and green coffee powders. *Journal of Dairy Science* 2017, 100(2), pp. 901-907.

33. Bulgaru, V.; Cuşmenco, T.; Macari, A.; Botezat, O. Reological and textural properties of goat's milk and mixture of goat's and cow's milk fruit yogurt. *Journal of engineering science* 2020, 27(4), pp. 172-182.

34. Schmidt, C.; Mende, S.; Rohm, H. Fermented milk products: effects of lactose hydrolysis and fermentation conditions on the rheological properties. *Dairy Sci. Technol.* 2016, 96, pp. 199–211.

35. Sfakianakis, P.; Tzia, C. Conventional and innovative processing of milk for yogurt manufacture; development of texture and flavor: A review. *Foods* 2014, 3, pp. 176–193.

36. Kárnyáczki, Z.; Csanádi, J. Texture profile properties, sensory evaluation, and susceptibility to syneresis of yoghurt prepared from lactose-free milk. *Acta Aliment.* 2017, 46 (4), pp. 403–410.

37. Vénica, C.I.; Bergamini, C.V.; Zalazar, C.A.; Perotti, M.C. Effect of lactose hydrolysis during manufacture and storage of drinkable yogurt. *J Food Nutr Disord* 2013, 2, 5 p.

38. Khabibullaev, J.; Zagorska, J.; Galoburda, R.; Cinkmanis, I. Rheological properties of lactose-free yoghurt inrelation to enzyme concentrations. *Conference: 13th Baltic Conference on Food Science and Technology "Food. Nutrition. Well-Being"* May 2019, 40-44.
39. Miocinovic, J.; Miloradovic, Z.; Hovjecki, M. Rheological and textural properties of goat and cow milk set type yoghurts. *International Dairy Journal* 2015, 58, pp. 43-45.

Submission of manuscripts: jes@meridian.utm.md

PHYTOPATHOGENIC MICROBIOTE OF SEA BUCKTHORN AND IMPACT ON STORAGE

Elisaveta Sandulachi, ORCID: 0000-0003-3017-9008,
Natalia Netreba *, ORCID: 0000-0003-4200-1303,
Artur Macari, ORCID: 0000-0003-4163-3771,
Iuliana Sandu, ORCID: 0000-0003-1266-3154,
Olga Boestean, ORCID: 0000-0002-0390-3550,
Irina Dianu ORCID: 0000-0001-8632-8987

Technical University of Moldova, 168 Stefan cel Mare Blvd., Chisinau, Republic of Moldova
∗Correspondence author: Natalia Netreba, *natalia.netreba@tpa.utm.md*

Abstract. Sea buckthorn (*Hippophae rhamnoides L.*) has considerable potential for the agri-food, pharmaceutical and cosmetic industries. This article presents an international and experimental research conducted on plantations in Dubasari district, Pohrebea village in the Republic of Moldova of the microbiota which is able to attack sea buckthorn plantations and reduce the harvest. This research includes changes in the microbiota that is able to attack sea buckthorn plants to reduce the harvest and shelf life of the berries. Information on potential diseases of sea buckthorn plantations, the causative agent and recommendations to reduce this risk are included in the article. The information presented is important for the development of sea buckthorn plant cultivation strategies and disease management also for improving the quality and preservation of post-harvest fruit.

Keywords: *Hippophae rhamnoides L., microbiota of sea buckthorn, microbiological investigation, safety indicators*

Rezumat. Cătina albă (*Hippophae rhamnoides L.*) are un potenţial considerabil pentru sectorul agro-alimentar, farmaceutic şi cosmetic. În acest articol este prezentat un studiu de sinteză internaţional şi experimental realizat pe plantaţiile din raionul Dubăsari, satul Pohrebea din Republica Moldova al microbiotei fitopatogene care poate ataca plantaţiile de cătină, diminua recolta şi calitatea fructelor. Studiul include identificarea microbiotei fitopatogene (fungice şi bacteriene) de pe suprafaţa organelor cătinei albe (a frunzelor, fructelor în diferite stadii de coacere), folosind metode de izolare şi obţinere a culturilot pure în condiţii de laborator. În baza studiilor efectuate a fost identificată microbiota fitopatogenă de origine micotică, bacteriană şi elaborate recomandări de prevenire şi combatere a bolilor cătinei albe. Informaţiile prezentate sunt importante pentru dezvoltarea strategiilor de cultivare a plantelor de cătină, gestionarea posibilelor boli, precum şi pentru îmbunătăţirea calităţii şi conservării fructelor post-recoltare.

Cuvinte cheie: *Hippophae rhamnoides L., microbiota cătinii, investigații microbiologice*, *indicatori de siguranță*.

1. Introduction

In recent years, sea buckthorn has attracted considerable attention from researchers around the world, especially for its relevant nutritional and medicinal value. Berries contain many bioactive substances, including organic acids, amino acids, flavones and vitamins [1, 2]. It should be noted that few scientific papers are published that address the issue related to the impact of the phytopathogenic microbiota associated with the sea buckthorn *(Hippophae rhamnoides L.)*. The authors of the publications [3-5] reported that the yeasts *Cryptococcus, Rhodotorula, Sporobolomyces* and *Aureobasidium pullulans* were associated with the early stage of fruit ripening. According to the information presented by Juliana Lukša et al. [6] depending on the ripening stage, sea buckthorn showed a significantly different fungal microbiota. Fungi from the genera *Aureobasidium, Taphrina* and *Cladosporium* were present on unripe berries, while ripe berries were dominated by *Aureobasidium* and *Metschnikowia*. In another study by Xue Zhou [7], the authors report that in the sea buckthorn microbiota, fungi from the genera *Ascomycota, Basidiomycota* and *Zygomycota* had the dominant weight. The distribution of rhizospheric fungi in sea buckthorn is determined by both environmental and geographic factors. The authors of the studies [7, 8] mention that of all the soil characteristics examined, altitude and pH had a significant effect ($p < 0.05$) on the rhizospheric fungal microbiota

2. Bacterial and fungal mycobiota associated with sea buckthorn fruits

Sea buckthorn is a fruit plant with a high content of complex nutrients that compete in the natural nutraceutical market sold internationally. Sea buckthorn (*Hirrorhae rhamnoides* L.) has been cultivated in Europe since the 1970s [9, 10]. Due to the high demand of sea buckthorn fruits in the food industry and the increased demand for non-thermally processed foods, plant microbiota may have an important role in evaluating the impact of fruits on food quality and human health.

