

INTERNATIONAL SCIENTIFIC JOURNAL

INNOVATIONS

YEAR IX, ISSUE 2/2021, SOFIA
ISSN PRINT 2603-3763
ISSN WEB 2603-3771

Publishers:



SCIENTIFIC TECHNICAL UNION OF MECHANICAL ENGINEERING
"INDUSTRY 4-0"



NATIONAL SCIENTIFIC TECHNICAL SOCIETY
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INTERNATIONAL SCIENTIFIC JOURNAL INNOVATIONS

YEAR IX, ISSUE 2 / 2021

ISSN (PRINT) 2603-3763, ISSN (ONLINE) 2603-3771

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CONTENTS

THEORETICAL PROBLEMS IN INNOVATIONS

An exemplary approach to measure an innovation strategy with a questionnaire-based business survey in SMEs Victoria Wolf, Renata Dobrucka, Robert E. Przekop, Stephan Haubold	43
---	----

INNOVATION POLICY AND INNOVATION MANAGEMENT

Innovation management systems - reality and perspectives Tzvetelin Gueorguiev	48
Financial goals of the European Union – analysis of a target-driven budget concept from the perspective of the innovative SME sector Zsolt Fülöp, Norbert Bajkó	51
Innovations and corporate social responsibility in bulgarian SMEs – status and prospects Yanica Dimitrova	57
Lean concept transformation of the packaging procedure in the process of final processing Marko Jukić, Perica Šimunović, Krsto Mijanović	60

INNOVATIVE SOLUTIONS

Effective determination of clomazone and oxyfluorfen in aqueous samples by differential pulse stripping voltammetry Milan Momčilović, Marjan Randelović, Jelena Milićević, Aleksandar Bojić	63
Innovative hybrid fiber-reinforced shotcrete for thin repairing overlays - long term mechanical and structural characteristics Valeriy Naidenov, Mirona Mironova	66
Application of progressive technologies in the restoration of functional areas of products Janette Brezinová, Ján Viňáš, Miroslav Džupon, Jakub Brezina, Henrich Sailer	69
Energy saving crystalline phosphating Studenekin G., Mazurova D., Grigoryan N., Abrashov A., Vagramyan T.	73
Efficiency of processing of recycle from porous PVC Tomasz Garbacz, Ludmila Dulebova	75
Study of temperature changes in ceramic cavity walls of beehives Lyuben Lakov, N. Stoimenov, Mihaela Aleksandrova, Todorka Lepkova, Gergana Mutafchieva	78
Current state of research in the field of measures regarding the prevention and combat of the freeze-thaw phenomenon of roads from the patent literature Sebastian Petru Boboc	83

An exemplary approach to measure an innovation strategy with a questionnaire-based business survey in SMEs

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Abstract: The expanding globalization causes growing competition, especially in the context of Industry 4.0. One of the most important success factors for companies to managing the growing competition is to generate constant innovation. Companies show a high number and diversity of different business activities and follow different approaches to enable the constant generating of new innovations, but the problem is how to prove if the business activities were helpful to generate innovations in controlled and measurable way to avoid that the innovation activities were only time and resource consuming. The whole process which includes all activities for generating innovation for example the development of a basic idea to a new innovation with a successful business model is defined by the term innovation strategy. Especially for Small and Medium Enterprise (SME) is the measurement of the effectiveness of innovation activities of high importance caused by limited resources. This article presents one possible approach how an innovation strategy can be measurable through the method of a questionnaire-based business survey for SMEs which enables companies to evaluate the effectiveness of an innovation strategy. The result of the article is the description and presentation of possible indicators and their units for measurement as a fundament for a questionnaire for a business survey which helps to identify the degree of an innovation and consequently of an innovation strategy.

Keywords: INNOVATION STRATEGY, MEASUREMENT OF INNOVATION, BUSINESS SURVEY, QUESTIONNAIRE DEVELOPMENT

1. Introduction

Innovation is one of the key factors of the success of a company and is an essential strategic component to survive in a growing competitive market environment. The term innovation is of Latin origin and means renovation or change. In general innovation stands for the three-step process of an idea, invention and diffusion [1]. Therefore, in a business context innovation can be conceptualized as an incidence (idea) for a product (invention) which has not been there before and which results in a high market acceptance (diffusion) [2]. The meanings of the term innovation are of high complexity and therefore result in a large number of existing definitions. According to Haddad and Williams (2019) common and general characteristics of the term innovation exist and is defined as an implementation of change that introduces improvements [3]. Gault (2018) specifies the word implementation stating that innovation is an implementation of a new or significantly improved product. A product can be a good or a service [4]. In summary the mentioned aspects and characteristics of the term innovation lead to the following definition applied in the context of this article: An innovation is an idea which is developed to an invention which creates change in the form of new products/services or product/service improvements, accompanied by high market diffusion.

A strategy is defined as a plan which has some sort of consciously intended course of action and can be regarded as a guideline to deal with a situation [5]. If a company chooses to generate innovation, strategically, it needs a plan according to the given corporate strategy [6]. Innovation strategy is therefore defined as a set of actions fostering all procedures in an organization, including strategic goals and guidelines which have the goal to generate innovations and therefore innovation does not have to be an accident, with a well-defined strategy a company can control and manage the generation of innovation [7-10].

In the Indicators Report on the Innovation Survey 2017 it becomes clear that large companies have a higher innovation success and have more innovation funds available. In contrast, Small and Medium Enterprise (SME), companies with less than 250 employees show less innovation activity [11]. Due to their size, SMEs are confronted with different challenges than large companies. These include lack of financial or human resources. These challenges have to be mastered in order to be able to survive in the competition and to innovate successfully [12]. Therefore, the observation of the efficiency of the activities which are implicated to generate innovations and are bundled up in an innovation strategy

is of high importance for SMEs to avoid expensive but inefficient activities.

The aim of the article is to present a possible approach to measure a specific innovation strategy. This is achieved by describing business surveys and their general approaches and implications as a possible method to measure innovation strategies. After this general introduction in the topic of business survey this article presents the transfer of conceptualization, measuring innovation and operationalization of a business survey for the measurement of the effectiveness of an innovation strategy. The final result of this article presents a possible structure for a questionnaire of a business survey which evaluates and therefore helps to identify the effectiveness of an innovation strategy of SMEs.

2. Prerequisites and means for solving the problem

A business survey which analyzed the efficiency of an innovation strategy represents a powerful tool to control the innovation activities of an organization and to prove their functionality especially for SMEs. But the measurement of an innovation strategy represents a challenge to generate a survey approach which includes all relevant aspects of the concept of innovation because this topic is characterized through a high number of different aspects and dimensions. Concerning a survey which tests a specific innovation strategy, the survey has to include questions which are well-defined to generate a meaningful result. To achieve this, it is of high relevance to define the conceptualization and operationalization of the survey [13].

2.1 Conceptualization and operationalization of a business survey

Before developing a survey and its questions, the most important step is to define the concept that should be analyzed through the survey. This starting point is critical for getting the data the researcher wants. The researcher first decides what concepts are of interest, and secondly, how to define those concepts [14]. Within a theory, the concepts of interest are described in a network of relationships among attributes. Attributes are the building blocks of concepts. An attribute stands for the smallest piece of measurable information that can be identified. A concept is defined by associating a concept to one or more attributes. In the case of a survey, questions in a questionnaire are specified to measure these attributes. Questions are the operationalization of these attributes; they are the operational definitions according to which the attributes are measured. This includes the wording of a question and answering options. [13].

The following figure demonstrate the way of conceptualization and operationalization and how to get from the theory to the concept and their attributes and the further step of developing specific questions which build a questionnaire and which in turn is used for a business survey to collect data.

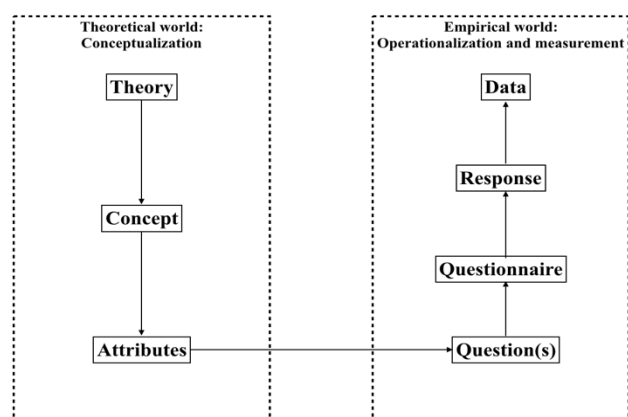


Fig. 1: From theory to data [13]

In Fig. 1 two different methods exist to achieve an approach for conceptualization and operationalization: The top-down and the bottom-up approach. The different between both methods is the starting point and way from theory to data.

2.2 Conceptualization of the measurement of innovations strategies

A business survey which tests the efficiency of an innovation strategy has the underling concept of innovation and the measurement of innovation. For the topic innovation and its measurement there exists a high number of established concepts in the literature [15]. Especially the work of the global community of practitioners in the OECD Working Party of National Experts on Science and Technology Indicators give a well-analyzed overview how to conceptualize and measure innovation in business contexts [16]. Caused by the high variety of concepts of measuring innovation a survey about testing an innovation strategy should be built on the method of top-down from theory to data and with the approach of dimension/attribute analysis. This approach includes the idea that empirical attributes or indicators are specified for the concepts in a theory. The process of concept specification is driven by an existing theory, logical reasoning, or may also be based on results from previous research. To evaluate a strategy, one cannot measure the strategy itself, but one has to analyze the dimensions which are affected through a strategy and to measure the attributes which define the dimension of the strategy through its specific indicators. To demonstrate this logic: If one wants to evaluate the climate it would be necessary to analyze the dimension weather with its attributes temperature and amount of precipitation which are indicated through degree Celsius and milliliter per square meter. It is reasonable to analyze an innovation strategy by measuring the dimension of innovation. The survey for the efficiency of an innovation strategy has to analyze the influence of this strategy on innovation of the organization through the attributes of innovation through its indicators. The following figure displays the different aspects to measure the innovation strategy.

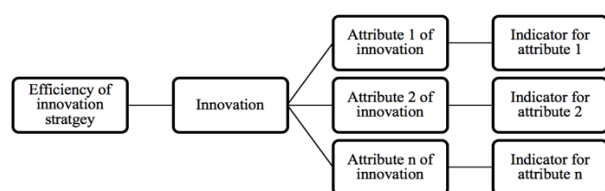


Fig. 2: Aspects of the measurement of the efficiency of an innovation strategy

To summarize the mentioned aspects in the idea of the theory-driven top-down method to develop a survey, the efficiency of an innovation strategy can be measured through the concept of innovation and its attribute which are defined through specific indicators. To define the concept of innovation and its measurement, this article is based on the framework of the Oslo Manual 2018 which is described in detailed in the following chapter.

3. Solution of the examined problem

The current standard work for the measuring of innovation is the Oslo Manual 2018 established by the Organisation for Economic Co-operation and Development (OECD). The Oslo Manual provides guidelines for collecting and interpreting innovation data. It is intended to facilitate international comparability and offers a platform for research and experiments to measure innovation. The guidelines are primarily intended to support national statistical offices and other producers of innovation data in the design, recording and publication of innovation measures in order to meet a range of research needs. In addition, the guidelines are intended to be of direct value to users of information about innovation. These guidelines can be seen as a combination of formal statistical standards, best practice recommendations and suggestions for extending the measurement of innovation. [16].

A survey of an innovation strategy through the measurement of innovation includes the identification of a set of one or more attributes that the innovation is expected to achieve. The measurable attributes can be grouped by areas of influence into the following categories: Markets for the firm's products, Production and delivery, Business organisation and Economy, society or environment. The Oslo Manual listed innovation attributes for measurement through the classification into the four mentioned categories [16].

Table 1: Attributes for measurement of innovation through the classification^[16]

Areas of influence	Markets for the firm's products	Production and delivery	Business organization	Economy, society or environment
Attributes for measuring innovation	Upgrade goods or services	Upgrade outdated process technology or methods	Improve capabilities for absorbing, processing and analysing knowledge	Reduce negative environmental impacts /deliver environmental benefits
	Expand the range of goods or services	Improve quality of goods or services	Improve sharing or transfer of knowledge with other organisations	Improve public health, safety or security
	Create new markets	Improve flexibility for producing goods or services	Improve the efficiency or function of the firm's value chain	Improve social inclusion
	Enter new markets or adapt existing products to new markets	Increase speed of producing goods or delivering services	Improve or develop new relationships with external entities (other firms, universities, etc.)	Improve gender equality
	Increase or maintain market share	Reduce labour costs per unit of output	Increase business resilience and adaptability to change	Improve quality of life or well-being
	Increase the reputation, brand	Reduce material, energy	Improve working conditions,	Comply with mandatory regulations

	awareness, or visibility of goods or services	costs or operating costs per unit of output	health or safety of the firm's personnel	
	Comply with market regulations		Implement a new business model	
	Adopt standards and accreditation	Reduce time to market	Contribute to the development of standards	

In the following chapter the transfer of the attributes of innovation defined by Oslo 2018 to a business survey approach with the intend to analyze the efficiency of a specific innovation strategy is presented.

4. Results and discussion

Following by the process displayed in Fig. 2 a survey which evaluates an innovation strategy can be built through possible indicators and their unit of measurement for each attribute which can be asked through question. In addition to this, every indicator is categorized into a qualitative or a quantitative indicator. The logic behind this categorization is that only an indicator is categorized quantitative if a quantitative measurement or calculation can be made which are only caused comprehensibly by the specific innovation strategy. Non-clear quantifiable indicators are categorized qualitative. Through choosing a qualitative indicator in the case of a no-clear quantifiable indicators one is able to guarantee that no over interpretation of the influence of an innovation strategy is made. One example for this logic of categorization can be seen in the attribute *Increase the reputation, brand awareness, or visibility of goods or services* and its indicator (shown in the next table) *increase of publication, likes on social media, customer request* because the increase of for example likes on social media can hardly lead back to one specific innovation strategy. It cannot be excluded that activities of the marketing or other departments have an influence, too. A qualitative evaluation with an adding literal explanation of the given answer, which is explained in further sections, is more helpful to get meaningful results. Table 2 shows the attributes of innovation of Oslo 2018 and their transferred indicators and units of measurement.

Table 2: Indicators and their units of measurement of the Attributes for measurement of innovation

Area of influence	Attribute	Indicator	Unit of measurement
Markets for the firm's products	Upgrade goods or services	qualitative literal evaluation of improved properties	yes / partially / no
	Expand the range of goods or services	quantitative number of added products or goods	in number of added products or goods
	Create new markets	quantitative number of added markets	in number of added markets
	Enter new markets or adapt existing products to new markets	quantitative number of added markets	in number of new markets
	Increase or maintain market share	quantitative increase of market share	in % of increased market share
	Increase the reputation, brand awareness, or visibility of goods or services	qualitative literal evaluation of increase of publication, likes on social media, customer request	yes / partially / no
	Comply with market	qualitative literal evaluation of	yes / partially / no

Production and delivery	regulations	compliance of regulations	
	Adopt standards and accreditation	qualitative literal evaluation of adaptation of standards	yes / partially / no
	Upgrade outdated process technology or methods	qualitative literal evaluation of improved process properties	yes / partially / no
	Improve quality of goods or services	qualitative literal evaluation of improved goods and services	yes / partially / no
	Improve flexibility for producing goods or services	qualitative literal evaluation of flexibility for goods and service	yes / partially / no
	Increase speed of producing goods or delivering services	quantitative evaluation of time difference of old and new speed of producing goods or delivering services	in % increase of speed
	Reduce labour costs per unit of output	quantitative labour costs difference per unit of output difference	in €
	Reduce material, energy costs or operating costs per unit of output	quantitative material, energy costs or operating costs difference per unit of output difference	in €
Business organisation	Reduce time to market	qualitative evaluation of time of market entrance	yes / partially / no
	Improve capabilities for absorbing, processing and analysing knowledge	qualitative literal evaluation of capabilities for absorbing, processing and analysing knowledge	yes / partially / no
	Improve sharing or transfer of knowledge with other organisations	qualitative literal evaluation of sharing or transfer of knowledge with other organisations	yes / partially / no
	Improve the efficiency or function of the firm's value chain	qualitative literal evaluation of efficiency or function of the firm's value chain	yes / partially / no
	Improve or develop new relationships with external entities (other firms, universities, etc.)	qualitative literal evaluation of new relationships with external entities (other firms, universities, etc.)	yes / partially / no
	Increase business resilience and adaptability to change	qualitative literal evaluation of business resilience and adaptability to change	yes / partially / no
	Improve working conditions, health or safety of the firm's personnel	qualitative literal evaluation of working conditions, health or safety of the firm's personnel	yes / partially / no

	Implement a new business model	qualitative literal evaluation of implement new business model	yes / partially / no
	Contribute to the development of standards	qualitative literal evaluation of contributing to the development of standards	yes / partially / no
Economy, society or environment	Reduce negative environmental impacts /deliver environmental benefits	qualitative literal evaluation of environmental impacts /deliver environmental benefits	yes / partially / no
	Improve public health, safety or security	qualitative literal evaluation of public health, safety or security	yes / partially / no
	Improve social inclusion	qualitative literal evaluation of social inclusion	yes / partially / no
	Improve gender equality	qualitative literal evaluation of gender equality	yes / partially / no
	Improve quality of life or well-being	qualitative literal evaluation of life or well-being	yes / partially / no
	Comply with mandatory regulations	qualitative literal evaluation of compliance with mandatory regulations	yes / partially / no
	Comply with voluntary standards	qualitative literal evaluation of compliance with mandatory regulations	yes / partially / no

4.1 Exemplary questionnaire structure for the measurement of an innovation strategy

To follow the logic of Figure 2 and the aspects of the measurement of the efficiency of an innovation strategy, every attribute, defined by the Oslo 2018 has its indicator and a specific unit of measurement like shown in Table 2. Like displayed in Figure 1 out of the attributes, questions can be constructed which build together a questionnaire for a business survey.