The authors Lukša, J. [6] and Yang, M. [11] studied the predominant microbial taxonomic units on sea buckthorn fruits, presented by bacteria and fungi. The effect of plant age, season, and weather conditions was studied only in sea buckthorn plants on the microbiota of the rhizosphere [6, 12]. The fungal pathogen tolerates a period of low temperatures in the form of spores around the kidneys and in closed areas of the cortex. In early spring, under favorable conditions, primary infection occurs. Mushrooms intensively develop and grow until the appearance of buds and the first leaves. The most intense infection occurs at elevated and stable humidity for more than two nights [13].

- *Aureobasidium*

Aureobasidium pullulans, the most common saprophytic mold, is a major source of environmental pollution. The mold of the genus *Aureobasidium* is mainly found in places with a temperate climate - in the British Isles, the USA, Canada, Alaska, Antarctica, Europe and Russia [14]. The main habitat of this type of mold is forest soil, fresh water, air and leaf surfaces. *Aureobasidium* is a spoilage agent in fruits and fruit drinks, vegetables such as tomatoes, cereals. For the growth of this type of fungus, the optimal growth temperature is 25 °C, and high humidity - a_w (available water) 0.89−0.90, but they are able to grow in a fairly wide temperature range from 2 to 35 °C [14, 15]. *Aureobasidium pullulans* can usually be

separated from permanently wet surfaces. These fungi produce d-gluconic acid and d-glucono-1,4-lactone from saccharides, mainly glucose [16].

The study and analysis of the types and structure of molds on the surface of the berries makes it possible to determine a clear difference between immature sea buckthorn fruits and fruits that have reached full maturity. According to the data reported in the work [6], unripe sea buckthorn fruits were dominated by microorganisms from the genera *Aureobasidium, Taphrina, Cladosporium and Filobasidium (Cryptococcus),* while *Aureobasidium and Metschnikowia* predominated on ripe fruits. Juliana Lukša et al. [17], reported that fungal microorganisms of the *Dothioraceae* family predominated on sea buckthorn fruits, which at the lower taxonomic level were assigned to uncultivated *Aureobasidium*. The results of the study [17] are consistent with the conclusions of other authors [18, 19], which confirm that among the diversity of mold microflora on sea buckthorn, the most common representative is Aureobasidium sp.

- ### *Cryptococcus neoformans*

Crytococcus neoformans and *Crytococcus gattii* are pathogenic microorganisms, namely yeasts, that cause diseases that are life-threatening to humans [20]. The yeast cell is covered with a capsule consisting of polysaccharides; for this reason, cryptococcus is often referred to as "sugar yeast" [21]. *Crytocuccus neoformans* is a fungus that occurs almost everywhere in the environment, most often in the soil, which produces extracellular enzymes. Lasses and phospholipase B are localized in the cell wall and ensure the formation of melanin and the integrity of the cell wall. The phospholipase enzyme also contributes to the fungal infection of the host's lung tissues and the rapid spread of an infectious and inflammatory disease [22].

- ### *Rhodotorula*

Rhodotorula spp. belong to basidiomycete fungi, that is widely distributed in nature - in the air, soil, water, colonizes plants, fruits, humans and other mammals. The genus *Rhodotorula* includes eight species, some of which are pathogenic to humans - *Rhodotorula musilaginosa, Rhodotorula glutinis* and *Rhodotorula minuta* [23, 24]. Unlike ascomycetous yeasts such as *Saccharomyces spp.* and *Candida spp., Rhodotorula spp.* belong to the *Sporidiobolale* family. Typically, *Rhodotorula spp.* produce carotenoids that form pink to red colonies on agar [25].

- ### *Sporobolomyces*
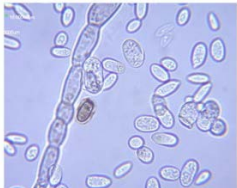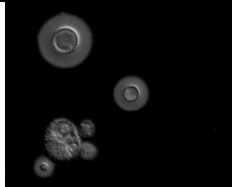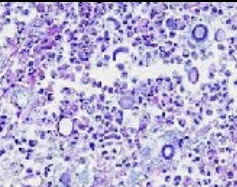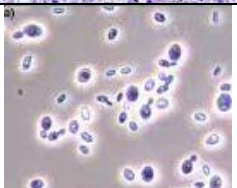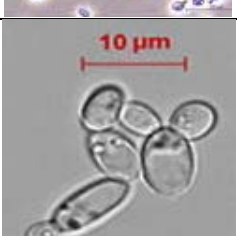
Sbololomyses is a yeast that naturally inhabits most environmental objects - air, tree leaves, many plants, birds, humans, and other mammals [26]. Microorganisms of the genus *Sporobolomyces*, like many fungi, are mesophilic, with an optimal growth and development temperature of 25-30 °C. Colonies are smooth, often wrinkled, from shiny to dull. Genus *Sporobolomyces* contains about 20 species that can cause infections [27].

Table 1 shows the results regarding the identification of microorganisms isolated from the sea buckthorn surface.
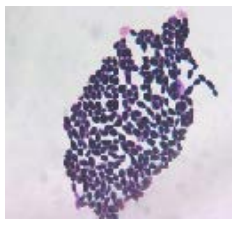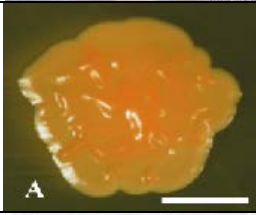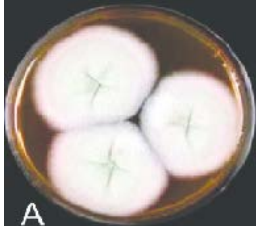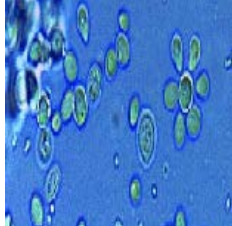
The purpose of the study by Maas P.W.Th. [30] was to evaluate the analysis of the influence of soil microflora on the root system and growth of sea buckthorn. The results of studies [31, 32] have shown that parasitic roundworms living in the soil alone or in combination with soil fungi can help retard the growth of sea buckthorn in the field, especially when the soil content and availability of nitrogen is low.

Table 1

Cultural and morphological properties of some microorganisms isolated from the surface of sea buckthorn fruits

Microorganism	Cultured cells	Microscope image	Year, country	Source
Aureobasidium pullulans			2020, Lithuania; 2018 Republic of Corea	[6] [28]
Cryptococcus wieringaie			2016, USA; 2012, USA	[20] [21]
Metschnikowia pulcherrima			2016, Basilicata	[29]
Pichia kudriavzevii			2020, Sweden	[30]
\Ascomycota			2017, China	[7]
Basidiomycota			2017, China	[7]
Zygomycot			2017, China	[7]
Taphrina			2020, Lithuania	[6]

Continuation Table 1

|				
Rhodotorula			1995, USA	[23, 24]
Sporobolomyces			1924, USA	[26]
Cladosporium			2020, Lithuania	[6]
Filobasidium			2020, Lithuania	[6]

Sea buckthorn grows best in sandy soils and requires abundant watering. Lack of moisture can lead to loss of foliage, damage to branches, shedding of fruits and, as a result, death of the tree.

The lack of water in the soil reduces the resistance of sea buckthorn trees to cold, increases the susceptibility to winter temperature changes, and also increases the level of attack by pests and diseases [33].

For a long time, farmers believed that sea buckthorn was not problematic in cultivation. To date, it has been proven that there are many health problems with sea buckthorn. Verticillosis and other fungi pose a great danger to the health and life of the plant, especially for sea buckthorn cultivated in Russia and Western Europe [9]. On the Figure 1 shows an analysis of healthy and diseased parts of sea buckthorn plants [33].

Verticillium was detected in only 2% of the analyzed samples. In moderately damaged and diseased parts of the plant, verticillosis was not found. The more the parts of the plant were affected, the more was the infection with microorganisms of the genera *Alternaria* and *Phytophora*.

Among other maintenance works of sea buckthorn plantations, special attention must be paid to the supply of water and nutrients to the plants. In Lithuania, sea buckthorn is harvested mainly on sites with irrigation and controlled fertilization [33]. Global standards for sea buckthorn are currently being developed. Consumers in Europe demand quality, so farmers must ensure that sea buckthorn has a clear identity based on combined product and service standards [9, 10].
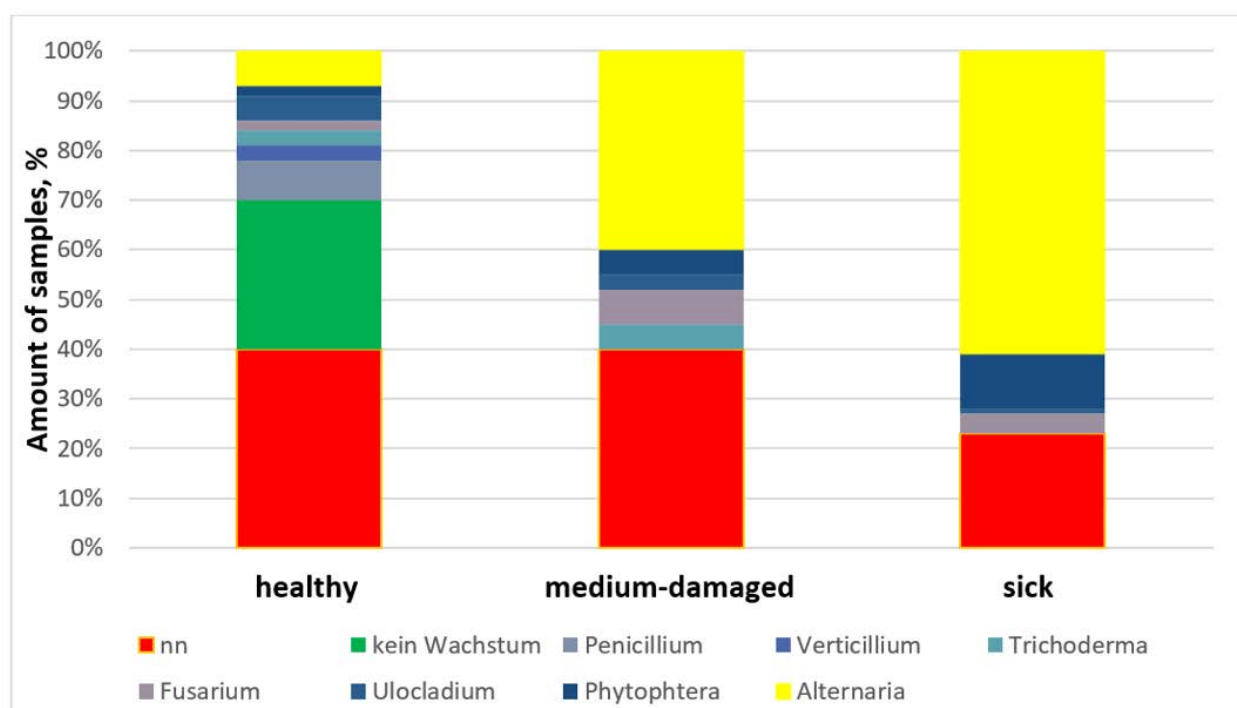
Figure 1. Species of fungi that were found from healthy, medium-damaged and diseased parts of sea buckthorn plants from Lithuania [33].

3. Diseases of the sea buckthorn culture and responsible agents

There are many studies on the relevant properties of *Hippophae rhamnoides* L. (chemical composition [34-38], antioxidant properties [39-42], antimicrobial properties [43-48]), but the study and research of sea buckthorn plantation diseases is limited. In general, only a few diseases were recorded, such as cancer caused by *Stigmina sp.,* wilt *(Verticillium spp.*), bud blight *(Pseudomonas syringae)* and dry shrinkage disease *(Fusarium spp.)*.

The most common diseases of sea buckthorn, scientists call verticillium wilt caused by soil fungi *Verticillium albo atrum* and *Verticillium dahliae*, fusarium wilt and rot caused by microorganisms *Pythium sp., Alternaria sp., Fusarium sp., Botrytis sp*. Verticillium wilt of sea buckthorn is a very dangerous disease and is widespread in both temperate and and in tropical regions of the world [49]. This disease usually appears in sea buckthorn plantations 5–8 years after planting [50, 51]. *Verticillium sresis* is widespread in many soils and more often affects tomatoes, peppers, strawberries, blackberries, sea buckthorn, etc. [52].