4.1.1 General construction of a question to measure an innovation strategy

To construct a business survey for the measurement of the efficiency of an innovation strategy a question can be built in general like the following:

Question: Did the “specific name of the innovation strategy” contribute to “attribute”?

Please evaluate. Answer option:

- If qualitative indicator: yes / partially / no
- If quantitative indicator: Adding unit of measurement

The following example represents how a question for a qualitative indicator can be built as provided above:

(1) Did the cooperation-orientated innovation strategy contribute to upgrade goods or services? Please evaluate. Yes / Partially / No
A question for a quantitative attribute can be answered through adding a concrete quantitative unit of measurement.

The following examples represent how a question for a quantitative indicator can be built as provided above:

(2) Did the cooperation-orientated innovation strategy contribute to expand the range of goods or services? Please evaluate. Yes / Partially / No

Please mark the number of expanded goods or services.

0 good or service / 1 good or service / 2-3 goods or services / + 3 goods or services

4.1.2 Description of construction of questions

Like shown, a question is built through the combination of an attribute of innovation and if its qualitative evaluation (yes / partially / no) and if its quantitative by adding a quantitative unit of measurement which fits the attribute. In general, the formulation of “Did (specific name of the innovation strategy) contribute to (attribute)?” asks in an appropriate degree for the efficiency of an innovation strategy caused by the degree of the influence of a specific innovation strategy. As described an innovation strategy is the scope of actions for all innovative procedures and therefore stands for the frame in which innovation activities are happen. A stricter formulation of the question about the efficiency of an innovation strategy like “Did (specific name of the innovation strategy) to (attribute)?” would stand for a too big influence of a specific innovation strategy and is therefore not suitable for a questionnaire which asks for the efficiency of a specific innovation strategy. In the case of a quantitative attributes the displayed question only shows an example how the unit of measurement in Table 2 can be transferred. The scope of the answer option has to be adjusted to the general scope of the business survey and its interrogated innovation strategy. To expand the expressiveness of a question and to understand the background of a giving answer it is helpful to add to the displayed closed question, open questions like: “Please explain your evaluation in up to three sentences.” Through adding an open question which asks for the explanation of a giving evaluation generates more specific background information and can be analyzed through a tag cloud method, a visual analysis of text data [17].

4.2 Practical implications

The described exemplary questionnaire structure for the measurement of an innovation strategy which was reviewed and verified for applicability through the review of 6 renown experts of academia and industry. The proposed business survey approach therefore represents one possible method how the complex project of measuring an innovation strategy can be managed in a solid and, through the utilization of Oslo 2018, well-founded approach. The presented structure is currently used by the authors of this article to measure 12 different cases in SMEs of a startup-orientated cooperative innovation strategy and has proven its functionality concerning the measurement of the efficiency of a specific innovation strategy up until now. The overall results are going to be published after finishing the business survey in 2021.

5. Conclusion

An objective verification of all activities concerning generating innovations which are bundled up in an innovation strategy is important especially for SMEs to avoid expensive but inefficient investments to generate innovation. One possible method to measure a specific innovation stagey is the business survey. This article describes the general approach of a business survey and how the conceptualization of a business survey can be managed concerning the measurement of the efficiency of an innovation strategy. By transferring the theory of conceptualization of a business survey through the existing framework of Oslo 2018 this article presents concrete attributes, indicators and units of measurement for an innovation strategy. As a final result an exemplary method, how a question can be construct out of the concrete attributes, indicators and units of measurement, is presented. This method stands for a practical approach how a questionnaire for a business surveys which has the goal to measure the efficiency of an innovation strategy can be build. The practical implication of the method is currently used for testing the efficiency of a startup-orientated cooperative innovation strategy and will generate results which will be published in the future.

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Innovation management systems - reality and perspectives

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Abstract: Innovations require professional knowledge augmented by personal skills, creativity being the most frequently cited. It is a common misconception that standards limit creativity. The recent trends in management system standards prove that they can be used as best practices in order to improve organizational performance in various domains. The seven parts of CEN/TS 16555 are the precursor of the ISO 56000 series of standards for innovation management systems. The article provides a chronological review of these standards, their relation to the United Nations' sustainable development goals (SDGs) and the way they can provide benefits for the organizations.

Keywords: INNOVATION MANAGEMENT SYSTEMS, ISO 56002, ISO 56000 SERIES, UN SDG, INTELLECTUAL PROPERTY, TECHNOLOGY TRANSFER.

1. Introduction

'Eureka!' is the popular exclamation used to express the joy from making a discovery or an innovation. Scientists and researchers do not rely on chance and use their knowledge and experience to explore unique phenomena. Furthermore, the scientists of today have to consider global challenges, technological developments and ethical issues in their research. In fact, innovation is not just a buzzword. It should be characterized by the value it brings to society.

By the definition given in ISO 9000, innovation is 'new or changed object realizing or redistributing value' [1]. This definition already makes a clear distinction between innovation and improvement, the latter one being an 'activity to enhance performance'. In other words, improvements enhance the effectiveness of processes, whereas innovations also enhance their efficiency. Generally, innovations are expected to bring significant effect, to have high return on investment (ROI) and add value.

In order to achieve this, the activities resulting in innovation need to be managed. As is the case with the vast majority of ISO management system standards, this is done by implementing the Plan – Do – Check – Act (PDCA) cycle [2].

The popularity of the PDCA cycle to a large extent is as a result from its penetration in all ISO 9001 quality management systems [3]. According to the latest ISO Survey [4] conducted in September 2020 more than 1,2 million organizations have successfully developed, implemented and maintain quality management systems conforming to ISO 9001:2015. This undisputed leader is followed by ISO 14001 with 312580 valid certificates for 487950 sites [5]. Third in this ranking is ISO/IEC 27001 for information management systems [6], followed closely by ISO 45001 for occupational health and safety management systems [7]. This fourth place is partly due to the fact that this standard is published only recently and practically is the ISO answer to the need to adopt BS OHSAS 18001 as an internationally recognized best practice.

The quality professionals from around the world have made numerous efforts to introduce 'innovation management' as a core discipline in quality management. Fig. 1 presents a graph showing the results of a query, performed on the website of the 'Quality Progress' Magazine of the American Society for Quality. It can be said that innovation management was a significant topic right from the first version of ISO 9001, published back in 1987.

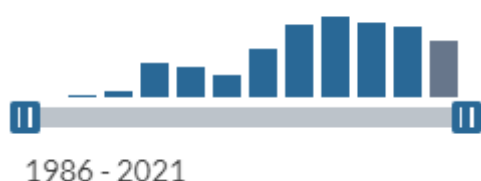


Fig. 1 Innovation management articles published in the ASQ Quality Progress Magazine (1986 – 2021).

The spikes on the graph actually represent a reaction of the innovation community to the new edition of ISO 9001 in that specific time period.

2013 is the year when the European Committee for Standardization (CEN), and more specifically- its Technical Committee CEN/TC 389 has published its technical specifications in the domain of innovation management. By 2015, the CEN/TS 16555 series included 7 standards ranging from requirements for an innovation management system, through creativity management and innovation thinking, to more specific standards for innovation management assessment and intellectual property management [8].

This context has set the stage for the International Organization for Standardization (ISO) to develop a standard, or rather, a series of standards dealing with innovation management.

2. Discussion

2.1 Relationship between innovation management and quality management system standards

When the ISO 9000 series are discussed, usually this set of standards includes:

- ISO 9000:2015,
- ISO 9001:2015,
- ISO 9002:2016, and
- ISO 9004:2018.

Below is a list which is customized for the topic of this paper.

ISO 9000:2015 provides the definition for the term 'innovation'. In its clause presenting the quality management principles, 'innovation' is specifically addressed in the key benefits from implementing improvements.

As a consequence, Clause 10 'Improvement', more precisely the note to Clause 10.1, and potentially Clause 10.3 'Continual improvement' suggest that innovations are one of the possibilities to implement improvements in organizations.

ISO 9002:2016 [9] has identical structure to ISO 9001 and provides more detailed guidelines on how to apply the requirements of ISO 9001. Here also, Clauses 10.1 and 10.3 deal with innovations.

ISO 9004:2018 [10] stands out from the abovementioned standards. With its guidance for achieving sustained success by organizations ISO 9004 goes beyond the concept of conformity to requirements. It can serve as a roadmap and a basis for defining action plans which will improve not only the effectiveness, but also the efficiency of the quality management system. The structure of this standard differs from the clauses of ISO 9001 and ISO 9002.

Innovation has a specially devoted clause (11.4) covering general guidelines, application, timing and risk. Clause 11.4.1 points the

attention to the fact that changes in the context of the organization, be it internal or external, as well as changes in the needs, expectations and requirements of interested parties may be causes provoking innovation initiatives. In addition to promoting innovative thinking and providing the resources for supporting innovation initiatives, the organization is advised to establish and maintain processes for innovation management. Thus, ISO 9004 can be seen as the precursor of the ISO 56000 series of standards.

Clause 11.4.2 deals with the implementation of innovations at all levels of the organization. This would require changes in processes, products and services, in the technologies applied by the organization, in the organization's structure, business model and its management system. The latter can be integrated with the ISO 56000 series as well.

The timing of implementing innovations by following specific action plans is presented in Clause 11.4.3. The implementation of innovations should take into consideration relevant opportunities and risks, time horizons, and the way in which innovations support its strategic direction.

In addition to the widely used evaluation methods such as reviews, audits and monitoring of Key Performance Indicators (KPIs), ISO 9004 devotes more than half of its page volume to a self-assessment tool. For each of the clauses, this tool specifies maturity criteria divided into 5 levels. The lowest level- Level 1, signifies chaotic and ad hoc reactions, Level 2- some management of the process but lack of a system, Level 3- evidence of a well-maintained quality management system. The more advanced levels- Level 4 and Level 5, can serve as opportunities for improvement of 'ordinary' organizations. At maturity level 4 the organizations can be considered as industry leaders in the specified criteria. Maturity level 5 denotes world class performance. These criteria can be modified to better suit the specific context of the organization. One should be mindful that the maturity levels defined in ISO 9004 also evolve. For example, the high levels in the previous edition of the standard- ISO 9004:2009, are now considered as 'basic/musts' or 'performance/wants' if we make an analogy with Noriaki Kano's model for product development and customer satisfaction. The 'attractive/exciters' characteristics are where an innovative organization should aim at. For example, Maturity Level 5 for innovations can be described by the following:

- Innovations take into consideration possible changes in the organization's context;
- Preventive action plans are in place in order to mitigate the risks that generally accompany innovation initiatives;
- Innovations are applied at all levels of the organization.

2.2 Innovation management system and supporting standards

The foundations for innovation management, laid out in the ISO 9000 series and in the CEN/TS 16555 series of standards have produced a sequence of initiatives undertaken by Technical Committee ISO/TC 279 'Innovation management'. In 2019, three innovation management standards have been published:

- The guidance standard for innovation management systems- ISO 56002 [11];
- The standard with tools and methods for innovation partnership- ISO 56003 [12];
- And the standard with guidance on how to assess innovation management- ISO 56004 [13].

The approach of ISO/TC 279 is notably different from the established practice in most management system standards. For example, in the case of quality management systems, the requirements standard- ISO 9001 is published, and then it is followed by ISO 9002 with the guidance on how to apply the requirements. The position of ISO/TC 279 on this issue can be summarized by an analogy with a best practice in our daily lives: 'monitor the route people take, and then pave the path'. The proof of this fact is that the standard with

requirements for an innovation management system- ISO 56001 is still at a very early stage of its development by the end of 2020 [14].

In 2020, two more standards have been published:

- The fundamentals, principles and vocabulary in ISO 56000 [15], and
- A key standard with guidance for the tools and methods for managing intellectual property- ISO 56005 [16].

This document can serve as a best practice for all organizations dealing with intellectual property (IP)- patent offices, research centres, technology transfer offices, and other interested parties. The ISO 56000 series considers intellectual property and an integral part of innovation management. The strategy, policy, objectives and processes for intellectual property management are integrated with the strategic innovation efforts and with the overall management system of the organization.

The University of Ruse 'Angel Kanchev' has developed its strategy, policy, objectives and processes for innovation management and IP management. The Technology Transfer and Intellectual Property Centre (TTIPC) is in the process of implementing its internal innovation management system and be prepared for certifying this system when the requirements standard (ISO 56001) is published. In 2020, the TTIPC has published its first innovation portfolio which contains the valid IP products and focusses on the new applications and registrations of patents, utility models, designs and trademarks. The innovation portfolio is being used as a tool to raise awareness amongst all relevant interested parties, both internally and externally. This portfolio is based on a database of intellectual products which is maintained and updated regularly by the TTIPC. The information is cloud-based and can be accessed anytime, anywhere by authorized staff.

Innovation management is intrinsically involved with change management. The dynamic world we live in is characterized by volatility, uncertainty, complexity and ambiguity (VUCA). The abbreviation VUCA stands for the rapid change of the organization's context, the many unknowns it needs to consider in its operations, the complicated interplay of various influencing factors and the many possible interpretations of facts. ISO 56006 is meant to help organizations achieve their business objectives by navigating the VUCA environment [17]. This guidance for tools and methods for strategic intelligence management can help decision-makers with meticulously collected, summarized and presented information, converted into specific knowledge about the innovation trends. ISO 56003 is in the last step before becoming a Final Draft International Standard (FDIS) and the innovation professionals are really looking forward to its official publication.

The international community eagerly expects in 2021 the development and/or publication of the following innovation management standards:

- ISO 56007- tools and methods for idea management [18],
- ISO 56008- guidance for tools and methods for innovation operation measurements [19], and
- ISO 56010 with illustrative examples of ISO 56000 [20].

As can be seen from this exhaustive list of standards, innovation management systems are multi-faceted. In order to reap the full benefits of the ISO 56000 series all the documents and best practices listed above should be implemented as a coherent system.

The advanced level of these standards is confirmed by the European Committee for Standardization (CEN) which has already adopted ISO 56000, ISO 56002, ISO 56003, ISO/TR 56004 as European Norms (EN) replacing and/or adding to its CEN/TS 16555 series.

2.3 Innovation management and sustainable development

The United Nations has published its 2030 Agenda with

Sustainable Development Goals. ISO supports this effort with aligning its standards to the UN SDGs [21]. ISO contributes to all of the SDGs. Fig. 2 shows the number of ISO standards that are directly applicable to each Goal.

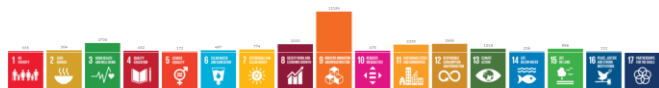


Fig. 2 ISO standards and UN SDGs.

The highest column on the chart represents the 12194 standards related to innovation management, industry and infrastructure.

Fig. 3 presents a matrix showing the relationship between the ISO 56000 series of standards and the UN SDGs.

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
ISO 56000																	
ISO 56001	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
ISO 56002				✓													
ISO 56003				✓													
ISO/TR 56004				✓													
ISO 56005				✓													
ISO 56006				✓													
ISO 56007				✓													
ISO 56008				✓													
ISO/TS 56010																	

Fig. 3 The ISO 56000 series of standards and UN Sustainable Development Goals (SDGs).

It is clear that 3 of the SDGs are supported by most of the innovation management standards. These SDGs are:

- Goal 4: Quality Education;
- Goal 8: Decent work and economic growth;
- Goal 9: Industry, innovation and infrastructure.

The target for SDG 9 is to 'enhance scientific research, upgrade the technological capabilities of industrial sectors in all countries, in particular developing countries, including, by 2030, encouraging innovation and substantially increasing the number of research and development workers per 1 million people and public and private research and development spending'. The achievement of this target is supported by two specific indicators:

- Research and development expenditure as a proportion of GDP, and
- Researchers (in full-time equivalent) per million inhabitants.

The 2020 UN Report on the SDG summarizes the trend in these indicators as 'Despite progress in recent years, investments in research and development need to accelerate, in part to cope with COVID-19'.

This finding is supported by the publication of strategic documents such as The Oslo Manual [22], policy and initiatives by the European Commission [23], 'ISO and Innovation' [24], etc.

3. Conclusion

In line with the Strategy for Higher Education in Bulgaria [25], the University of Ruse is taking active steps towards its implementation. One of the initiatives of the Technology Transfer and Intellectual Property Centre is to develop and implement an innovation management system. A core module in this system in the intellectual property management with its main purpose to create value for the university, its research community, partners and other interested parties.

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Financial goals of the European Union – analysis of a target-driven budget concept from the perspective of the innovative SME sector

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Abstract: The budget of the European Union is a cornerstone factor in the financial existence of the continent, and a highly important factor in the global economy. Due to both the global Coronavirus pandemic, and the new formations of global trade in the Asia region, the entire world is focusing even more on the way the European Union will finalise its directives for investment for the next programme period between 2021 and 2027. Any impact on the budget will reflect upon programmes supporting Member State innovation.

With the aid of five experts on the field of the European Union, economics, financial culture of Europe, and other relevant topics, a round-table focus group was formed online to obtain a qualitative insight into the changes in the perspective of the Union towards 2021. The main idea of the authors is to wrap a larger research around parametrising the measurement of innovation in the Union, and making a simplified system for Hungarian SMEs for ease of understanding, and more accessible tender application process. Changes in the finer directives of the Union are necessary, to serve as a basis for selection among the several dozens of criteria the EU employs.