Fusarium wilt and rot are fungal diseases caused by *Fusarium oxysporum* that affect many perennial and annual plants. The causative agent, *Fusarium oxysporum*, overwinters in the soil. When the conditions for its development are favorable - moist, poorly drained soil - the fungus infects the roots of plants. It is extremely persistent in soil. For this reason it is necessary to extract the plant and it is also proposed to replace or sterilize the soil [53].

Another problem for sea buckthorn plantations is the fruit fly. Across Europe, for example, in Lithuania and Belarus, the sea buckthorn fruit fly is a big problem, while in Finland, Sweden and Estonia this problem does not exist [9, 49].

In recent years, growers are increasingly concerned about the problems related to diseases spreading in sea buckthorn plantations [51]. Since most of the infections that affect this plant are fungal, the diseases can be detected visually. Signs of disease onset include [54, 55]:
- yellowing, blackening, premature wilting and falling of leaves;

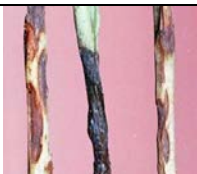- changes in the color of the bark of a tree, the appearance of stains, plaque, mold, outbreaks of rot on it;
- premature fall of berries, change in their consistency, drying, deterioration of quality;
- appearance of growths, neoplasms.

Table 2 shows the most common sea buckthorn diseases and the agents that cause these diseases.

Table 2

Sea buckthorn diseases

The Name	Image	Pathogen	Result	Source
Endomycosis. Sea buckthorn fruit rot		Soil fungi g. *Phytium, alternaria* and *Botrytis*	Affected berries soften, lesions appear on the fruit, fungal spores affect healthy fruits.	[9, 54, 55, 57, 66]
Wilting		g. *Fusarium Verticillium spp.*	Complete wilting of the plant. The shoots are affected, the leaves dry up, the fruits do not ripen, although they have a characteristic color.	[42, 54, 56-58]
Vertical wilting. Verticillosis		*Verticillium spp. Pseudomonas syringae*	Plants stop growing, dry up and wither prematurely. Intense rotting of the roots occurs.	[53, 55]
Sea buckthorn stem rot		*Coriolus hirsutus (Fr.) Quel*	Affected wood disintegrates into thin plates. Infection occurs through cracks, dry bays, mechanical damage to the bark.	[54]
Black cancer		*Stigmina sp.*	Characteristic black spots appear on the trunk, the affected bark cracks and falls off, and the wood underneath turns black.	[54]
Rust		g. *Puccinia*	The first symptoms - the appearance of chlorotic spots on the upper part of the leaves. The disease evolves, and orange colored pustules (fungal colonies) appear on the lower part, causing a severe deformation of the leaves.	[56, 59]
Brown spot		*Coniothyrium olivaceum Bon*	*Irregular light brown spots appear on the leaves, which grow rapidly. The disease manifests itself both on the fruit and on the bark of the branches.*	[54]

Continuation Table 2

Ulcerative necrosis of the cortex		A fungus that grows in the bark of a tree	At the site of the injury, an accumulation appears, which then breaks, the fungal spores affect the tree.	[54, 57]
Nectric necrosis		*Nectria cinnabarina (Tode) Wint*	*The bark of the tree is affected. Clumps of red spores appear throughout the film. The shoot affected by fungi dries up and dies over time.*	[54]
Blackleg		Soil fungi	*A disease caused by soil fungi affects seedlings. The subcotyledonous knee becomes thinner at the point of contact with the ground. The hawthorn stem simply spins and falls to the ground.*	[54]
Septoria stain		*Septoria hippophaes Desm. et Rob. Sacc.*	*On the upper side of the leaves, rounded, dark brown spots appear, surrounded by a discolored ring of leaf blade tissue. The frost resistance of sea buckthorn drops sharply.*	[54]
Scab Heterospory		*Heterosporium*	*Dark spots with a purple border appear on the surface of the leaves. Over time, they grow, the leaf completely fades.*	[54]

4. Materials and Methods

The study was carried out as part of the project: *Development of the production technology of white sea buckthorn in an ecological system and the processing of fruits and biomass* at the Technical University of Moldova.

Object of study. The purpose of this case study was to investigate the specific and phytopathogenic microbiota of 8 varieties of sea buckthorn fruits: R1, R2, R4, R5, L1, C6, AGG, AGA, harvested in September 2020 originating from Dubăsari district, Pohrebea village, Republic of Moldova [60, 61]. The harvesting of the white sea buckthorn fruits was carried out in the complete sweeping phase.

Methods. Sampling of sea buckthorn fruits was carried out in accordance with the SM SR ISO 874:2006 standard [62]. The microbiological investigation of the sea buckthorn samples was conducted according to the methods referred to in the sources [63-66]. Ten sea buckthorn fruits of each variety were placed in 50 cm^3 centrifuge tubes (three samples from each sea buckthorn variety) with 25 cm^3 of physiological solution and stored in a dark and cool place for further analysis (24 samples). For microbiological analysis, the sea buckthorn samples were kept in physiological solution no more than 24 hours after harvesting. The dilution method was usedfor the quantitative calculation of colony forming units (CFU) in determining the corresponding groups of microorganisms in 1 cm^3 of bacterial solution. Basic dilutions (10^1, 10^2, 10^3) were prepared as follows: 1 cm^3 of sample was added to a test tube

containing 9 cm^3 of saline (0.85%). In Petri dishes with a gel-like nutrient substrate, 1 cm^3 of a bacterial solution from the surface of sea buckthorn was inoculated in triplicate. For the cultivation of microorganisms, two types of nutrient media were used to separate groups of microorganisms: Sabouraud for yeast (5 days of incubation at 25 °C, aerobic cultivation method) and agar for bacteria (48 hours of incubation at 28 °C). After incubation, the results were analyzed and different types of microorganisms were identified. Colonies grown after preliminary microscopy were isolated in cultures and subjected to diagnostic testing for cultural and morphological characteristics, according to which yeast identification was performed. The bacterial culture was further identified by Gram staining.

4. Results

Table 3 shows the results of the microbiological analysis of the 8 varieties of sea buckthorn fruits: R1, R2, R4, R5, L1, C6, AGG, AGA, harvested in September 2020 originating from Dubăsari district, Pohrebea village, Republic of Moldova. The harvesting of the sea buckthorn fruits was carried out in the complete sweeping phase.