Keywords: INNOVATION, INNOVATIVENESS MEASUREMENT, HUNGARY, SME SECTOR, EUROPEAN UNION, BUDGET, SUBSIDY,

1. Introduction

One of the most significant problems of the current European Union is the allocation of the budget, and the interests by which said allocation needs to happen. The European Union is a community strongly committed to advanced policies, such as sustainability, circular economy, innovation, responsible research, digitisation, and other such modern perspectives aimed towards improving quality of life through awareness and technological advancement. This perspective mainly comes to life throughout a low-carbon economy, and the rationalisation of both the inner workings of the EU itself, and its systems, by which it happens. [1] Such an example is the digitisation affecting another cornerstone of the EU – the small- and medium enterprise (SME) sector, which is in the focus of the European Union's current budget, inherited from the previous programme "Horizon2020". [2] Said SME sector is an important cornerstone of the European Union, because it has a significant majority in the employment of the European Union in terms of EU citizens employed, all while having a less pronounced role in generating the total enterprise sector's revenue within the European Union, according to the European Union's statistical authority EUROSTAT. [3] The importance of the sector is not only pronounced in the shares among statistical data, but the fact that the sector is responsible for the local-level value chains in the European Union, by which it became a strong focus in budget planning in the European Union to begin with.

Member State governments are required to organize the subsidy system of their own countries along the guidelines of the European Union's policy. These local subsidy systems are very important for the small- and medium enterprises in not only underdeveloped regions, but literally everywhere. The most notable reason for this is that small- and medium enterprises find it harder to obtain capital for growth and sustaining operations, unlike large enterprises, which have much less trouble with it, making it imperative for the European Union to offer opportunities for financing. [4] This is another important factor in the way the SME sector works within the European Union. In order to facilitate smoother operations of the subsidy system, the European Union has strict guidelines for tenders, and how they are to be conducted in the Member States, to increase efficiency of the European Union funds as much as possible.

This, however, often gives trouble to the actors of the local sector, since the high-level policies and factor groups make it an issue to conform to requirements, not to mention, the European Union's policies also have significant impact on the budget, and vice-versa. Therefore, authors decided that they will create a system, which simplifies the expectations the European Union holds towards the small- and medium enterprise sector in Hungary, with clear focus on innovative SMEs, most notably dealing in sustainability, responsible research and innovation. The current

paper's contents are a part of authors' data, which has to do with the European Union's budget, and the analysis for the future, ravaged by the Coronavirus pandemic.

2. Discussion

The goal of authors is to understand how the European Union will change its perspective due to the enormous restoration fund after the Coronavirus global pandemic dealt a serious blow to the Member States, and if a greater shift could have happened due to it in European Union policy towards the future cooperation of Member States. For this goal, it's important to understand the European Union's budget considerations, and how they seem from Hungary.

2.1. State of the budget, future concerns

The European Union's budget is a shared budget managed by the committee of Member States, focusing heavily on financial and organisational directives. As part of the cooperation of Member States, they all invest 1% of their Gross Domestic Product into the operation of the European Union. However, there are several issues, most notably that the European Union's costs planning methodology is not modern enough. This causes several issues in not only planning, but execution. Furthermore, due to a notable event in the European Union, the United Kingdom's discontinuation of participation, popularly dubbed BREXIT, the budget suffered a 94 billion Euro cut as well. [5] In order to accept a new financial programme, which is to sustain the European Union's operations for several years in advance, a consensus among all Member States is mandatory – this leads to the issues with the pre-BREXIT and Coronavirus budget enlarge even more, which necessitates the radical restructuring of the budget. It is questionable, however, if the policies of the European Union for the next programme period of 2021-2027 have to be significantly altered due to this issue as well. Yet, there are anomalies compared to earlier expectations, for example, the expenditures for border security increasing compared to before. [6]

As the European Union is not a homogeneous structure, concluding these questions is impossible, unless the inner workings of the community are understood properly, as facilitating change without the proper knowledge on the structure may cause internal errors that make external issues more severe as well.

One thing of note is that the developed economies within the European Union have a large government sector, which means that the role of their sector is even more pronounced. [5] This constitutes a strong focus on specific sectors in Member States (examples are healthcare, social insurance, security and such other sectors) within the bounds of national economy, and its influence. This system is fundamentally different to that of the United States of America for

example, where the strong influence towards important sectors comes from the federal level. [7]

Similarly, a concern about the European Union's budget comes from the vastly different economic conditions of the Member States. While there are several Member States that have very different economic maturity, constructing a budget for an average is not an option, especially as side by side with post-Socialist countries that have less mature economic systems, three Member States of the European Union are individually on the list of the World's ten most advanced countries (Germany, France and Italy). [8] There's an increasingly looming necessity to improve the state of the current European Union policy, because in the event of them continuing as is, no European Union Member State will remain in the aforementioned "ten most developed countries of the World" group by 2050. [9] The most pressing issues in the development problems of the European Union are as follows:

- the reduction in active civilian population. As Europe has a high number of developed societies, the age tree is getting increasingly narrow on the base, while aging society syndrome is more of an issue each year.
- the changes in the structure of production chains. These are specific to each Member State, and are affected by a multitude of external effects, such as VAT and customs, and trade between larger economic bodies, like the United States.

Due to such issues, however, it is important to note that opportunities also arise. The European Union has the chance to take its first steps towards a federal economic union state, which could not only conserve its advantage in global competition and market position, but even propel it forwards in the race against the technological development level of the United States of America, or the mass production capabilities of China. These are important factors which have to be noted and integrated into the European Union's policies. [10]

2.2. EU priorities for 2021-2027

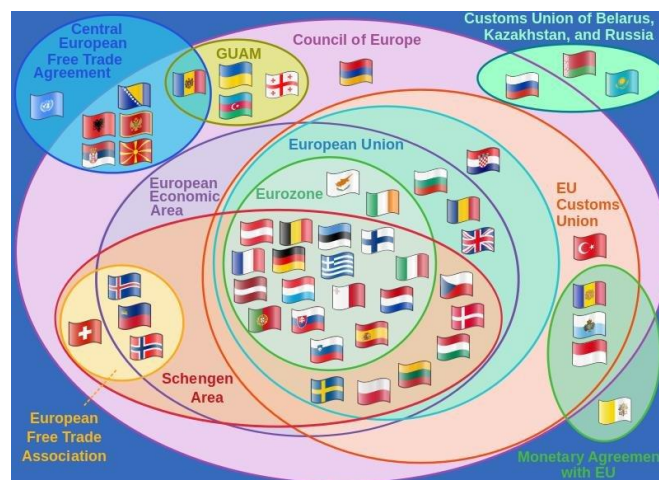
The main source of the European Union's budget comes from two different sources:

- The Gross Domestic Product of Member States. This is an important factor in a different meaning as well, as it determines not only the performance of the European Union itself, but the performances of individual Member States in many of the European Union's calculations. However, note that the criteria towards the Member States are not simplified to GDP for Member States, as the European Union goals necessitate a more complex calculation and responsibility methodology.
- The taxation the European Union employs. This taxation has various effects towards the market actors, partners and business stakeholders the European Union has connections to, but not all facets of taxation are relevant to the current topic. [11]

European Union Member States have been holding increasingly fiery debate on the joint budget recently. [12] There are a multitude of reasons, and even more effects of separate arguments down to the smallest detail, but the main takeaway of the debate is that Member States have specific issues in their own sectors. It's important to note that while Member States see an issue differently, depending on what facet of said issue is the most taxing for said Member State, and how that facet interacts with the joint goals, the most important requirement for gaining subsidy for the processes relevant to the European Union's integration effort is to have a joint incentive regarding Member State professional policy. This 'harmonisation' is grounds for many a debate, because while the European Union has, as mentioned before, vastly differing Member States in terms of financial strength and development level, the joint policy will have an effect on each of them nonetheless – with regards to said

effect having more or less discomfort, depending on who the Member State in question is. Naturally, this results in the obvious conclusion of translating the Member States' unique conditions into different perspectives, and in the meantime, said perspectives developing into local differences between them, further spiralling into inequalities, serving as the basis for further conflict. [13]

Yet another source of differences is that the European Union's Member States have different affiliations regarding foreign trade and politics as well. An Euler diagram of the European Union's Member States can be seen below [14]:



1. Figure: Euler diagram of European Union Member States' foreign relations. [14]

In the event the Member States have goals dependent on common values, these need to serve as the basis for a joint budget. These common values have been under scrutiny for a while now, as the European Union struggles to find common ground among its Member States in contested questions, which further intensified with the advent of the Coronavirus pandemic. Regardless, the European Union's first and foremost goal is to harmonise its Member States, which necessitates the presence and integral importance of joint initiative and common goals.

The most notable point of the European Union's stability is the economic aspect – the European Union therefore has a strong grip on its monetary policy. Deciding the European Union's budget is an integral step in developing the Member States' future prospects, and is handled with great care, debated by the entirety of the economic area. However, further aspects of the economic and financial stability are within the bounds of Member State privilege. The most simple and obvious reason to this is that the European Union's overall size is far too great to allow it to serve as a macro-economic stabiliser, as such, supportive tasks and functions have to be delegated to the specific Member States in order to achieve an overall stability of the system. [15]

There are several requirements to achieving a stable operational environment for the European Union, as an organisation housing several Member States with different details to them:

- Strengthening the European Union's economic influence within the Member States. In order to better enhance the consistency of the common economic area, the European Union, as a higher management organisation has to have a detailed coverage of Member State economic information and a means to affect policies and operations – with consideration to national sovereignty – within the individual Member States.
- Strengthening the European Union's political influence within the Member States. There are a multitude of reasons and explanations as to why this is important, which we will not detail in this study. However, as political strife took hold in the European Parliament, we could see in recent years, how there are several issues, rooted deeply in the

European Union's political operations, that necessitate a stronger cooperation between the various Member States.

- Strengthening the European Union's operational effect influencing the inner workings of the Member States. This basically refers to the European Union's policies having a harmonising effect on the various national policies not covered by the previous two points.

All are necessary to reaching the point where the lability caused by the different attributes of the Member States are brought into equilibrium, and stabilised. [16] Currently, the method for determining the investment of Member States into the European Union's budget is by employing the GNI (Gross National Income) indicator. Gross National Income is calculated as follows: the Member State's entire population's output (for both within and across country borders) are summarized, which includes the income produced by the people of the country living and working in foreign countries (as a type of factor income), are summarized, then reduced by the amount of total income produced within the borders of the country by the residents of other countries. [17] With this system, the incomes also include about 80% of tolls and sugar levies. A further twenty percent coming from these can be held by the Member States, to cover tax collection costs. [5] The European Union has some further income, mostly from sanctions and other fines, and has several smaller incomes. Altogether, the complete budget of the European Union is roughly 1% of the total GNI of Member States, whereas the Member States redistribute about 35-65 percent of their national budgets. [15] However, this system, in its currently introduced form and range is no longer perfectly in effect: apart from the issues with the conditionality and the cohesion policy of the European Union causing strife across the various political topics between Member States, a much larger conclusion came from Euroscepticism and differences in opinion: as of 31. January, 2020, the United Kingdom is the first Member State to officially leave the bonds of the European Union, and as of 31. December, 2020, also left the European Customs Union, and the European Single Market as a member and participant. [17] Although the Brexit process made a dent on the European Union's financial strength, and brought with it a significant change in the fundamental ideas of the European Union itself, it is also an exceptional opportunity. Looking at the issues touched upon earlier, the European Union has a large amount of issues, many of which come from its internal management, which have to be corrected, or otherwise negated. Such is the system principle of individual allowances – the European Union makes exceptions for a multitude of Member States, for a variety of reasons, some of which are more detrimental for the common goals of the Member States than advantageous. It is not a priority to do away with such a system, or at least, there are no suggestions that it would be, however, several key points in the 2021-2027 financial budget, and its additional interventions suggest that at least some individual agreements need to be revised in order to maintain the budget allocation.

During the 2013-2020 Horizon2020 Programme Period, the European Union's incomes that notably come from the payments of the Member States based on the GNI data suffered a violent lapse in ratios. As this is an issue that affects not only the European Union, but its individual Member States, and has a huge impact on the joint goals of the European Union itself, this event caused the financial subsidy autonomy of the Member States to drastically reduce. A consequence is that we simply can't mention individual, self-propelled economising because of this phenomenon. This is the result of the added value-based, and GNI-based payments of the Member States cover more than 85% of the European Union's income side. [15] This further constricts the options of the European Union in allocating its own budget more efficiently.

The European Union's minor shift in policy, however, will likely offer a chance at more subsidy and better opportunities for the SMEs of innovative nature. The SME sector being a highlight in national economy makes it just as important as a factor in Member States' priorities. "Micro-, small and medium-sized enterprises are

key players of national economies, and the successful operation, performance and development of these enterprises play an important role in developed and underdeveloped regions of the world." [18] Due to the integration policy gaining more ground, those better equipped for the requirements of the European Union's system of subsidizing innovative SME activity, and supporting various fields of research will prove more valuable due to the abovementioned reasons.

In the following chapter, the round table conference between authors and five recruited experts will describe the reasons for such opportunities in detail.

2.3. Research sample

The financial framework of the European Union is exceedingly important for the various small- and medium enterprises exactly because a lot of funding comes from the European Union. Whenever the European Union has a shift in policy that affects either the budget, or the small- and medium enterprise sector, the enterprises in question should aim to follow up on the shift in policy, and aim to satisfy new or changed requirements to secure more funding from the European Union's budget. [19] The authors therefore contacted five experts of various fields related to small- and medium enterprises, the European Union, and the Hungarian SME sector, to obtain their opinions and insight as to what changes should be expected due to the ongoing shift in policy, and what the small- and medium enterprises in question should prepare for, how to consider their opportunities for the future, and where to focus their development in order to gain access to better funding.

The five experts who cooperated with authors came from various backgrounds across the enterprise sector and the public sector, with the following expertise:

- The first expert came from a financial background – has worked as a finance auditor, and a financial consultant for 13 years with various small- and medium enterprises in the service sector. Has experience in financial project management, and has had a diverse relationship with small- and medium enterprises from across the European Union. Trained and competent in the details of the Horizon2020 programme period's requirements towards the small- and medium enterprises of the European Union, and has both experience and qualification certificate in tender writing. Currently employed as a consultant by a medium enterprise that consistently advertises itself as a partner for the European Union's 2021-2027 framework programme on the construction sector.
- The second expert is a political scientist and economist with over 30 years of background in financial policy. One of the earliest experts in Hungary to have been admitted into the European Union's expert programme, knowledgeable in financial and SME policy management according to European standards. Had several medium-, and some large enterprises that she completed analyses for as an outside expert, but never worked for a large enterprise as anything but a contractual business partner. Has a wide berth of knowledge about European Union financial goals, and an in-depth knowledge of both the budget and the political mechanisms of the European Commission, the European Parliament and the related institutions concerning finance and policy.
- The third expert is a consultant for the Hungarian financial authorities, as an expert of the European Union. Also taught at university-level, courses include European Union sciences, European Union policy and European Union framework studies. Published several articles about the European Union's Horizon2020 programme period, and as the chief executive officer of a micro-enterprise, also has experience in tenders funded by the European Union. Spent several months in Brussels, conducting analyses on

the financial and political climate of the European Union's main institutions.

- The fourth expert is a professional tender writer, skilled in mainly the agricultural enterprise sector's tenders. As a professional, he has written more than fifty tender applications during the Horizon2020 programme period, and has a deep understanding about the European Union's requirements towards the small- and medium enterprise sector's agricultural line. Has not only good connections regarding the scene of professional tender writing in Hungary, but also in Europe.
- The fifth expert is a finance expert mainly dealing with financial culture and financial awareness in the European Union. Had several instances of researching the effects of European Union policy affecting financial awareness and financial culture trends in the European Union. Also an expert of various topics related to small- and medium enterprises, like financial culture within an organization, enterprise finance awareness and leadership and management financial awareness.

With the aid of the five experts, a round table conference was set up online during early 2021 that constructed a possible scenario in light of COVID-19 and the general outline of the shifts in the EU budget. The main focus was on the budget itself, however, detailed discussion was made into the changes relevant for the small- and medium enterprise sector, and especially the micro-enterprises that were the most impacted by COVID-19 across Europe.

3. Results

The conclusions drawn by the round table conference served as the basis for the future scenario. These conclusions were based on the experts' own ideas, experiences, and the end results of debate on recent trends and events within the European Union, Europe, and the World. As the online round table conference took more than three hours, only parts of the conclusions that have the most significant relation to the current topic will be listed out.

The main questions of the round table were:

Will the European Union's future hold a significant shift in cohesion policy?

Will the European Union redesign its budget due to recent events affecting the European Economic Zone?

Will the European Union implement one of the White Book's solution scenarios in its entirety?

Have recent events changed anything for the small- and medium enterprises within the European Union?

Should there be any expectations towards the European Union's budget significantly shifting priorities in funding goals?

- The European Union will keep a strong focus on the small- and medium enterprise sector even in the future. There is no specific concept which supports the trending towards large- and multi-national enterprises as far as the European Union's subsidiary focus goes. This is a very important facet of information for the innovative enterprises, and especially the small- and medium enterprises, as they're the main beneficiary of the European Union's responsible research and innovation, and sustainability policies. [20] The European Union has a cooperative relationship with the larger and multi-national enterprises, however, the European directive of subsidiarity, local governance and de-centralised economy fit better with the sector of smaller enterprises.
- The innovation policy of the European Union, and the requirement towards innovation, sustainability and responsibility will not shift. Climate goals will not be removed, reduced, or counted differently. As an integral focus of the European Union, these core concepts will strongly adhere to earlier cooperative agreements. The

recent steps taken by the United States of America have been a detrimental impact on the European Union's future vision, however, these are expected to change with the advent of the Biden-administration. The climate goals of the United States' newest financial construct designed to enrich the economy will most likely have an advantageous effect on the European Union's related aims as well. Small- and medium enterprises should expect to be in the focus with cleantech and low-carbon innovation especially, where the European Union can be expected to gradually increase the intensity of climate goals, attested to the current international political climate, and the importance of climate defence.