Table 3

The results of testing the microbiota of sea buckthorn on the surface of fruits in laboratory conditions

Varieties of sea buckthorn fruits	Detected microorganisms									
	P		**Cl**		**As**		**Rh**		**M**	
	+/total	**%**	**+/total**	**%**	**+/total**	**%**	**+/total**	**%**	**+/total**	**%**
R1	1/24	4.1	0/24	0	0/24	0	1/24	4.1	0/24	0
R2	0/24	0	0/24	0	0/24	0	0/24	0	1/24	4.1
R4	0/24	0	1/24	4.1	0/24	0	1/24	4.1	0/24	0
R5	0/24	0	0/24	0	0/24	0	0/24	0	0/24	0
L1	0/24	0	0/24	0	1/24	4.1	1/24	4.1	0/24	0
C6	0/24	0	1/24	4.1	0/24	0	1/24	4.1	1/24	4.1
AGG	1/24	4.1	0/24	0	0/24	0	1/24	4.1	0/24	0
AGA	0/24	0	0/24	0	0/24	0	0/24	0	0/24	0
Sea buckthorn fruits	**2/198**	**1.0**	**2/198**	**1.0**	**1/198**	**0.5**	**5/198**	**2.5**	**2/198**	**1.0**

Varieties of sea buckthorn fruits	Detected microorganisms								
	Al		**Ge**		**C**		**Pi**		
	+/total	**%**	**+/total**	**%**	**+/total**	**%**	**+/total**	**%**	
R1	0/24	0	0/24	0	0/24	0	0/24	0	**P-** *Penicillium:*
R2	0/24	0	0/24	0	0/24	0	1/24	4.1	**Cl-** *Cladospurium*
R4	0/24	0	1/24	4.1	0/24	0	0/24	0	**As-**-*Aspergillus niger*
R5	0/24	0	0/24	0	1/24	4.1	1/24	4.1	**Rh**-*Rhodotorula*
L1	0/24	0	1/24	4.1	0/24	0	0/24	0	**M-** *Mucor*
C6	1/24	4.1	1/24	4.1	1/24	4.1	1/24	4.1	**Al**-*Alternaria*
AGG	0/24	0	1/24	4.1	0/24	0	0/24	0	**Ge**-*Geotrichum*
AGA	1/24	4.1	0/24	0	0/24	0	0/24	0	**C-** *Candida*
Sea buckthorn fruits	**2/198**	**1.0**	**4/198**	**2.0**	**2/198**	**1.0**	**3/198**	**1.5**	**Pi**- *Pichia*

In the selected samples of sea buckthorn, the following were determined: the quantity of mesophilic aerobic and facultative anaerobic microorganisms – QMAFAnM (CFU)*, the total number of yeasts and molds (CFU), the presence/absence of coliform bacteria and *Salmonella*. The results of microbiological control of sea buckthorn fruits are included in Table 4.

Table 4

Quality and safety indicators of sea buckthorn

Microbiological indicators	Admitted level [67]	Experimental data
QMAFAnM, CFU, max.	$5*10^4$	$<1*10^2$
Coliform bacteria	Not allowed in 0.1 g	Were not found
Pathogenic microorganisms, including *Salmonella*	Not allowed in 25 g	Were not found
Yeast, CFU, max.	$2*10^2$	<10
Fungi, CFU, max.	$5*10^2$	<50

Note. QMAFAnM – quantity of mesophilic aerobic and facultative anaerobic microorganisms; CFU – colony-forming unit.

4. Discussions

The surface of the sea buckthorn fruit was inhabited by various types of bacteria, molds and yeast. The descriptions obtained from the microscopy were compared with the descriptions of other authors.The analyzed sea buckthorn microbiota is represented by Gram pozitive bacteria of the genus *Micrococcus, Staphylococcus, Lactobacillus*, *Streptococcus* and Gram negative bacteria of the genus *Acetobacter*. There were isolated and identified 9 different species of eukaryotes of the genius *Penicillium, Cladospurium, Aspergillus niger, Rhodotorula, Mucor, Alternaria, Geotrichum, Candida, Pichia*.

The results of the study of phytopathogenic microbiota of 8 varieties of sea buckthorn, collected in the phase of full ripeness, are the average values for 3 determinations for each variety. The percentage of detected microorganisms was calculated. The results of the study showed that the phytopathogenic microbiota identified on the studied sea buckthorn fruits formed the following percentage sequence: *Rhodotorula* (2.5%) > *Geotrichum* (2%) > *Pichia* (1.5%) > *Penicillium, Cladosporium, Mucor, Candida, Alternaria* (1%) > *Aspergillus niger* (0.5%).

Proper agricultural technology and timely disease prevention are the key to an abundant and healthy sea buckthorn harvest. The main cause of many diseases of sea buckthorn plantations *(Hippophae rhamnoides L.)* lies in the ignorance of the biological characteristics and the improper technology of growing horticultural crops.

According to the bibliographic study, in both Asia and Europe, sea buckthorn losses occur mainly due to environmental stress, wilt disease and insect pests.

Specialists [68-70] recommend a set of preventive measures against sea buckthorn diseases. Most sea buckthorn diseases are fungal infections, which thrive best in high humidity and temperature conditions. Therefore, the general rule is sanitary care for sea buckthorn, which consists of cleaning fallen leaves, thinning plantings, cutting dry, broken and diseased branches. Do not allow water to accumulate at the roots of the shrubs.

According to specialists in the field, an important step in preventing the development of parasitic fungi would be to spray sea buckthorn plantings with 1% Bordeaux liquid solution

or its analogues. This should be done in early spring, before the growing season, and in autumn, after leaf fall. After the procedure of cleaning sea buckthorn trees, all sections should be treated with a solution containing copper, then processed with natural oil paint. This will prevent the development of infection.

On dense plantations, the probability of fungal infection increases significantly, the danger is greater when soil and air humidity are higher.

5. Conclusions

Based on the bibliographic study, it was found that the phytopathogenic microbiota of the sea buckthorn plantations varies depending on the climatic factors, the fruit harvesting period, the location of the sea buckthorn plantation, etc. Monitoring the microbiota present on the berry surface could be a suitable tool for the control of phytopathogenic and potentially antagonistic microorganisms that affect the development and quality of sea buckthorn fruits. This information is highly relevant for developing strategies for organic plant cultivation, disease management and prevention.

Based on the experimental study, it was found that the surface of sea buckthorn fruits is dominated by microorganisms of the species *Rhodotorula, Geotrichum, Pichia*. These types of microorganisms are part of the specific microflora of sea buckthorn fruits and can be carried by vector insects such as fruit flies, bees and wasps. Microbiological control of sea buckthorn fruits showed that the phytopathogenic microbiota does not exceed the established sanitary standards. Thus, the tested varieties of sea buckthorn have good post-harvest properties, can be recommended for storage and can be used throughout the year for a variety of food products.

Conflicts of Interest: The authors declare no conflict of interest.

References
1. Ercisli, S.; Orhan, E.; Ozdemir, O.; Sengul, M. The genotypic effects on the chemical composition and antioxidant activity of sea buckthorn (Hippophae rhamnoides L.) berries grown in Turkey. *Scientia Horticulturae* 2007, 115(1), pp. 27–33.
2. Cho, Ch. H.; Jang, H.; Lee, M.; Kang, H.; Heo, H. J.; Kim D. Sea Buckthorn (Hippophae rhamnoides L.) Leaf Extracts Protect Neuronal PC-12 Cells from Oxidative Stress. *Journal of Microbiology and Biotechnology* 2017, 27(7), pp. 1257–1265.
3. Janisiewicz, W.; Kurtzman, C.; Buyer, J. Yeasts associated with nectarines and their potential for biological control of brown rot. *Yeast* 2010, 27, pp. 389–398.
4. Vadkertiová, R.; Molnárová, J.; Vránová, D.; Slávikováet, E. Yeasts and yeast-like organisms associated with fruits and blossoms of different fruit trees. *Canadian Journal of Microbiology* 2012, 58, pp. 1344–1352.
5. Barata, A.; Malfeito-Ferreira, M.; Loureiro, V. The microbial ecology of wine grape berries. International *Journal of Food Microbiology* 2012, 153, pp. 243–259.
6. Lukşa, J.; Vepštaitė-Monstavičė I.; Apšegaitė, V.; Blažytė-Čereškienė, L.; Stanevičienė, R.; Strazdaitė-Žielienė, Z.; Ravoitytė, B.; Aleknavičius, D.; Būda, V.; Mozūraitis, R.; Servienė, E. Fungal Microbiota of Sea Buckthorn Berries at Two Ripening Stages and Volatile Profiling of Potential Biocontrol Yeasts. *Microorganisms* 2020, 8, 456.
7. Zhou, X.; Tian, L.; Zhang, J.; Ma, L.; Li, X.; Tian, Ch. Rhizospheric fungi and their link with the nitrogen-fixing Frankia harbored in host plant. Hippophae rhamnoides L. *Journal of Basic Microbiology* 2017, 57(12), pp. 1055-1064.

8. Zhang, Y.; Bi, Y.; Shen, H.; Zhang, L. Arbuscular Mycorrhizal Fungi Enhance Sea Buckthorn Growth in Coal Mining Subsidence Areas in Northwest China. *Journal of Microbiology Biotechnology* 2020, 30(6), pp. 848–855.

9. Daniel, C. Experiences of integrated management of European Cherry Fruit Fly (Rhagoletis cerasi) and how to utilize this knowledge for Sea Buckthorn Fly. *In 3rd European Workshop on Sea Buckthorn, EuroWorkS 2014*, Naantali, Finland, 14-16 October 2014, pp. 21-30.

10. Eagle, D. Are quality standards necessary for sea buckthorn - a business perspective. In *3rd European Workshop on Sea Buckthorn, EuroWorkS 2014*, Naantali, Finland, 14-16 October 2014, pp. 44-48.

11. Yang, M.; Yang, D.; Yu, X. Soil microbial communities and enzyme activities in sea-buckthorn (Hippophae rhamnoides) plantation at different ages. *PLoS ONE* 2018, 13(1).

12. Kumar, S.; Sagar, A. Microbial associates of Hippophae rhamnoides (Seabuckthorn). *Plant Pathology Journal* 2007, 6, pp. 299–305.

13. Robert, V.; Stegehuis, G.; Stalpers, J. The MycoBank engine and related databases. International Mycological Association. 2005. 9-9-2009.

14. Institut national de santé publique du Québec. Available online: https://www.inspq.qc.ca/en/moulds/fact-sheets/aureobasidium-pullulans (accessed on 15.01.2021).

15. Flieder, F. Centre de recherche sur la conservation des documents graphiques. (2007). Moisissures et biens culturels. Ministère de la culture et de la Communication, France. http://journals.openedition.org/histoire-cnrs/679 (accessed on 25.03.2021).

16. Tomasik, P.; Horton, D. Enzymatic conversions of starch. *Advances in Carbohydrate Chemistry and Biochemistry* 2012, 68, pp. 59-436.

17. Lukša, J.; Vepštaitė-Monstavičė, I.; Yurchenko, V.; Serva, S.; Servienė, E. High content analysis of sea ˙ buckthorn, black chokeberry, red and white currants microbiota – A pilot study. *Food Research International* 2018, 111, pp. 597–606.

18. Vadkertiová, R.; Molnárová, J.; Vránová, D.; Sláviková E. Yeasts and yeast-like organisms associated with fruits and blossoms of different fruit trees. *Canadian Journal of Microbiology* 2012, 58, pp. 1344–1352.

19. Barata, A.; Malfeito-Ferreira, M.; Loureiro, V. The microbial ecology of wine grape berries. International *Journal of Food Microbiology* 2012, 153, pp. 243–259.

20. Chrissy, M.; Wager, L.; Camaron R.; Hole, C., Wozniak, K.; Wormley Jr.F. Cryptococcus and Phagocytes: Complex Interactions that Influence Disease Outcome. *Frontier in Microbiology* 2016, 7, pp. 1-16.

21. Mcdonald, T.; Wiesner, D.; Nielsen, K. Cryptococcus. *Current Biology* 2012, 22(14), pp. 554–555.

22. Microbiology Info.com 2021. Available online: https://microbiologyinfo.com/cryptococcus-neoformans/ (accessed on 20.10.2021).