- Due to the consistency of the European Union's policy regarding the small- and medium enterprise sector, there will most likely be no significant changes in the fundamental policies related to funding. Small- and medium enterprises will be required to follow the earlier tender requirements, and there might be some minor simplifications in terms of application and reporting – at the same time, expected to bring a more strict supervision, mainly due to the current aims of the European Committee – but the most basic parts of the system should not change much, if at all. With this, the only shifts which may affect the small- and medium enterprises of Member States regarding funding will be the previously introduced scenarios concerning the European Union's future.
- The White Book's proposed solutions, brought to a wide social cooperative, namely: keeping the status quo (scenario 1); gradual shrinking of integration to the internal market (scenario 2); multi-velocity Europe concept (scenario 3); area-specific cooperation between Member States (scenario 4); and federal Europe, with deepening integration and unified internal market (scenario 5); will be realised through a fusion of all, or most concepts, to a greater or lesser degree. All the scenarios are expected to be too unrealistic at this point in time, especially because of the COVID-19 pandemic and its specific conclusions for the European Union. Experts highlighted that due to said pandemic and its conclusions, the European Union combined loans and grants of about 5.5% of EU27 2019 GDP to support member states' recovery policies, further distancing from the scenarios. [21] The experts had a heated debate on the final conclusion, however, in the end, four of them supported the claim of the fusion of all scenarios to a certain extent (likely to which the other scenarios aren't unrealistically set aside), and only one expert assumed that scenario 5 may be left out of the fusion, due to the excessive narrative on national sovereignty. The other four experts didn't fully refuse the claim, but consider the issue to be manageable in a diplomatic fashion, thereby creating an advantage/disadvantage solution with countries more rooted in nationalism.

Based on the round table's scenario, they further added details regarding the facets of the budget that are of interest to the small- and medium enterprises across Europe, with extra focus on the sector within Hungary:

- Small- and medium enterprises should expect to improve their innovativeness in the future, as the shift in importance will cover innovative solutions and digitisation more and more during the 2021-2027 programme period.
- The measurement of innovation and sustainability will become an important area to develop for the European Union after the effects of COVID-19 will disappear, and a stricter policy regarding the decisions related to tenders are to be expected. Authors' work regarding the

measurement of innovation in the European Union was validated by the experts in theory, and a practical model creation for simplifying innovation and sustainability measurement that adheres to the criteria system of the European Union was suggested as soon as possible.

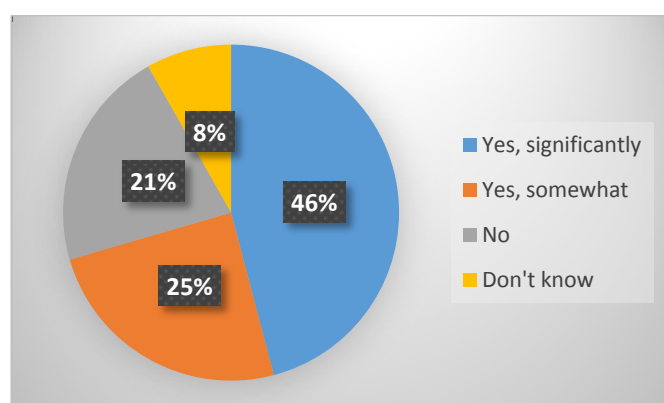
- Authors' earlier work related to the budget [22] was supported as well, and experts noted that the budget analysis and the innovation and sustainability policy measurement and parametrisation should be researched together after the COVID-19 impacts lessened enough to get a general outline of major changes in the European Union.

Based on the results of the round table conference held with the participation of the experts, the authors also conducted a short and simple questionnaire, to gauge the relative limitations of small- and medium enterprises in their upcoming financial and management policy changes. The importance of the European Union's budget for small- and medium enterprises, as previously stated, comes from the subsidy system, where tenders are designed for the sector to improve innovation and business capacity based on requirements given by the European Union, as the organisation subsidizing. This results in the necessity of small- and medium enterprises conforming to said requirements, in order to obtain funding.

Authors contacted several micro-, small- and medium enterprises to obtain data relevant to their findings. A short summary of the results from the experts' dialogue was given to the enterprises, and several key points were introduced to them. A short questionnaire then asked them about their expected investment changes and costs based on their evaluation of the information given.

- The enterprises came from varied sectors, and numbered 61 in total.
- The smallest was a SOHO (self-office or home office) enterprise made up of one person's self-employment.
- The average number of employees of the questionnaire's enterprises was 13,9.
- The largest enterprise in the sample employed 86 people.
- No unusable results came back from the sample.

Enterprises were first asked to provide feedback on the European Union's shift in policy, and if they believe there will be a necessary shift in management policy as well. Results can be seen on Figure 2.

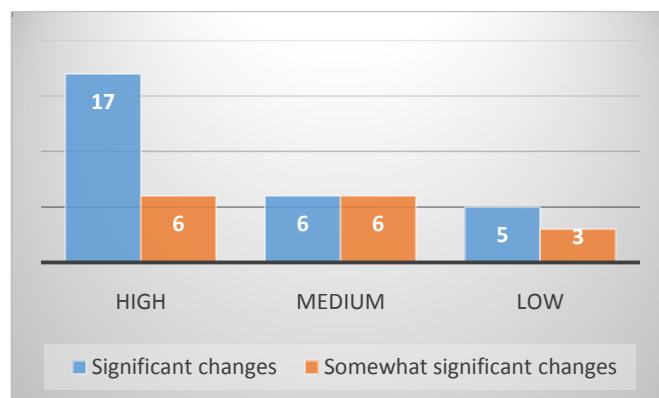


2. Figure: The expected changes in management investment costs of sample enterprises, relative to changes in EU budget directive, n=61. Source: self-made.

There was a significant number of enterprises believing that they will have to give some form of change in order to become available as an entrant for European Union tenders. There was no significant relation between the sector of the enterprise and the belief, which is an interesting result, as Hungary will soon be one of the receiving Member States profiting from an enormous European Union subsidy program aimed at the agricultural sector. Five enterprises

stated that as the data of specific changes isn't available, they can't make a judgement as of yet.

Enterprises providing a positive answer were also asked to estimate the excess costs relative to normal spending on management costs due to shifts in the European Union's budget. Answers can be seen on Figure 3 below.



3. Figure: The estimated levels of excess management investment costs, n=56. Source: self-made.

As seen above, those that expected significant changes also tended to expect generally high investment changes as well.

4. Conclusions, suggestions

Based on the round table, the authors concluded that the financial stability of the European Union is expected to increase, however, there needs to be a larger breathing room, currently unavailable due to the COVID-19 pandemic. The European Union's policies are expected to gradually shift towards a different route of development, but according to the consensus of the experts, the shift will happen slowly enough that it could be followed, and adaptability should be high for the innovative small- and medium enterprise sector.

The increasing stress on the Member States to cooperate with each other, especially due to the COVID outbreak, and the issues concerning rule of law and national sovereignty are a prelude to a chaotic issue regarding the Member States' financing via various subsidy programmes. Most Member States are of the opinion that a strong European Union must stand up to the challenges together, but there are some Member States that don't share this opinion as far as making gestures towards fellow Member States, in the form of relenting some authority over key issues. This leads to heated debates especially about the European Union's budget, however, similar issues are also appearing more and more on the national level within Member States. The experts highlighted this as the cause of a possible future fallout in regards to the subsidy programme, and foreseeing a weakening of joint goals. However, the critical points of the European Union's goals will likely not change.

Authors will continue the research into simplifying the criteria system of the European Union, and advise the relevant research to branch into the fundamentals of the European Union's theoretic policy about grading of innovativeness and sustainability potential.

Furthermore, authors recommend deep analysis into the European Union's key factors which determine the issues having significant effects on the budget discussion: issues such as sovereignty, national identity, European identity, differences of tolerance, acceptance, and refusal of foreigners by nation, and other topics. It's important to note that there are several nations in the European Union with vastly differing histories, and these differences need to be analysed, and bridged in order to facilitate a strong joint stance in most of the European Union's current issues. A good starting point would be to cross-reference the European Union's frameworks with available nationality data, for example, Hofstede's analysis into national specificities, and try to find a common ground

which makes it possible to factor in key differences between nations when designing the policy system of the European Union in the future, or when tailoring subsidy systems for Member States.

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Innovations and corporate social responsibility in bulgarian SMEs – status and prospects

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Abstract: *In today's business environment, companies need to take responsibility for their actions and their impact on stakeholders. The company-society relationship must be reciprocal. In this report, we consider implementing innovations in SMEs doing business in Bulgaria, the performance of CSR in practice and whether there is an understanding of the relationship between them. The theory is illustrated by data from an empirical study of 300 companies by the report's author.*

KEYWORDS: INNOVATION, CSR, COMPETITIVENESS, SMES

1. Introduction

Generating and implementing innovation is imperative for any company that seeks to achieve a competitive advantage in the conditions of Industry 4.0. The concept of Corporate Social Responsibility (CSR) is companies' commitment to becoming equal participants in the community's public regimes as corporate citizens. The connection between innovation and CSR is expressed in the reciprocity of the company-society relationship. In the initiative to achieve competitive advantages while increasing their financial results, companies must also contribute to improving society's welfare and implementing innovations in CSR.

2. Literature review

The concept of innovation involves generating and introducing new combinations of resources to create and add value to and from the organization and invoke both the stakeholder's well-being and competitiveness [1,2]. Innovation can be found in a variety of forms (product, marketing, process, social), produced from different sources (closed and open innovation) and associated with other aspects of change (incremental, radical, destructive) [3,4].

Business organizations are vital to transition to sustainable strategies and policies to address society's socio-economic and environmental challenges. Mentioned is where CSR's connection with innovation opens, which provides opportunities to offer real solutions that strengthen both the value of the brand and social prosperity.

Corporate social responsibility is also essential for increasing the competitiveness, sustainability and economic advantages of companies. Adopting CSR's concept, companies are much more successful in risk management, efficiency, and effectiveness in maintaining favourable relationships with stakeholders and human resource management.

One of the most popular definitions of CSR is Carroll [5], which defines a business's corporate social responsibility due to harmonious, lasting interactions of economic, legal, ethical, and philanthropic obligations.

The concept of CSR is based on three dimensions - environmental protection, social and economic elements.

In the modern research literature, two more have been added - the voluntary aspect and the stakeholders.

Another researchers, Van Marrewijk and Werre [6], understand CSR as an integrated process. Each organization must define its specific sustainable goals to be much easier to adapt to changes and challenges coming from the business environment.

The CSR implementation by companies has a significant contribution to economic development [7] and reflects companies' commitment to responsible management of their actions. Numerous empirical studies have shown that firms that implement CSR initiatives perform better than their competitors [8,9,10].

Several authors conclude that companies that neglect CSR activities are less successful in innovation [11].

When companies are strategically committed to adopting and acting by CSR principles, they also have a significant opportunity to create value and increase competitive performance [12].

Therefore, CSR stimulates innovation by incorporating policies associated with the concept into their competitive strategies, ie. CSR investments are a source of opportunities, innovation and competitive advantage. CSR stimulates the generation and introduction of innovations for several main reasons. In the context of CSR initiatives, companies improve relationships with their customers [13,14]. They attract investment and help retain highly skilled employees, talents who can adequately support the optimization of innovation processes [15]. The conducted empirical research proves the positive impact of the created value for the stakeholders and the applied management strategies associated with innovations [16].

We can argue that at the corporate level, CSR increases competitiveness [17,18,19] while helping to create shared value. [20].

From a theoretical perspective, the two-way connection between CSR and innovation is substantiated. The various aspects supporting the implementation of innovation processes are considered [21], and organizational performance [9]. The empirical evidence for the two concepts' relationship is still relatively limited [22].

The link between CSR and innovation is based on a reciprocal principle - by solving current social problems of communities, companies increase their overall competitive performance. In the context of the two-way nature of the CSR-innovation relationship, we can point out that it positively affects the corporate reputation and hence the overall performance, and on the other hand, helps to solve environmental and social problems.

Therefore, the link between CSR and innovation needs to be further studied and refined to create a more comprehensive, relevant research framework to support the successful implementation of companies' actions in the context of sustainability.

We can conclude that the implementation of innovations in the context of CSR leads to an increase in the overall organizational performance by responding to specific societal needs and current ones that require the implementation of innovations.

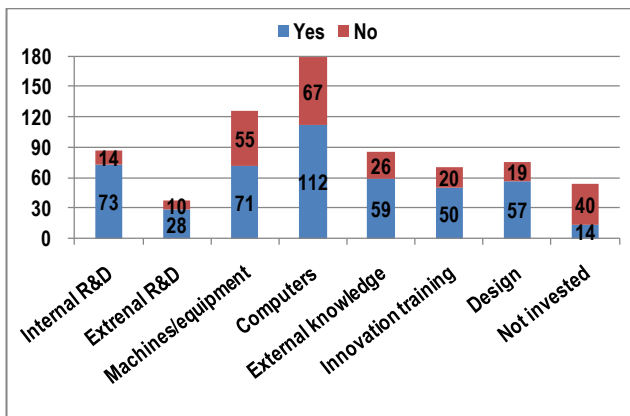
The balance between the interests of the stakeholders and the company contributes to the generation of innovations. They search for answers to current public needs and anticipate the influences and emerging on the horizon of expectations and trends. The presumption of their sustainability includes their compliance with the principles set out in CSR.

3. Empirical study – results and discussion

For this study, we consider the relationship between innovation and the application of CSR principles in the context of Bulgarian companies. The sample was compiled based on the number of companies that implement innovations in the Republic of Bulgaria

according to NSI data. The surveyed companies' profile is as follows: the number of respondents - 300 companies, the owners or managers of the companies were interviewed according to a pre-prepared survey with closed questions. The survey was conducted in the period March-April 2020, under the project "Innovation in business and education. Development of a conceptual communication model for innovations in a business-information organizational environment". The size of companies is determined according to the European classification - micro enterprises - 159 respondents, small - 92, medium - 32, large - 5. Twelve of the respondents did not answer to which group it belongs the company they run. From the demographic profile, we see that the respondents in the survey are mainly representatives of SMEs. The following areas of activity are presented - production, services, light and processing industry, machine building, energy, science and education, IT, construction, design, engineering.

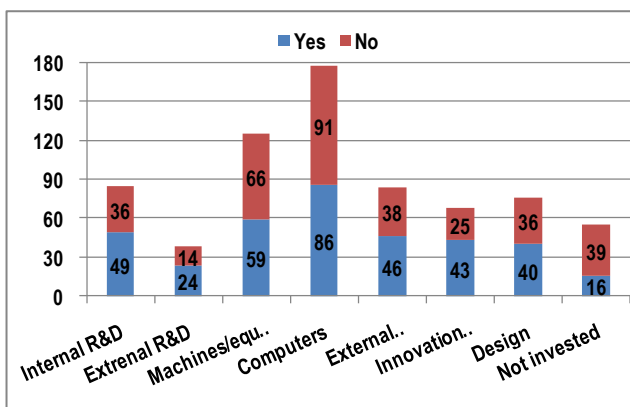
Fig. 1. In the last three years, has your company invested in/Has innovative products and services been developed in your company?



Respondents whose companies have developed innovative products and services invest mainly in the purchase of modern computers and equipment, in internal research, in an external examination, in design. They also focus on conducting training related to increasing organisations' capacity to generate innovations, external study, and external knowledge.

Companies that do not have developed innovative products and services invest mainly in purchasing computers and equipment. They also have a more significant percentage of those who do not have investments associated with innovation.

Fig. 2. In the last three years, has your company invested in / Do you apply the concept of CSR in your company?



Respondents in this study, who apply CSR principles in their work, invest mainly in purchasing computers, modern machines, internal research, external knowledge, external research, training, design. They support principally tangible assets - devices and computers, followed by intangibles - external R&D, internal R&D and acquisition of external knowledge.

Fig. 3. Are there any innovative products and services developed in your company? Do you have innovations related to Corporate Social Responsibility?

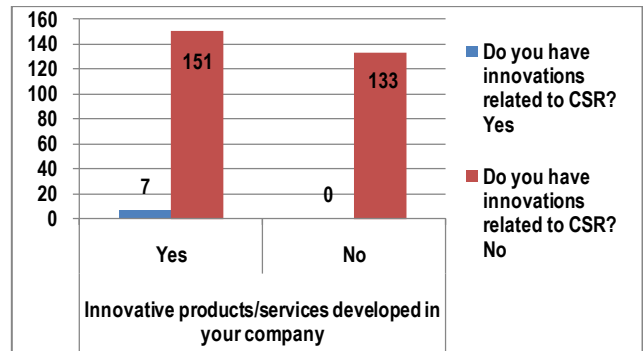
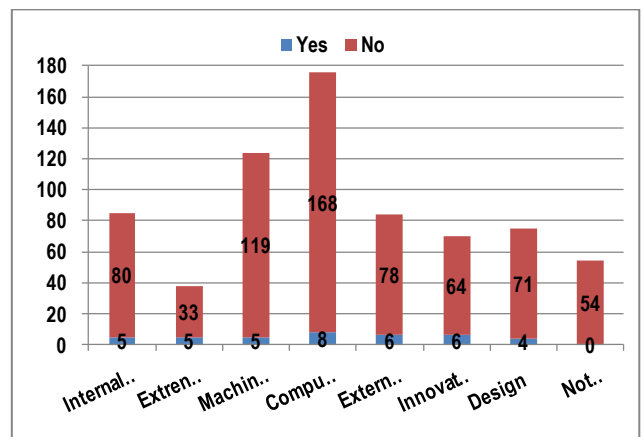


Fig. 4. In the last three years, has your company invested in / Do you have innovations related to the concept of CSR?



The innovations carried out by the respondent companies related to CSR are too few. This type of innovation is implemented by companies creating innovative products and services.

4. Discussion

As a result of empirical research, we can draw some basic conclusions. Companies operating in Bulgaria, which implement innovations, invest mainly in tangible resources - computers, modern equipment. There is also investment in intangible resources but to a lesser extent. This conclusion is in line with a previous study in which the author of this article participated [4].

Firms that apply practices in line with CSR's concept invest more in acquiring resources to support innovation efforts. They also show a predominance of investments in intangible resources.

A minimum number of respondents in the survey implement CSR innovations, even though they are oriented towards creating and introducing innovations in general.

5. Conclusion

The empirical study results clearly outline the trend of lack of innovation in CSR in Bulgarian business practice.

A possible reason for this is the lack of knowledge about the concept's essence and its opportunities to increase companies' overall competitive performance. Another reason could be the lack of a clear assessment of the extent to which the costs associated with implementing the CSR concept and compliance with its standards are financially justified.

It is essential to find the factors that imply access to information about their nature, creating appropriate communication programs. It is also necessary to formulate a framework through which SMEs can help realize CSR-related innovation's natural effect on competitiveness and maintain reciprocal business-to-society

relationships. One of the results will be that the generation and introduction of CSR innovations will help positive companies' financial performance.

Transparency and openness, which must be embedded in organizations' policies, are crucial for successfully implementing programs to promote CSR innovation's importance and subsequent performance. **Literature:**

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Lean concept transformation of the packaging procedure in the process of final processing

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Abstract: In production where is a possibility that the surface protection process takes place at the same location as the production of semi-finished products, but also at a remote location, internal transport, excessive processing, unnecessary stocks and movement of employees are often singled out as the main types of waste or losses in the packaging process. These wastes have economic and environmental consequences, so Lean transformation was imposed as a methodology for streamlining production flows in the packaging of finished products. The transformation involved a set of procedures that found activities that do not contribute to increased costs in the production process. This contributed to increasing the level of quality characteristics and reduced costs and time of realization of the production cycle. The results of this transformation in production flows are presented in diagrams and tables and serve to assess the economic efficiency and environmental compliance of the activities undertaken.

Keywords: TRANSFORMATION, PACKAGING, INTERNAL TRANSPORT, OVERPROCESSING, LEAN CONCEPT, LOSS, VALUE ADDED CONTRIBUTION

1. Introduction

Engineering prediction of the realization of a production task strives for a constant search for answers to three questions: How faster?; How better?; How about less costs? On this track, the idea came up to streamline material flows by transforming packaging processes where over-processing and internal transport caused increased consumption of auxiliary materials and loss of time. Searching for the results of research on this issue in the automotive industry in Europe, and published papers that could bring the experiences of other companies as empirical data, and engaged in the production of light alloy wheels, could get a clear picture of the defined subject of research.

As each production is a kind of laboratory, it was decided to monitor and record the existing situation, defining the paths of the material and its processing that apply waiting (hot spots), approach to transforming the finishing process Lean concept to reduce total production time and reduce the cost of auxiliary material by elimination and substitution of the storage site.

Lean transformation involves improving the way value is delivered to the customer. Withdrawing and removing non-value-added activities and reducing the time to perform value-added operations. In order to achieve this, it is necessary to identify activities that do not add value and companies to take immediate action to eliminate them. This means that workers need to learn to see waste. The first step in Lean transformation is for people to see waste and become aware that these are unnecessary losses for everyone. [1]

At the heart of the Lean concept is a worker-based system. The success of any Lean transformation depends on the engagement of each employee in the process of continuous business improvement. All employees must be ready and able to solve all complex problems. Those in senior positions, who make decisions that affect everyone, are the most responsible and they have the power and authority to drive change.

The paper will show the steps of transformation of material flows with the "Lean concept", and on the basis of which the production losses were influenced by the rationalization of material flows and the rationalization of production time. This will reduce the cost of the packaging process, and the consumption of wood as a natural resource.

2. Aspects of the packaging procedure in the process of final processing

The surface protection process can take place at the location where the semi-finished product is located, and also due to the specific requirements of customers, the surface protection process is possible at a remote location in relation to the location of the semi-finished product.

The packaging process will be viewed by management from an economic point of view, so packaging costs will be key to making a decision on the transformation of production flows, in order to optimize total costs. From an environmental point of view, the costs of energy consumption, packaging materials and transport will be key to making the same decision.

3. Transformation of the packaging procedure

All employees underwent training in the Lean concept and Lean tools. Also, employed engineers study new ways of thinking and behaving that support the development of a Lean culture, or a culture of continuous improvement. [2] Employees have mastered the PDCA (Plan - Plan, Do - Do, CHECK - Check, ACT - Act) cycle, which, as a discipline that holds all organizational processes together, improves them on a continuous basis.

3.1. Strategic and operational level of application of Lean transformation on the finishing line

Management decided to apply Lean tools that have proven their applicability in similar industrial plants and have gained their confirmation of the benefits provided by their application. The advantages of using 5S tools can be described as follows: 5S is a system for creating an organized workplace in a way that eliminates waste and useless activities, but also that irregularities that occur in everyday work are easily noticed and eliminated. The application of this tool is not only the achievement of a better organization with a one-time action, but the creation of conditions for the newly created packaging organization to be maintained and improved in continuity. This tool contains five groups of rules, ie steps or phases that were performed during the implementation of the observed packaging operations. Figure 1 shows the five phases for implementing the 5S tool. [3]

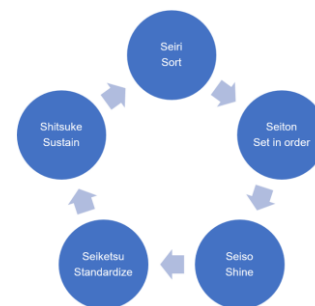


Fig. 1 Five phases of 5S tool [3]

Observing the finishing process, which precedes the surface protection process, it was observed that the semi-finished products are packaged in the same way for both locations where the surface protection process takes place. Due to the need to protect semi-finished products from the adverse effects of transport to a remote location, EPP pad, wooden cover, PET tape and stretch foil are used

for packaging. Also, semi-finished products, which remain in the same location where the production takes place, are packaged in an identical way. All semi-finished products after the finishing process are shipped to warehouse A (remote warehouse), which is used for storage of semi-finished products, from where they are re-shipped according to the surface protection process, according to Table 1.

Table 1: Time consumption by packaging phases before Lean concept transformation

Phase	Time (s)
Taking the pallet and bringing it to the machine	32
Recording in MES (Manufacturing Execution System) + manual completion of the delivery note	172
Installing the EPP pad and cover	43
Clamping with PET tape	159
Clamping with "stretch" foil	136
Transport of packed pallet to warehouse A	153
TOTAL	695

The situation in the plant is such that 58 % of semi-finished products are covered by surface protection at the location where they were produced, while 42 % of semi-finished products are shipped to surface protection at a remote location.

Based on the above, the opportunity to transform the Lean concept of the packaging process in the finishing process was recognized.

3.2. Experimental approach

The goals of this paper are:

- Reduce overprocessing, internal transportation, unnecessary inventory and employee movement;
- Develop and introduce a new packaging process in the finishing process based on the Lean concept;
- Assess the efficiency of the new packaging process based on the measurement of the time required for each individual phase of packaging after the transformation of the packaging process, and in relation to the situation before the transformation shown in Table 1.

3.3. Experiment plan

The experiment is planned in such a way as to eliminate the use of EPP pads, wooden cover, PET tape and stretch foil for semi-finished products, which will go into the surface protection process at the same location where the production of semi-finished products takes place. Also, the use of warehouse B, which is located in a location closer than the location of warehouse A, was considered, and there was an opportunity for adjustments in ERP (Enterprise Resource Planning), MES (Manufacturing Execution System) and WMS (Warehouse Management System) systems that would facilitate the implementation of planned activities.

3.4. Experimental results and discussion

First, the position for storage of semi-finished products in warehouse B was determined. The space was used in such a way that the entire quantity of products covered by the surface protection could be stored at the location where their production is. Then a new way of packaging was designed, which included organizational changes. The number of products per pallet remained the same as before the transformation, as well as the phase of taking the pallet from the warehouse and bringing it to the machine.

A new way of recording products on the packaged pallet was introduced, by eliminating the manual filling of paper records that served as a database of packaged products, and the same was replaced by full registration through the ERP system. EAN-13 (European Article Number) codes were introduced, which were linked to ERP-WMS, and a web application with an article database

was created. Also, what is most important is that the use of packaging material in the new packaging method has been completely eliminated.

The procedure for the new packaging method is as follows:

- Taking the pallet and bringing it to the machine;
- Recording the pallet through the database and printing a label containing the product data from the EAN-13 code and gluing it to the pallet;
- Transport of packed pallet to warehouse B and recording it through WMS.

Table 2 provides an overview of time consumption by packaging phases after transformation.

Table 2: Time consumption by packaging phases after Lean concept transformation

Phase	Time (s)
Taking the pallet and bringing it to the machine	32
Recording (MES – Web application)	70
Transport of packed pallet to warehouse B and recording it through WMS	100
TOTAL	202

4. Analysis of results

Observing from the economic aspect, and based on the data shown in Tables 1 and 2, and Diagram 1, it can be seen that the loss of time and auxiliary material due to overprocessing in the packaging of semi-finished products decreased by 81.18 %, and the loss of time due to transport of semi-finished products by 34.64 %. Taking into account the reduction of these losses, the efficiency of packaging in terms of saving time increased by 70.94 %. The transformation of the Lean concept optimized the packaging process of semi-finished products that are surface protected at the location where they take place, and by eliminating the use of EPP pads, wooden lids, PET tape and stretch foil, packaging costs were reduced by 89.37 %. From an environmental point of view, the non-use of packaging materials such as EPP pads, wooden covers, PET tape and clamping foil has led to a reduction in wood consumption as a natural resource and a rationalization of material flows, as shown in Figures 2 and 3.

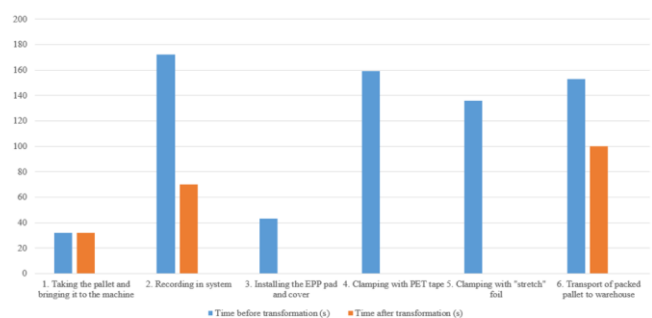


Fig. 2 Comparison of time consumption by packaging phases before and after transformation

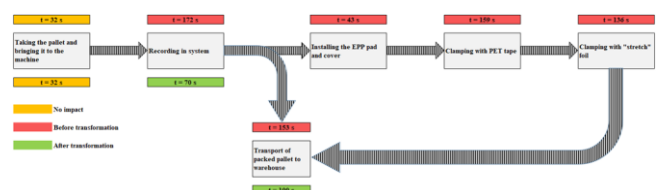


Fig. 3 Flow diagram before and after transformation

5. Conclusion

In a production process, such as a final processing, it often happens that it is possible to undertake a transformation with the

Lean concept, on the basis of which it is possible to eliminate or reduce to an acceptable level certain production costs.

The transformation with the Lean concept, presented in this paper, included activities related to the reduction of key losses in the process of final processing – overprocessing, internal transportation, unnecessary inventory and employee movement.

The following activities have been undertaken:

- The use of warehouse B, which is closer in location to the finishing process in relation to the previously used warehouse A;
- Changes in ERP, MES and WMS systems;
- EAN-13 codes have been introduced that are linked to the ERP and WMS system, and a web application with an article database has been created;
- The use of packaging material, EPP pads, wooden lid, PET tape and clamping foil has been eliminated.

Indicators of the new performance of the packaging process are:

- Reduced duration of the process by 70.94 % compared to the initial state;
- Reduction of stock of packaging material by 66.67 % compared to the initial state use of warehouse B, which is closer in location to the finishing process in relation to the previously used warehouse A;
- Free space for 50.00 % by removing unnecessary equipment and items;
- Reduced number of activities in individual packaging operations from 59.30 % to 100 %;
- Reduced unnecessary movement of employees by at least 63.31 %;
- Reduced internal transport by at least 34.64 %.

It is proposed to the management to define new material flows, a new package of auxiliary packaging material, and to incorporate them into constructive-technological preparation. Technologists and managers of individual production processes should apply the changed procedure, and with managers get used to them until routine application. In addition, quality control technologists should also harmonize their procedures with the new technological process.

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Effective determination of clomazone and oxyfluorfen in aqueous samples by differential pulse stripping voltammetry

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Abstract: Two novel types of electrochemical sensors were obtained by chemical deposition of Pt and MoO₂ nanoparticles onto multi walled carbon nano tubes (MWCNT) and used for determination of frequently used pesticides clomazone and oxyfluorfen in aqueous media via differential pulse stripping voltammetry (DPSV). Clomazone was determined in 0.1 M phosphate buffer solution at pH 7.0 in the concentration range 0.61–20.56 ng cm⁻³, with LOQ = 0.61 and LOD = 0.38 ng cm⁻³. Oxyfluorfen was determined with good reproducibility (RSD <2.4%) in the concentration range 2.5–34.5 ng cm⁻³, with $r = 0.99$ and a LOQ of 1.5 ng cm⁻³. Results were in the same range measured by HPLC/DAD, which was used as comparative method. Structural characterization of the doped MWCNT performed by Raman spectroscopy, field emission scanning electron microscopy (FE-SEM), high-resolution transmission electron (HR-TEM), and X-ray crystallography revealed a preserved MWCNT structure decorated with well dispersed species of the platinum and MoO₂ nanoparticles.

Keywords: ELECTROCHEMISTRY, SENSOR, VOLTAMMETRY, NANOPARTICLES, PESTICIDES, ENVIRONMENT

1. Introduction

In era of rapid industrialization and technological development, the environment is increasingly polluted on daily basis. There are numerous sources of pollution that in many ways emit different groups of pollutants into the immediate environment from where they can migrate to water resources, air, soil, the food chain and ultimately to human beings. Exposure of humans to harmful compounds of anthropogenic origin can have unforeseeable consequences for his health. Therefore, it is necessary to always keep in mind possible sources of pollution and ways of migration of toxic substances in nature and reaching the resources which are in direct contact with humans.

Pesticides are mainly organic artificially synthesized compounds that are nowadays widely used in agriculture to protect plants from insects, fungi, rodents, etc. Their use in the allowed quantities, respecting the time of spontaneous degradation, does not pose a great health risk. However, a major problem with them is that they are in some cases difficult to biodegrade, can accumulate in living organisms while their breakdown products are stable and toxic. The harmful effects of pesticides vary from case to case, but it has been proven that some of them can be carcinogenic, mutagenic, teratogenic or have a number of negative effects on some systems of organs. Therefore, monitoring of pesticides in the environment, and especially in samples that are closest to humans (food, water) is of great importance.

Pesticide concentration is determined mainly by the techniques of liquid or gas chromatography [1]. These techniques are precise but impractical due to the high cost of analysis and the cost of the instrument itself, the need for a highly educated workforce as well as the essential sample preparation steps. Therefore, it is necessary to develop new techniques that with the same levels of reliability, selectivity, accuracy and precision can eliminate these shortcomings.

Voltammetry embraces several electroanalytical methods acknowledged as a sophisticated means for sensitive and rapid determination of a lot of compounds, including pesticides. Electrochemical sensors are a type of sensors where working electrode in three electrode system acts as the transducer. The production and use of these sensors took off in the last decades and found commercial applications in many fields. They are characterized by the few facts: the use of the electron for signal acquisition, which is considered a clean model for analytical applications, with no generation of waste, miniaturization in portable devices (analyses with microvolumes of samples), fast analysis, and low production cost [2].

Multi-walled carbon nanotubes (MWCNTs) are chemically stable material with superior mechanical, electrochemical and optical properties. Their increased sensitivity in sensing platforms is among the rest due to high surface area to volume ratio and pronounced electrical conductivity. Electrochemical sensors based on multi or single walled CNTs can improve electrical and

mechanical properties of composite electrodes which result in higher peak currents and lower overpotential [3]. There are numerous reports where the role of nanocomposites of specific compositions in improvement of electrode response have been elucidated [4,5,6].

This study is based on the fact that immobilization of metallic nanoparticles onto carbon support can boost the advances of electrochemical sensors and lead to enhancements of their performance and application in pesticide determination.

2. Experimental

2.1. Synthesis of materials

The commercial glassy carbon (GC) electrode was modified by the following procedure. For a start, 50 mg of MWCNT (carbon > 95%, outer diameter × length: 6–9 nm × 5 μm) purchased from Sigma-Aldrich was dispersed in 20 cm³ mixture of ethanol and water (50v/v%). Then, 0.5 cm³ of aniline was introduced into suspension and sonicated for 90 min. In the first case, 26.7 mg of chloroplatinic acid hydrate (H₂PtCl₆ × H₂O) was dissolved in 2 cm³ of water and added to suspension which was then sonicated for the next 5 min and acidified afterwards with 0.5 cm³ of 5 M HNO₃. In the second case, 16.3 mg of sodium molybdate dihydrate (Na₂MoO₄ × 2H₂O) was dissolved in 2 cm³ of water and introduced into sonicated suspension. For the reduction of ions of platinum and molybdenum, excess of 20 mg of sodium borohydride was dissolved in 2 cm³ of water and then added to the suspension. It was stirred for a while and left for the next 24 h, then filtered and rinsed with deionized water to neutral pH. The obtained materials were dried at 90 °C for a couple of hours and used as the electrode modifier in voltammetric analyses.