23. Larone, Dh. *Medically Important Fungi: A Guide to Identification.* 3rd edition; ASM press, Washington, (D.C.), 1995, 274 p.

24. Wirth, F.; Goldani, L. Epidemiology of *Rhodotorula*: An Emerging Pathogen. *Interdisciplinary Perspectives on Infectious Diseases* 2012, 2012, pp. 1-7.

25. Hof, H. Rhodotorula spp. in the gut – foe or friend? *GMS Infect Dis.* 2019, 7, Doc02.

26. MSGERC DoctorFungus (Doctor Fungus). Available online: https://drfungus.org/knowledge-base/sporobolomyces-species/ (accessed on 15.10.2021).

27. Sutton, D.; Fothergill, A.; Rinaldi, A. *Guide to Clinically Significant Fungi.* 1st ed.; Williams & Wilkins, Baltimore, 1998, 471 p.

28. Lee, S.; Lee, H.; Kang, K. Identification of Aureobasidium pullulans from Sea Buckthorn (Hippohae rhamnoides L.) Fruits and Its Amylase Activities. *Journal of the East Asian Society of Dietary Life* 2018, 28 (3), pp. 225-230.

29. Tofalo, R.; Suzzi, G. *Yeasts. Encyclopedia of Food and Health* 2016. pp. 593-599.

30. Mozuraitis, R.; Aleknavicius, D.; Vepštaitė-Monstavičė, I.; Stanevičienė, R.; Emami, S.; Apšegaitė, V.; Radžiutė, S.; Blažytė-Čereškienė, L.; Servienė, E.; Būda, V. *Hippophae rhamnoides* berry related *Pichia kudriavzevii* yeast volatiles modify behaviour of *Rhagoletis batava* flies. *Journal of Advanced Research* 2020, 2, pp. 71-77.

31. Maas, P.W.Th; Oremus, P.A.I; Otten, H. Nematodes (Longidorus sp. and Tylenchorhynchus microphasmis Loof) in growth and nodulation of Sea Buckthorn (*Hippophaë rhamnoides* L.). *Plant and Soil* 1983, 73, pp. 141-147.

32. Zoon, F. Biotic and abiotic soil factors in the succession of sea buckthorn, Hippophaë rhamnoides L. in coastal sand dunes. Thesis Wageningen, Research Institute for Plant Protection (IPO-DLO), The Netherlands, 12.05.1995.

33. Brūvelis, A. Experiences about sea buckthorn cultivation and harvesting in Latvia. In *Producing Sea Buckthorn of High Quality. Proceedings of the 3rd European Workshop on Sea Buckthorn. EuroWorkS 2014*, Naantali, Finland,

October 14-16; Sanna Kauppinen, Ekaterina Petruneva; Publisher: Natural Resources Institute Finland (Luke), Helsinki, 2015.

34. Chandra, S.; Zafar, R.; Dwivedi, P.; Shinde, L.P.; Borkar P. Pharmacological and nutritional importance of sea buckthorn (Hippophae). *The Pharma Innovation Journal* 2018, 7(5), pp. 258-263.

35. Felipe L.; Alvarez G. Extraction et caractéristiques des huiles de l'argousier (*Hippophaë rhamnoides* L.). Chemistry, l'Université Laval, 2007,100 p.

36. Seglina, D.; Karklina, D.; Gailite, I.; Ruisa, S.; Krasnova, I.; Heidemane, G. Changes of biochemical compounds in seabuckthorn marc during storage. *Foodbalt-Proceedings* 2008, pp. 109-113.

37. Univer, T.; Jalakas, M.; Kelt, K. Chemical composition of the fruits of sea buckthorn and how it changes during the harvest season in Estonia. *Journal of Fruit and Ornamental Plant Research* 2004, 12, pp. 329-344.

38. Tiitinen, K.; Hakala, M.; Kallio, H. Quality Components of Sea Buckthorn (*Hippophaë Rhamnoides*) Varieties. *Journal of Agricultural and Food Chemistry* 2005, 53(5), pp. 1692–1699.

39. Ma, X.; Yang, W.; Laaksonen, O.; Nylander, M.; Kallio, H.; Yang, B. Role of Flavonols and Proanthocyanidins in the Sensory Quality of Sea Buckthorn (*Hippophaë rhamnoides* L.) Berries. *Journal of Agricultural and Food Chemistry* 2017, 65 (45), pp. 9871–9879.

40. Sytařová, I.; Orsavová, J.; Snopek, L.; Mlček, J.; Byczyński, L.; Mišurcová, L. Impact of phenolic compounds and vitamins C and E on antioxidant activity of sea buckthorn (Hippophaë rhamnoides L.) berries and leaves of diverse ripening times. *Food Chemistry* 2020, 310, 125784.

41. Kumar, M S Y.; Tirpude, R.J.; Maheshwari D.T.; Bansal, A.; Misra K. Antioxidant and antimicrobial properties of phenolic rich fraction of Seabuckthorn (Hippophae rhamnoides L.) leaves in vitro. *Food Chemistry* 2013, 141(4), pp. 3443-3450.

42. Chauhan, As; Negi, Ps; Ranteke, Rs Antioxidant and antibacterial activities of aqueous extract of sea buckthorn (Hippophae rhamnoides) seeds. *Fitoterapia* 2007, 78, pp. 590–592.

43. Dong, R.; Su, J.; Nian, H.; Shen, H.; Zhai, X.; Xin, H.; Han., T. Chemical fingerprint and quantitative analysis of flavonoids for quality control of Sea Buckthorn leaves by HPLC and UHPLC-ESI-QTOF-MS. *Journal of Functional Foods* 2017, 37, pp. 513-522.

44. Olas, B. Sea buckthorn as a source of important bioactive compounds in cardiovascular diseases. *Food and Chemical Toxicology* 2016, 97, pp. 199-204.

45. Yang, W.; Laaksonen, O.; Kallio, H.; Yang, B. Proanthocyanidins in Sea Buckthorn (Hippophaë Rhamnoides L.) Berries of Different Origins with Special Reference to the Influence of Genetic Background and Growth Location. *Journal of Agricultural and Food Chemistry* 2016, 64 (6), pp. 1274–1282.

46. Arimboor, R.; Kumar, K.; Arumughan, C. Simultaneous Estimation of Phenolic Acids in Sea Buckthorn (Hippophae Rhamnoides) Using RP-HPLC with DAD. *Journal of Pharmaceutical and Biomedical Analysis* 2008, 47(1), pp. 31–38.