2.2. Characterization

Field emission scanning electron microscopy (FE-SEM) was used for morphological characterization of the samples on microscope JEOL JXA-8500F. High resolution transmission electron microscope (HR-TEM) images were recorded by JEOL JEM-F200. Diffractometer (Rigaku Corporation, Japan), with CuKα radiation ($\lambda = 1.5406$ Å) and a scan speed of 5°/min was employed in X-ray analyses in the range between 0 and 60°2θ, with a step 0.02°.

2.3. Electrode preparation

Quantity of 5 mg of doped MWCNT sample was dispersed in 1 cm³ of ethanol/water mixture (40 v/v%) and homogenized in the ultrasonic bath for 30 min. The surface of the GCE electrodes was polished on fine cloth using alumina powder. A drop which contained 15 mm³ of the previously prepared suspension was transferred onto the GC electrode (working area was 0.04 cm²) and dried under nitrogen stream. After the thin carbon layer was dried, it was covered with 10 mm³ of 0.05 wt.% Nafion in ethanol. The solvent was removed by evaporation.

2.4. Voltammetry

Pesticides were analyzed by the means of differential pulse stripping voltammetry (DPSV). Working solutions with pesticide in 0.1 M phosphate buffer were analyzed in a conventional voltammetric cell with operating volume of 10 cm³ by using 797 VA Computrace analyzer (Metrohm, Switzerland) controlled by 797 VA Computrace software (version 1.2). A three-electrode system consisted of Ag/AgCl electrode (saturated with KCl) as a reference, platinum wire as auxiliary and dopant-MWCNT modified GCE as working electrode.

Prior to each run, the working electrode was electrochemically activated by potential cycling in various range with speed 0.1 V s⁻¹ for 10 cycles. The background voltammograms of the supporting electrolyte were recorded under the same conditions. The blank probe containing only the supporting electrolyte was recorded under the same conditions. Before starting a new set of measurements, the supporting electrolyte was deaerated by suprapure nitrogen for 5 min and then volumes from pesticides stock solutions were added to probes in order to make desired concentrations. The parameters for DPSV differed in the case of two different materials applied. Detailed instructions and descriptions of the working conditions can be found in the referred literature [7,8]. All the measurements were performed at room temperature (23 ± 1 °C).

As comparative analysis, concentrations of pesticides were determined by Shimadzu Prominence high performance liquid chromatography (HPLC) system (Shimadzu, Japan).

3. Results and discussion

3.1. Structure of doped MWCNTs

The microstructure of the materials can be seen in the SEM micrographs (Fig. 1A and Fig. 1D) at magnifications of 100,000 times. Morphology typical for MWCNT is noticed at both images. It is seen that MWCNT bundles are randomly orientated. Diameter of carbon nanotubes is in range of 10 nm while the length can reach more than several micrometers. However, the exact length is difficult to be measured because bundle is always in the meandering form. Pt nanoparticles are observed as light spots or clusters on the surface of MWCNT with diameter of up to 30 nm. In the case of MoO₃ doped MWCNT the dimensions of doping particles is estimated to be in the range mainly of 10 to 20 nm.

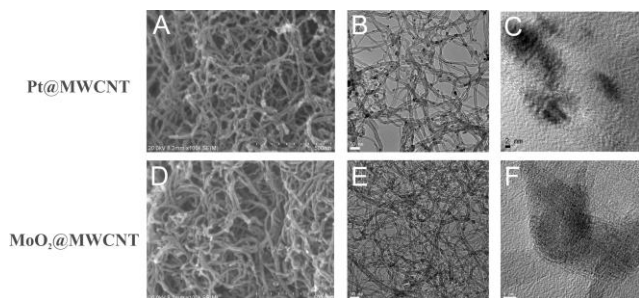


Fig.1. FE-SEM and HR-TEM images of Pt@MWCNT and MoO₃@MWCNT

The TEM image of Pt doped MWCNT given in Fig. 1B indicates that Pt nanoparticles are well dispersed and successfully deposited on the carbon support. There is no evidence that the interior of the MWCNT is filled by Pt phase. Additionally, a higher magnification TEM image reveals that Pt nanoparticles intrinsically agglomerate and form aggregates with an average diameter of ~10 nm (Fig. 1C). It should be noted that Pt nanoparticles are firmly attached to the surface of CNTs since there is no single observation of freely (detached) Pt nanoparticles after analysis a large numbers of samples.

HR-TEM microphotographs of MoO₃ doped MWCNTs given in Fig. 1E and 1F display a hybrid structure of samples consisting of MoO₃ nanoparticles firmly attached to the surface of MWCNT, forming a hairy morphology with discrete dark spots of MoO₃ nanoparticles. Such distribution of nanoparticles was achieved by in situ reduction of dissolved Na₂MoO₄ in a suspension containing well-dispersed MWCNT according to reaction (Eq. 1):



Higher magnification (Fig. 1F) provides a better view on MoO₂ nanoparticles revealing their polyhedral or round morphology and diameter of the particles in the range from 7 to 50 nm. Larger particles are agglomerates composed of smaller ones which are stacked together. Aniline is used for obtaining stable nanoparticles of MoO₂. It is assumed that most of aniline present on the surface of MWCNT was washed out during the rinsing step and that the remained surface concentration was not significant for electrocatalytic effects.

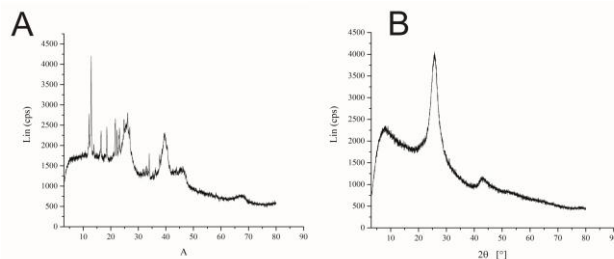


Fig.2. XRD patterns A) Pt@MWCNT and B) MoO₃@MWCNT

The powder XRD technique was used to investigate the crystal structure of the platinum and molybdenum oxide modified MWCNT. As seen from Fig. 2A, the XRD profile of Pt@MWCNT contains peaks for MWCNTs at 26 and 43° 2θ which correspond to the planes of (220), (301) and (002), respectively. These peaks represent the graphitic reflections. In the case of MoO₂ doped MWCNTs (Fig. 2B), a strong intensity at 26° and a weak intensity at 44° are noticed, which correspond to the (002) and (100) planes of the graphite crystal structure as in previous case.

3.2. Electrochemical analysis

The cathodic response of clomazone by the Pt modified MWCNT was evaluated with respect to the several supporting electrolytes including potassium chloride, phosphate buffer and Britton–Robinson (B-R) buffer. Phosphate buffer was found to be the most suitable medium while in case of MoO₃@MWCNT it was B-R solution. For the first case, the DPSV curves were recorded in 0.1 M phosphate buffer (pH range 5.8 – 8.0) to study the effect of pH on the voltammetric behavior of clomazone. As clearly noticeable from Fig. 3, there is no linear relationship between DP peak potential and pH. It is indicated that mechanism of clomazone reduction is complex with no common electrochemical behaviour involving addition of hydrogen. It has been ascertained from experimental data that the best peak shape and the maximum peaks current occurred in the 0.1 M phosphate buffer at pH 7.0. This is the reason why it was chosen for further investigation as optimal parameter from the analytical point of view. For comparison, there was no electrochemical response of clomazone with bare GC or MWCNT/GC electrodes. The optimal value of –0.25 V was chosen for deposition potential which coincides with the reduction potential for clomazone at selected conditions.

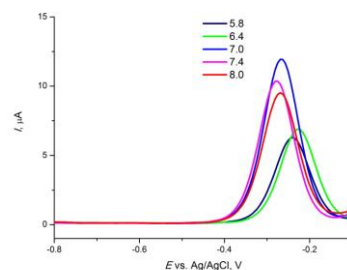


Fig.3. DPSV curves for 10.2 ng cm⁻³ of clomazone at Pt@MWCNT/GCE

Enhanced adsorption and accumulation of clomazone on the Pt@MWCNT/GC electrode after its pretreatment at –0.25 V can be exploited here to significantly increase sensitivity of DPSV. Fig. 4B shows that the signal has hyperbolic shape with prolongation of the deposition time to 900 s. The peak current increased rapidly with

the increasing of deposition time and almost reached a platform after 120 s. For analytical purpose, Pt@MWCNT/GC electrode was initially pretreated during 900 s into the solution of 10.2 ng cm^{-3} of clomazone 0.1 M phosphate buffer solution before it was used for measuring its concentration to achieve down to the low ppb level. The additional preconcentration time of 120 s was employed before each measurement. The calibration graph for an accumulation time of 120 s was linear in the range from 0.61 to 20.56 ng cm^{-3} . Clomazone could be determined by DPSV in the concentration range of $0.61 - 20.56 \text{ ng cm}^{-3}$ (Fig.3). Thereby, the relative standard deviation does not exceed 1.85%.

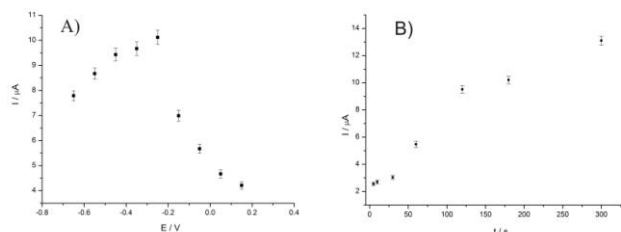


Fig.4. Key parameter optimized for DPSV of clomazone: A) Influence of deposition potential B) Effect of deposition time

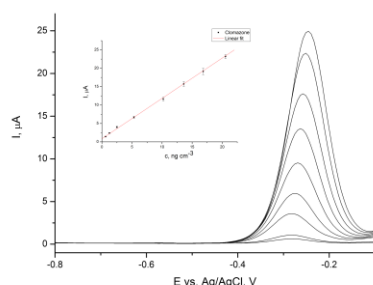


Fig.5. DPSV recorded at Pt@MWCNT/GCE for different concentrations of clomazone in 0.1 M phosphate buffer solution

In case of MoO_2 modified MWCNTs, the sharpest and most favorably developed oxidation peak of oxyfluorfen was obtained at pH 6.0. For DPSV analysis, the current intensity of the signal of interest was found to be dependent upon the deposition potential (E) between $+0.15$ and -0.55 V . As shown in Fig.7, the peak current increased to a potential of -0.15 V and then started to decrease. The optimal value of -0.15 V was chosen for deposition potential. Another optimized parameter was the deposition time (t). Figure 7B shows that the signal has increased with the prolongation of the deposition time to 300 s. As the optimum, a period of 80 s was selected to achieve down to the ppb level.

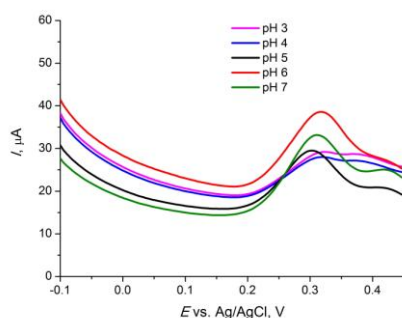


Fig.6. DPSV recorded at MoO_2 @MWCNT/GCE for 24.7 ng cm^{-3} of oxyfluorfen in Britton-Robinson buffer

The quantitative DPSV determination of oxyfluorfen is based on the linear relationship between the peak current intensity at $+0.3 \text{ V}$ and pesticide concentration. As it can be seen, it could be determined by DPSV in the concentration range from 2.5 to 34.5 ng cm^{-3} , with $r = 0.99$ (Fig.8A) and the limit of detection (LOD) of 1.5 ng cm^{-3} , while the RSD did not exceed 2.4%. The RSD value indicates a relatively good precision of the developed method.

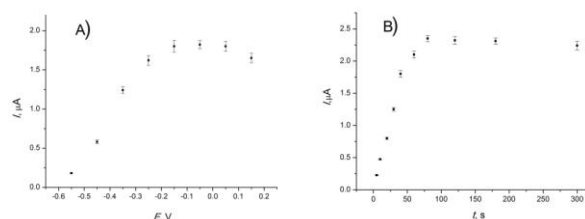


Fig.7. Key parameter optimized for DPSV of oxyfluorfen: A) Influence of deposition potential B) Effect of deposition time

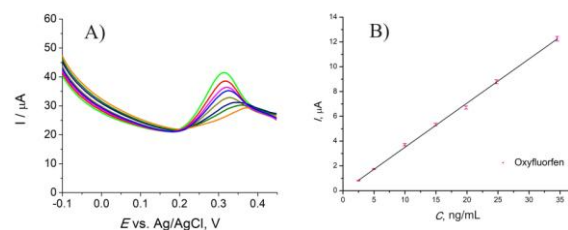


Fig.8. DPSV recorded at the MoO_2 @MWCNT/GCE for different concentrations of oxyfluorfen in B-R buffer (pH 6.0)

The interference of some common ions such as Ca^{2+} , Na^+ , Ag^+ , K^+ , Cl^- , HCO_3^- , CO_3^{2-} , NO_3^- along with common pesticides such as clomazone, carbendazim, tebufenozide, and glyphosate have not influenced significantly the peak potential nor peak current intensity of the determined pesticides.

4. Conclusions

In this study, decoration of MWCNTs with nanoparticles of elemental platinum and molybdenum oxide of various dimension ranges was proved as important factor in enhancing electrochemical/sensing response of GC electrode. The obtained nanohybrid materials possess a favorable morphology with fast electron-transport throughout MWCNT network which enables advantageous electrocatalytic behavior in clomazone and oxyfluorfen detection in water solutions. DPSV have shown to be fairly efficient technique for rapid and sensitive determination of these pesticides, especially in comparison to robust HPLC technique. DPSV attained relatively low limits of detection and quantification which in comparison to concurrent HPLC method leads to conclusion that DPSV can be acknowledged as handy and fast alternative for pesticide determination especially for on-site applications.

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Innovative hybrid fiber-reinforced shotcrete for thin repairing overlays - long term mechanical and structural characteristics

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Abstract: Solvay Sodi JSC, Devnya, Bulgaria, is the biggest calcinated soda production plant in Europe. It is built in 1974 and it works in three-shift production regime up to present days. The exploitation environment is highly influenced by numerous factors, acting corrosively on main reinforced concrete structures – active atmospheric processes (in continental type moderate climate), sea water born chloride-aerosols, as well chemical attack caused by carbon dioxide and nitric acid, presented in the air in form of evaporation. The most reinforced concrete structures need urgent remedial measures to restore the load-bearing capacity of its components.

The "light soda" silos are reinforced concrete facilities (cylindrical shells) which repair has to be executed during continuous production process. The options for repair of these structures and restoration of their load bearing ability by specially designed innovative high-tech hybrid reinforced sprayed concrete, containing capillary crystalline admixture, are discussed in the report.

It is well known that two basic methods of shotcrete exist - "dry" and "wet" ones.

The report discusses the main advantages of the specially designed innovative hybrid fiber-reinforced "wet" shotcrete for thin repairing overlays, compared to industrially made "dry" one. By purposeful using of standard and direct physical test methods the basic long-term mechanical and structural characteristics of two composites are presented and discussed. The effectiveness of the proposed "wet" method is based to real possibilities to involve in concrete mix design different chemical innovative admixtures - ultra high range water reducing (HRWRA), shrinkage compensating (ShCA), internal crystallization one, etc. The mechanical properties of "wet" one dramatically exceeded respective ones of the "dry" one. The mechanical test results fully correspond to the respective long-term structural characteristics, obtained by using of X-Ray, DTA, SEM and BET-methods.

It could be concluded that the innovative hybrid fiber-reinforced "wet" shotcrete is preferable for high-performance thin repairing overlays for structural strengthening of damaged concrete structures.

Keywords: SHOTCRETE, "DRY" AND "WET" SHOTCRETE TECHNOLOGIES, SPECIALIZED CONSTRUCTION REPAIRING WORKS, THIN CONCRETE STRENGTHENING OVERLAYS, INNOVATIVE CONCRETE MIX DESIGN, CONCRETE STRENGTH-DEFORMATION CHARACTERISTICS, X-RAY, DTA, SEM AND BET-METHODS, SPECIALIZED CONSTRUCTION REPAIRING WORKS, THIN CONCRETE STRENGTHENING OVERLAYS

1. Introduction

Currently, the SOLVAY SODI JSC light-weight soda silos "4" and "5" are used for temporary storage and subsequent production distribution. Serious corrosion damages are observed on the concrete surfaces, visible corroded areas of the steel-reinforcement, too. (Photo 1 and 2).



Photo 1 LIGHT SODA SILOS "4" & "5" general view

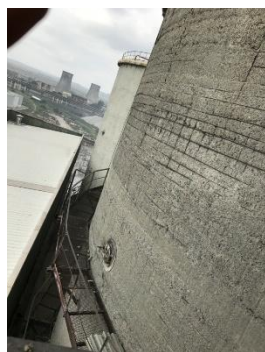


Photo 2 LIGHT SODA SILOS "4" & "5" typical concrete damages

According SOLVAY SODI JSC preliminary experience such type of steel-reinforced concrete structures was repaired by dry ready mix, type "Torkret W81", MC Bauchemie, Germany (MC). The new method proposed was optimized to avoid some disadvantages of the MC - very expensive dry mixture, technical requirements for very thin shotcrete single layer (up to 2 cm), need for at least three layers to reach the design repairing thickness of about 6-7 cm, possible dusting during work, relatively large rate of rebound, etc.

All mentioned above gave me enough reason to propose different type of repairing approach based on wet technology technical and technological basic advantages - high performance productivity (3-5 m³/h), single layer thickness – up to 20 cm (all repairing concrete section deep will be executed in single stage!!!),

rebound reduced (up to 15%), possible to used special designed high-performance fiber-reinforced fine-grained sulfate-resistant sprayed concrete, strict control of concrete mix design (fresh concrete mixed at batching plant under control), possibilities to incorporate in concrete mix different type of fibers (control the shrinkage processes in different hydration stages and dramatically increases resistance to brittle fracture, increase the residual strength after the first crack strength of fatigue in long-term load and impact strength, which ensures the integrity of the concrete even in partial depletion of bearing capacity) and incorporated in concrete mix special deep crystallization admixtures (self-healing, water-proof concrete, 4G - impermeable concrete).