47. Sandulachi, E.; Cojocari, D.; Balan, G.; Popescu, L.; Ghendov-Moşanu, A.; Sturza, R. Antimicrobial Effects of Berries on Listeria monocytogenes. *Journal of Food and Nutrition Sciences* 2020, 11, pp. 873-886.

48. Sandulachi, E.; Bulgaru, V.; Ghendov-Moşanu, A.; Sturza, R. Controlling the Risk of Bacillus in Food Using Berries. *Food and Nutrition Sciences* 2021, 12(6), pp. 557-577.

49. Jacobs, K.; Wingfield, M.; Gibbs, J. Pathology: vascular wilt diseases. In *Encyclopedia of Forest Sciences*, 1st ed.; Burley, J., Evans, J., Youngquist, J.; Elsevier Academic Press, Kidlington, Oxford, 2004, 2093 p.

50. Ruan, Cj.; Rumpunen, K.; Nybom, H. Advances in improvement of quality and resistance of a multipurpose crop: sea buckthorn. *Critical Reviews in Biotechnology* 2013, 33(2), pp. 126–144.

51. Du, Hj. Identification and screen of resistance to dried-shrink disease of sea buckthorn cultivars. *Hippophae* 2002, 15, pp. 13–14.

52. Du, Hj. Development and causative agent of shrivelled disease of sea buckthorn: survey and analysis. *Hippophae* 2001, 14, pp. 13–15.

53. Missouri Botanical Garden. Available online: https://www.missouribotanicalgarden.org/gardens-gardening/your-garde (accessed on 15.10.2021).

54. Sea buckthorn diseases and pests. Available online: https://ro.domesticfutures.com/bolezni-i-vrediteli-oblepihi-929 (accessed on 23.08.2021) [in Romanian].

55. Sea buckthorn diseases and pests. Available online: https://sotkiradosti.ru/bolezni-i-vrediteli-rasteniy/bolezni-i-vrediteli-oblepihi (accessed on 25.08.2021) [in Russian].

56. Botanists Encyclopedia. Available online: https://www.botanistii.ro/blog/tratamente-informatii-arbusti-catina/ (accessed on 15.10.2021) [in Romanian].

57. Stalažs, M. Review of sea buckthorn pests in Latvia. In Producing Sea Buckthorn of High Quality. In *Proceedings of the 3rd European Workshop on Sea Buckthorn. EuroWorkS 2014*, Naantali, Finland, October 14-16; Sanna Kauppinen, Ekaterina Petruneva; Publisher: Natural Resources Institute Finland (Luke), Helsinki, 2015

58. Xia, B.; Liang. Y.; Hu, J.; Yan, X.; Yin, L.; Chen, Y.; Hu. J.; Zhang, D.; Wu Y. First Report of Sea buckthorn Stem Wilt Caused by Fusarium sporotrichioides in Gansu, China. *Plant Disease* 2021, 105 (12), 4156.

59. Carson, M. Crown Rust Development and Selection for Virulence in *Puccinia coronata* f. sp *avenae* in an Oat Multiline Cultivar. *Plant Disease* 2009, 93(4), pp. 347-353.

60. Macari, A.; Netreba, N.; Boeştean, O.; Sandu, Iu.; Dianu, I. The physical and organoleptic properties of the fruits of sea buckthorn *Hippophae rhamnoides* L. grown in the Republic of Moldova. *Știința agricolă* 2021, 1, pp. 51-55. [in Romanian].

61. Sandu, Iu.; Macari, A.; Netreba, N.; Boeştean, O.; Sandulachi, E.; Dianu, I. Prospects using sea buckthorn berriws for healthy food production. In Food connects people and shares science in resilient world. In *Book of Abstracts*, Galaţi, România, 7-8 ᵗʰ October 2021; Galaţi University Press, p.69.

62. SM SR ISO 874:2006. *Fresh fruits and vegetables. Sampling (taking samples)*. Aplicate from 01-01-2007. Moldova-Standard, Chisinau, Republic of Moldova, 2006. 13 p. [in Romanian].

63. SM EN ISO 6579-1:2017 *Microbiology of food and feed products. The horizontal method for the detection of bacteria of the genus Salmonella spp* . Aplicate from 28-07-2021. ISO copyrite office, Jeneva, Switzerland, 2017, 50 p. [in English].

64. SM ISO 4831:2010 *Microbiology of food and animal feeding stuffs. Horizontal method for the detection and enumeration of coliforms. Most probable number technique.* Aplicate from 12-07-2010. Institute for Standardization of Moldova, Chisinau, Republic of Moldova, 2010. 21 p. [in Russian].

65. SM EN ISO 4833-1:2014 *Microbiology of the food chain. Horizontal method for the enumeration of microorganisms. Part I.* Aplicate from 01-07-2014. Institute for Standardization of Moldova, Chisinau, Republic of Moldova, 2014. 21 p. [in English].

66. Sandulachi L.; Rubţov S.; Popescu L.; Costiş, V.; Gurmeza, I. Microbiological control of food products. Tehnica, UTM, Chisinau, Republic of Moldova, 2017, 128 p. [in Romanian].

67. SanPiN 2.3.2.1078-01 *Hygienic requirements for the safety and nutritional value of food products*. Aplicate from 01-09-2002. Russia. Saint Petersburg: Rid Group, 2012, 448 p. [in Russian].

68. Household plot. Available online: http://strgid.ru/en/yagody-oblepikhi-kak-vyrastit-oblepikhu-na-priusadebnom-uchastke-svoimi-rukami (accessed on 20.07.2021).

69. Kauppinen, S.; Petruneva, E. Producing Sea Buckthorn of High Quality. In *Proceedings of the 3rd European Workshop on Sea Buckthorn. EuroWorkS 2014*, Naantali, Finland, October 14-16; Sanna Kauppinen, Ekaterina Petruneva; Publisher: Natural Resources Institute Finland (Luke), Helsinki, 2015.

70. Peste.ro. Available online: https://www.pestre.ro/blog/catina-de-la-plantare-la-beneficii/ (accessed on 20.03.2022) [in Romanian].

Publisher's Note: JES stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.

Submission of manuscripts: jes@meridian.utm.md