Table 1: Wet-shotcreting mix design, type WS

Ingredients	Quantity, kg/m ³
Sulfate resistant Portland cement CEM I 42,5 SR5	550
River sand, SILISTRA, "Polaris - 8", 0-4 mm	745
Crashed washed sand, ESKANA, "Sini vir", 0-4 mm	300
Crashed stone, PATSTROY, Karnobat, 4-8 mm	505
High water reducing admixture DYNAMON SX, MAPEI, Italy – 0,6% of cement weight	3,30
PP fibers FM150, Propex Concrete Systems, USA	0,900
PP fibers FM300, Propex Concrete Systems, USA	0,900
KMC – thixotropic polymer modifier increasing mix compactness (water solution of 10% dry compound -conforming to a prescription)	10,00
Internal-crystallization chemical admixture KRYSTALINE Add +, Spain	6,0
Mixing water for dry aggregates	~190
Consistency (regarding to slump test), cm	S1

The design for repair of steel-reinforced concrete structures was based on the principle of reasonable sufficiency, without providing for a change in the geometry of the existing sections of the building structure and partial or complete closure of the technological openings in the structure [1].

A special repair wet-shotcreting mix **WS** was designed for this purpose. The mix consists of polymer-modified hybrid fiber-reinforced concrete (maximal size of large aggregate $D_{\max} = 8$ mm), thixotropic polymer modifier and internal-crystallizing chemical admixture (Table 1).

2. Test procedures and analysis of results

Following the SOLVAY SODI JSC requirements before designing and site construction procedure starting, an approved test program was performed. The main purpose of the tests was to compare in parallel the basic technical characteristics of **MC** and **WS** shotcretes, established in real production conditions at different ages.

The basic mechanical and structural results at 7- and 28 days of age, based on respective BDS EN standards, were already published [2].

Trying to confirm the **WS** shotcretes advantages the prolonged tests are additionally performed at 2 years of age. All specimens tested were alternatively seasoned under water and in lab-dry air conditions following the periodic of 1 month for both of them. This type of specimen's aging is very reasonable trying to obtain sufficient differences between two types of concrete – **MC** (based on ordinary concrete based on some water reducing admixtures in dry form) and **WS** (based of special concrete with crystalline technology).

Index tested	Composition	
	MC	WS
Volume density (kg/m^3), at:		
7 days	2230	2320
28 days	2230	2300
2 years	2226	2250
Compressive strength (MPa), at:		
7 days	34,00	51,90
28 days	44,20	60,10
2 years	45,70	64,80
Ultrasonic speed velocity (km/s), at:		
7 days	4,202	4,450
28 days	4,333	4,509
2 years	4,356	4,680
Static modulus of elastic deformation (MPa), at:		
7 days	24,00	35,80
28 days	28,80	37,20
2 years	29,00	39,40
Water penetration depth (mm), at:		
7 days	44	35
28 days	35	20
2 years	33	7
Splitting tensile strength (MPa), at:		
7 days	2,02	2,39
28 days	3,18	3,39
2 years	3,20	3,56
Frost resistance class (cycles passed), C_{fr} , at:		
28 days	(4) $C_{fr}150$	(5) $C_{fr}200$
2 years	(4) $C_{fr}150$	(6) $C_{fr}250$

Crystalline technology products function within the concrete to increase calcium silicate hydrate (C-S-H) in the concrete pores and capillary voids allowing for a better concrete hydration to the point of waterproofing the concrete through internal C-S-H growth.

While a true crystalline technology does lower the w/c ratio, increase the strength, and lower the depth of water penetration as a natural part of the technology, it also goes a step further and offsets the problems that a simple HRWR/SP cannot resolve. Crystalline technology unique abilities not only include reducing shrinkage cracking, but also enhance the concrete's natural hydration process giving it the unique ability to enhance the concrete's natural ability to autogenously heal concrete cracks even years in the future.

The summarized test results are presented in Table 2.

Comparing the test result it is easy to state that, at 28-days and 2 years, the **WS**-compressive strength increased with (7,8%), while that of **MS** increases by about (3,4%). It could be concluded that respective comparison between nominal values shows that, at 2 years of age, **WS** concrete surpass **MC** one with around 42%. A similar pattern is observed in the comparisons of the tensile strength by splitting and the modulus of elastic deformations.

The above applies with increasing importance with regard to water penetration depth and frost resistance - at 2 years of age **WS** water penetration depth is 7 mm, compared to 33 mm for **MC** concrete. All this explains the dramatically difference between two type of concrete concerning their frost resistance – 150 cycles for **MC**, and 250 cycles for **WS**.

Trying to emphasize the differences between **MC** and **WS** mixes a set of additional high-tech structural tests were performed by using direct physical methods:

- X-ray diffraction (XRD);
- Differential thermal analysis (DTA);
- Low temperature gas adsorption (BET-method);
- Scanning electronic microscopy (SEM).

The structural tests visualization is given on Fig. 1-18 and Photos 3-6.

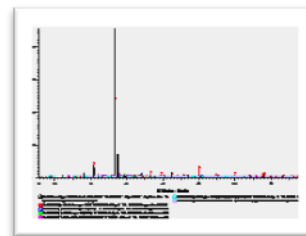


Fig. 1 X-ray MC – 7 days

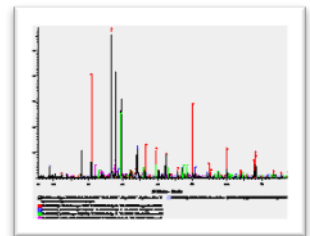


Fig. 2 X-ray WS – 7 days

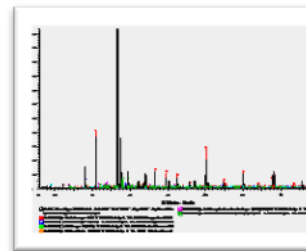


Fig. 3 X-ray MC – 28 days

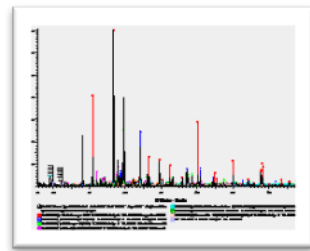


Fig. 4 X-ray WS – 28 days

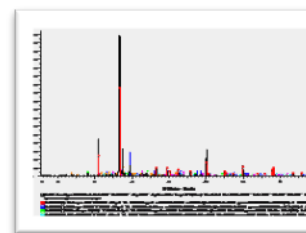


Fig. 5 X-ray MC – 2 years

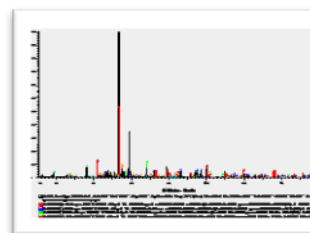


Fig. 6 X-ray WS – 2 years

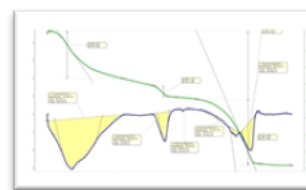


Fig. 7 DTA MC – 7 days

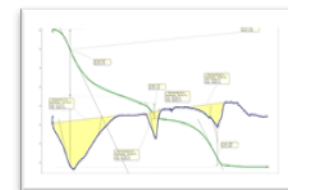


Fig. 8 DTA WS – 7 days

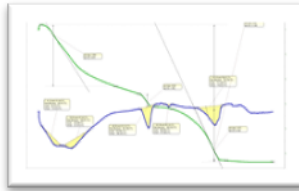


Fig. 9 DTA MC - 28 days

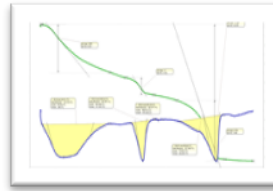


Fig. 10 DTA WS - 28 days

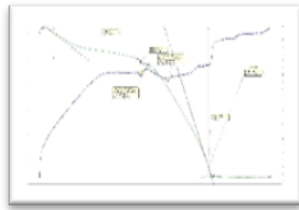


Fig. 11 DTA MC – 2 years

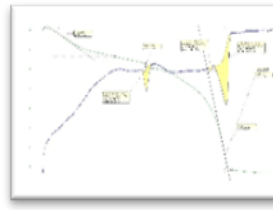


Fig. 12 DTA WS – 2 years

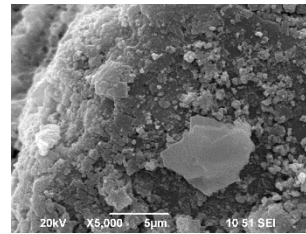


PHOTO 2 SEM x7500,
2 years - MC

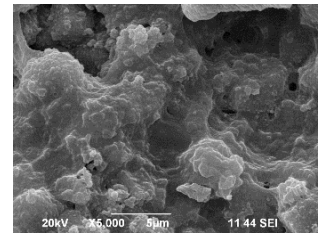


PHOTO 3 SEM x7500,
2 years - WS

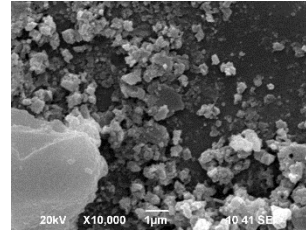


PHOTO 4 SEM x10000,
2 years - MC

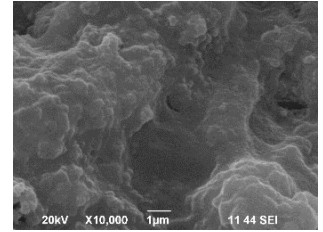


PHOTO 5 SEM x10000,
2 years – WS

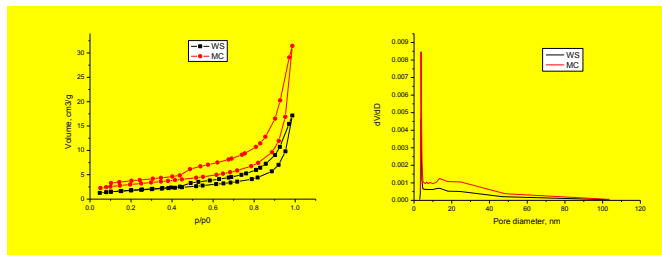


Fig. 13 BET Total porosity at 7 days of age

Fig. 14 BET Pore diameter size distribution at 7 days of age

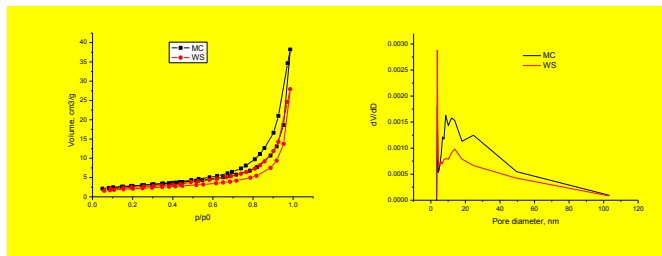


Fig. 15 BET Total porosity at 28 days of age

Fig. 16 BET Pore diameter size distribution at 28 days of age

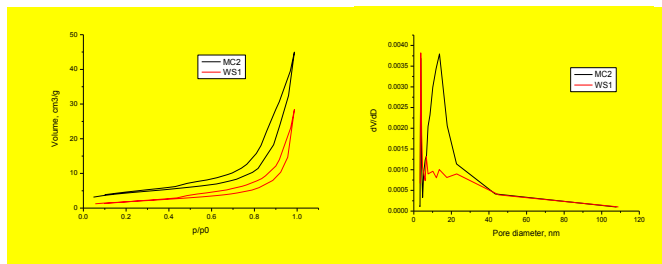


Fig. 17 BET Total porosity at 2 years of age

Fig. 18 BET Pore diameter size distribution at 2 years of age

The respective structural characteristics obtained by using professional complex analysis are given in Table 3. It is clear visible that basic WS hydrate products (C-S-H), leading to concrete strength-deformation characteristics increasing, dominated in comparison with MC ones. Concerning the pore structure parameters there are a lot of WS advantages – minor total porosity, minimal specific pore surface and mean pore diameter. All this explains the dominate mechanical characteristics (reported in Table 2.) of proposed WS shotcrete. Something more, the SEM photos additionally prove, that the WS total structural density is clearly observed.

4. General conclusion

Taking into account 2 years of aging the analysis of the experimentally found physical-mechanical and structural characteristics of the analyzed mixes for shotcreting yields the following **CONCLUSION**:

The physical-mechanical and structural characteristics of the proposed of the repair mix **WS** for "wet" shotcreting significantly surpass those of mixes used for the repair of silos for heavy soda **MC**.

Something more, the prolong time of ageing additionally emphasizes this well-grounded advantage.

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Acknowledgments

The financial support of the National Science Fund of Ministry of Education and Science, Bulgaria, contract KII - 06 - H 27/7, 2018, is gratefully acknowledged.

Application of progressive technologies in the restoration of functional areas of products

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Abstract: The paper presents the results of research focused on the restoration of functional areas of products by the application of PTA technology. Hardfacing layers intended for the renovation of the surfaces of aluminum alloy injection molds were evaluated. The base material was medium alloy steel X38CrMoV5-1 (H11). Three types of powder additives were applied to form the functional layers: two types based on iron, designated HSS 23 and HSS 30, and one type based on nickel, designated DEW Nibasit 625-P. The quality of the layers was evaluated using non-destructive and destructive tests. The surface integrity of the layers was assessed by visual and capillary tests. The microstructure of the materials and its hardness were evaluated. The tribological properties of the welds were determined by the pin-on-disc method.

Keywords: PLASMA WELDING, PTA TECHNOLOGY, MOLDS REPAIR, WEAR PROPERTIES

1. Introduction

To reduce CO₂ emissions and to implement electric mobility, it is necessary to use low weight technology on the effective weights of the vehicle and its components [1]. Injection molding and die casting are very high economic key technologies for the production of a complex of thin-walled components from plastics or light metals in large quantities [2]. Molds for tools have a significant impact on the economic efficiency of process production. About 5 - 15% of production costs can be allocated to the production of components.

Injection molds used in the automotive industry are often subjected to cyclic thermomechanical loading and are therefore subject to local damage and wear [3,5]. Mold repair is an economical alternative to the production of a new mold, especially for large and complex molds [6,8]. The repair process is usually a very demanding process performed manually by a qualified worker. Traditional repair methods such as tungsten inert gas (TIG), thermal spraying, high speed oxygen fuel (HVOF) or gas arc welding (GMAW) have inherent problems that can lead to thermal damage and deformation of the finished component [5]. Matrices and molds often have complex 3-D shapes and require very precise shaped deposition that must not generate any harmful residual stresses. Highly accurate and focused heat transfer is required during the mold repair process. Therefore, in order to maintain the properties of the base material, it is necessary to minimize contamination of the cladding with the base material and vice versa [9]. In this respect, a promising technique is laser deposition technology, such as laser cladding, characterized by local heating and rapid synthesis of materials. This is because the laser beam creates a relatively narrow zone of dilution and heat (HAZ). The quality and integrity of the resulting repaired component with the new layer is affected by a number of physical phenomena, such as melt morphology, microstructure development and residual stress formation [10,13].

X38CrMoV5 (H11) is a martensitic steel with a hardness in the range of 40–56 HRC depending on the heat treatment parameters. Satisfactory hardness is usually maintained up to a temperature of 600 °C. High mechanical strength and good resistance to thermal shocks are provided by the presence of vanadium, molybdenum and chromium [14]. The excellent properties of martensitic steels for high-temperature applications results from their complex microstructure obtained by special heat treatment. Carefully controlled austenitization, hardening and tempering. The mechanical properties of these martensitic steels are firmly linked to the complex microstructure. After heat treatment, which is usually performed in order to achieve very good hardness and tensile strength with sufficient ductility (> 12 J). Decreased dislocation density and coalescence of secondary carbides help to reduce the ultimate stress during tempering and fatigue [15].

The high-temperature flow stress of martensitic steels is related both to the extremely high dislocation density. These two factors are introduced during the martensitic transformation and to the alloy

carbides that precipitate at tempering [16,18]. These microstructural properties offer several ways to improve the high temperature mechanical properties by adjusting the heat treatment conditions. However, the dislocation density is fixed by hardening conditions. They strongly depend on the tool geometry, and a slight modification of the hardening conditions can lead to a catastrophic result. In addition, the mechanical properties show that the volume fraction of small precipitates (VC, Fe₃Mo₃C) directly affects the mechanical resistance at high temperatures, but has an adverse effect on the impact energy [19].

However, repairing molds with vanadium carbide metallurgical steel powders can potentially solve the existing problems of excessive damage / wear after repair. This is because vanadium carbide steels have an excellent combination of toughness, hardness and wear resistance [20].

2. Materials and Methods

Medium alloy tool steel marked X38CrMoV5-1 (W. Nr. 1.2343; HRC 50) was used as the base material to produce test hardfacing layers. It is characterized by high heat strength and resistance to tempering, as well as very good toughness and plastic properties at both normal and elevated temperatures. Furthermore, the steel shows very good resistance to thermal fatigue cracking and low sensitivity to sudden thermal shocks. It is well malleable when hot and well machinable in the soft annealed condition. The chemical composition of the base tool steel and its mechanical properties are presented in Table 1.

Table 1: Chemical composition of the base material X38CrMoV5-1 (wt. %).

C	Mn	Si	Cr	Mo	V	P	S	Fe
0.37	0.45	1.0	5.3	1.3	0.4	0.017	0.011	Bal.

As additive, the powder made of HSS 23 and HSS 30 was used, which are high-alloy high-speed steels produced using progressive methods of powder metallurgy. Both materials have high purity, low content of nonmetallic inclusions, high hardness, high compressive strength, toughness, machinability, and high resistance to abrasive wear. Both materials are used to make cold working materials, such as cutting tools for cutting harder materials. In addition, HSS 30 is also used in the production of cutting tools. The DEW Nibasit 625-P additive in powder form is used for high-quality hardfacing and hardfacing joints of materials with a high content of Mo, Ni, and Cr. Refractory and heat-resistant steels used for hardfacing are steels resistant to scale, operating at low temperatures, with heterogeneous joints, with low alloy content, and made of difficult-to-weld materials. The chemical composition of the powder filler materials used is presented in Table 2.

Table 2: Chemical composition of the powder filler materials HSS 23, HSS 30, and DEW Nibasit 625-P (in wt %), Fe Ball.

HSS 23									
C	Cr	Mo	W	V	Co	Si	Mn	Fe	Ni
1.28	4.2	5.0	6.4	3.1	-	-	-	Bal.	-
HSS 30									
C	Cr	Mo	W	V	Co	Si	Mn	Fe	Ni
1.28	4.2	5.0	6.4	3.1	8.5	-	-	Bal.	-
DEW Nibasit 625-P									
C	Cr	Mo	W	V	Co	Si	Mn	Fe	Ni
0.02 5	22	9	-	-	0.01	0.4	0.7	0.5	Bal.

For hardfacing, Vanadis HSS 23, Vanadis HSS 30, and DEW Nibasit 625-P powders were used on the base material X38CrMoV5-1, and a plasma hardfacing machine (Fig. 2 and Fig. 3) PPC 250 R6 (KSK s.r.o., Česká Třebová, Czech Republic). This automatic hardfacing machine is designed for the hardfacing of rotating and nonrotating parts using the PTA method. Components can be hardfaced on the forehead or around their perimeter. The machine enables pulse hardfacing with a current of 50–250 A and a frequency of 0–200 Hz.



Fig. 1 The plasma hardfacing machine PPC 250 R6 with plasma transferred arc (PTA) technology.

We applied additional material to the base material in two hardfacing layers using the hardfacing machine. Before proceeding with the hardfacing process itself, the surface of the base material was prepared properly. Then we preheated the base material to 250 °C and started the hardfacing process with the hardfacing parameters for individual layers shown in Table 4. Heat treatment was performed as follows: quenching from 1230 °C/2 min, followed by triple tempering at 550 °C/1 h.

Table 3: Hardfacing parameters for the first and the second layer.

First Layer	
Pulsating direct current:	110/45 A, with a frequency of 44.4 Hz
Trajectory:	swing step 3.9 mm (step speed 2.1 mm·s ⁻¹) swing length 80 mm (speed 3.1 mm·s ⁻¹)
Serving:	26.4 g·min ⁻¹
Second Layer	
Pulsating direct current:	140/45 A, with a frequency of 44.4 Hz
Trajectory:	swing step 5 mm (step speed 4.5 mm·s ⁻¹) swing length 80 mm (speed 3.3 mm·s ⁻¹)
Serving:	27.8 g·min ⁻¹

Weld-on layers were evaluated under dry friction adhesive wear conditions. (Adhesive wear according to EN 1071-13: Determination of wear by ball-on-disc method ISO 20808). High temperature tribometer by CSM Instruments, model 4.4.V, the friction coefficients of the evaluated weldments were determined by the ball-on-disc method.

Test conditions: temperature 23 °C, ball - counterpart Si3N4 with a diameter of 3 mm, radius of the circle 2.5 m, linear velocity of the ball 0.1 m·s⁻¹ on the track 500 m; normal load 20 N.

3. Results

The results of the hardface metallographic analysis are presented in Fig. 2, Fig. 3, Fig. 4. For the hardfacing layers HSS 23 and HSS 30, cracks are visible in the hardface, separating the hardface from the base material. The DEW Nibasit 625-P hardfacing layer is free of microscopically visible defects.

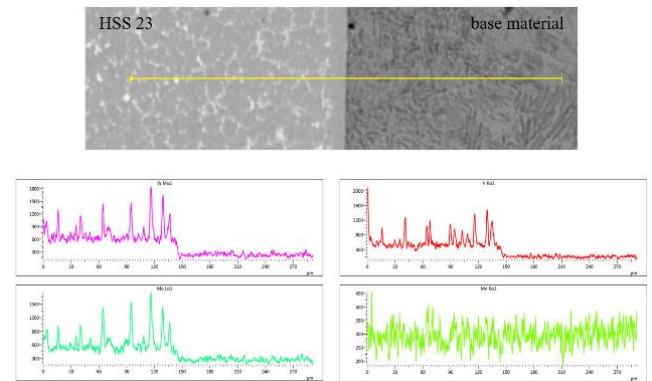


Fig. 2 Energy-dispersive X-ray spectroscopy (EDX) line analysis of the transition region between hardface HSS 23 and base material.

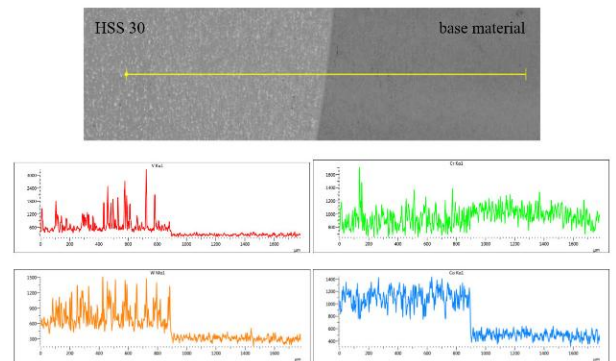


Fig. 3 EDX line analysis of the transition region between hardface HSS 30 and base material.

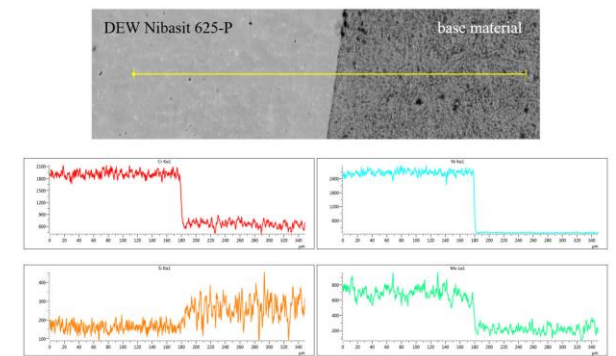


Fig. 4 EDX line analysis of the transition region between hardface DEW Nibasit 625-P and base material.

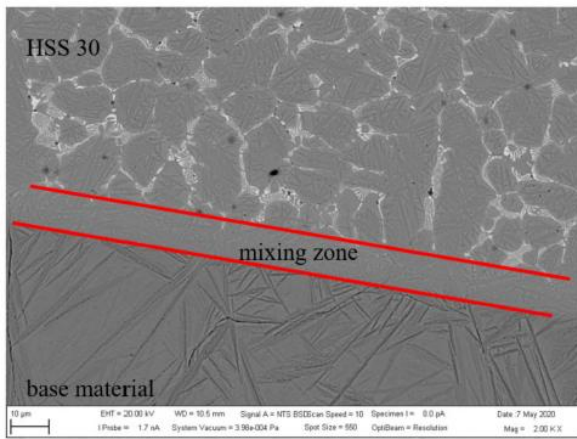


Fig. 5 HSS 30 welding layer on the base material, visible martensitic structure with missing eutectic phase in the mixing zone.

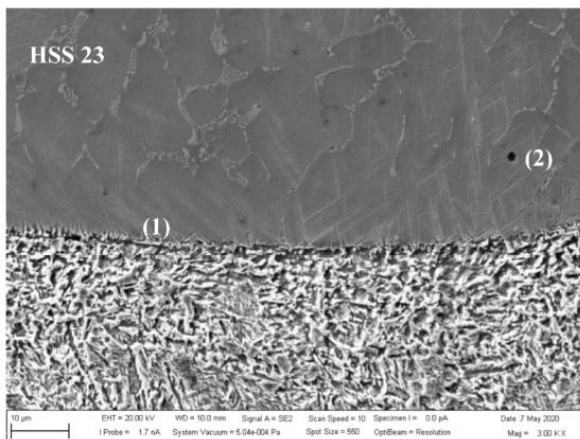


Fig. 6 Weld-on layer HSS 23 with base material.

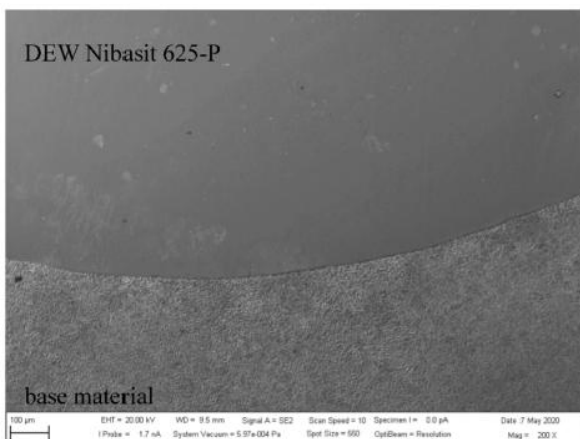


Fig. 7 Mixing zone - DEW Nibasit 625-P weld metal and base material.

The hardness of HSS 23 and HSS 30 hardfacing layers ranged from 650 to 850 HV 0.5. The materially different hardfacing layer DEW Nibasit 625-P reached the base material hardness level, i.e., 210–250 HV 0.5, Fig. 8.

The tribological and wear properties of the investigated materials are given in Table 4. The lowest value of the coefficient of friction at an ambient temperature of 20 ± 2 °C

was achieved by DEW Nibasit 625-P. The cover hardfacing layers made using the additive HSS 23 showed the highest average friction coefficient. The measured values for respective test samples correspond to the chemical composition of the evaluated covering layer surfaces of the assessed samples and to the base material of the casting die. Carbon content and content of the carbide-forming additives Cr, Mo, W, and V have a primary impact on friction coefficients.

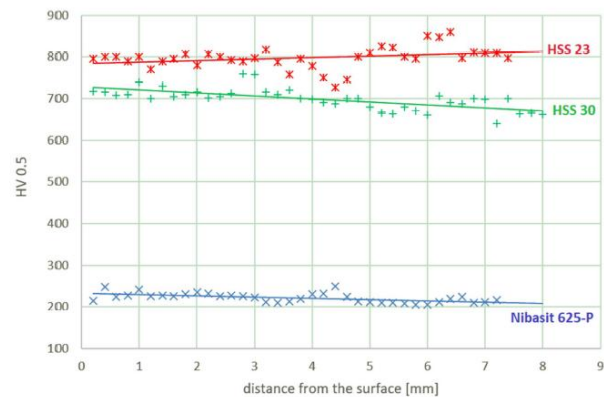


Fig. 8 Hardness comparison of individual hardfases.

Table 4: Tribological and wear properties of the investigated materials.

Experimental Materials	Sliding Speed (mm.s ⁻¹)	Normal Load (N)	Distance (m)	Coefficient of Friction	Wear Rate ×10 ⁻⁶ (mm ³ /m·N)
Base material	50	20	50	0.55 ± 0.09	33.96 ± 1.2
HSS 30	50	20	50	0.53 ± 0.04	7.6 ± 0.4
HSS 23	50	20	50	0.56 ± 0.12	14.49 ± 0.6
DEW Nibasit 625-P	50	20	50	0.35 ± 0.09	21.39 ± 0.8

4. Conclusions

The paper presents the results of research on the quality of functional layers made from the powder filler materials HSS 23, HSS 30, and DEW Nibasit 625-P by plasma transferred arc (PTA) hardfacing technology. Layers were made using the plasma hardfacing machine PPC 250 R6 on X38CrMoV5-1 base material. This type of medium alloy steel is used in the production of die-casting molds for aluminum parts for automotive production. The hardfacing was made in two layers. The quality of the hardfacing layers was evaluated using nondestructive and destructive tests. Surface integrity as well as the occurrence of surface defects were evaluated using visual testing (VT) according to the standard EN ISO 17637 and penetration testing (PT) according to the standard EN ISO 23277. Surface defects were not detected on the hardfacing layers. As part of the destructive testing, metallographic sections were made for observation by light microscopy and electron microscopy, as well as for the evaluation of the chemical composition of the surface spectra of selected parts of the samples. The occurrence of inner defects in the hardfacing layers was recorded on the cross sections. The defects were classified as rare pores. EN 12517-1 classifies this type of defect as permissible in this quantity. After metallographic analysis, the hardness in the lines passing through the hardface metal into the base material was evaluated on the sections. The maximum values of the hardness in the interval 720 HV 0.5–850 HV 0.5 were measured in the covering layers of the HSS 23 hardface metal, which is in accordance with their chemical composition and the observed martensitic structure.

The lowest hardness values were measured for DEW Nibasit 625-P samples (210 HV 0.5–250 HV 0.5). As part of the experiments, the tribological properties of the newly formed hardfacing layers were analyzed. The lowest friction coefficient was shown by surfaces with the DEW Nibasit 625-P layer and the highest values of the friction coefficient were measured on surfaces with the HSS 23 hardfacing layer. Based on the experiments performed, it can be stated that the selected energy beam (PTA) technology for the renovation of the functional surfaces of casting molds is suitable. As an additional material for the restoration of exposed mold areas, all investigated additional materials can be used, but due to its low friction coefficient, it is possible to recommend the nickel-based additive DEW Nibasit 625-P.

Acknowledge This work was supported by scientific grant agency of the Ministry of Education of the Slovak Republic VEGA No. 1/0497/20 and KEGA 001STU-4/2019.

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Energy saving crystalline phosphating

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Abstract: The object of the research was a low-temperature phosphating solution, in which the nitrate accelerator was replaced with a more environmentally friendly hydroxylamine. Research on the process of the low-temperature crystalline phosphating has been carried out, which allows to obtain fine-crystalline phosphate layers weighing $5.0\text{--}7.5\text{ g/m}^2$ with an increased protective ability up to 200 seconds, measured by the drop method using Akimov's reagent. At different temperatures, phosphate coatings of various thicknesses are formed, which can be used to form anti-corrosive layers for oiling with a specific gravity of 7.5 g/m^2 and adhesion layers for painting with a specific gravity of up to 5.0 g/m^2 , which corresponds to the recommendations for phosphating for ensuring good physical and mechanical properties and increasing the corrosion resistance of paint and varnish coatings.

KEYWORDS: LOW-TEMPERATURE PHOSPHATING, ANTI-CORROSION PROTECTION.

1. Introduction

Such valuable properties as high adhesive power to a metal base, adsorptive capacity, corrosion resistance, protective ability, low electrical conductivity, ease of production and relatively low cost allow wide application of phosphate coatings for corrosion protection of various metal surfaces [1]. Phosphate coatings due to significant harshness and porosity firmly adhere to the surface of the product and provide good adhesion of the organic coating to the surface of the product. Anti-corrosive phosphate coatings with a mass of at least 5.0 g/m^2 are treated with oils, while coatings with a mass of not more than 5.0 g/m^2 are allowed for coloring (for electrophoretic painting less than 3.5 g/m^2). The protective ability increases when the mass increases, however the relatively fragile phosphate film is destroyed under strong deformation, and the paint on top cracks and then peels off. A well-known disadvantage of solutions of anticorrosive crystalline phosphating is the high operating temperature of the processes, which causes high energy consumption [2]. Analysis of the relevant publications reveals that all known phosphating compositions contain derivatives of nitric or nitrous acids, which complicate cleaning of the wastewater [1-3].

At present, the requirements for the quality of phosphate coatings and technologies for their application have increased significantly. Meanwhile, such improvements as lowering the process temperature, reduction of the toxicity of solutions, and improving the quality of the resulting coatings are required [4].

2. Preconditions and means for resolving the problem

For the preparation of phosphating solutions, reagents of "pure", "chemically pure" qualifications and distilled water were used. The acidity of the solutions was controlled by the addition of a NaOH solution. As samples for the deposition of phosphate coatings, plates of cold-rolled low-carbon steel 08 were used.

The samples were degreased and activated before coating. Surface morphology was studied using a LEXT-OSL 4100 Olympus confocal microscope. The protective ability (PA) of phosphate coatings was determined by the drop method using a solution of the composition $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ 82 g/l; NaCl 33 g/l; 0.1N HCl 13 ml/l (Akimov's reagent). The criterion for assessing the quality of the coating was the time of the color change of the control area under the drop from blue to red-brown. The corrosion resistance of the surface of phosphated and painted with polyester powder paint Ecocolor PE RAL 9016 / P steel after treatment in the Ascott S120iP salt spray chamber was evaluated in accordance with [5] and the international standard ASTM B117 (SCAB test). The width of propagation of underfilm corrosion from the notch h (mm) was measured. The adhesion of polyester powder paint Ecocolor PE RAL 9016 / P was determined in accordance with the international standard ASTM D 3359 and [6] with a help of an Elcometer 107 adhesion tester using the lattice notch method.

3. Results and discussion

The choice of the main components of the low-temperature solution for the formation of crystalline phosphate coatings is a result of the analysis of numerous scientific developments [7-13].

The object of the research was a phosphating solution, in which the nitrate accelerator is replaced by a more environmentally friendly hydroxylamine, and the composition contains few components - layer-forming zinc ions and phosphoric acid anions. It was found that the phosphating process can be carried out at temperatures close to room temperature without deteriorating the characteristics of the coatings.

The main objectives of this work are: 1) to study the influence of the parameters of the phosphating process on the coatings characteristics, 2) to determine the optimal process conditions for obtaining anticorrosive crystalline coatings for subsequent oiling and coloring, 3) to develop a low-toxic low-temperature deposition process for phosphate coatings with improved protective characteristics.

It was found that at room temperature the maximum specific gravity of the phosphate film is formed in 15 minutes and is about 7.5 g/m^2 . The mass of metal etched away during phosphating changes insignificantly after 5 minutes of the process. A further increase in the duration of the process leads to a slight decrease in the thickness of the phosphate layer, but at the same time, apparently, the pores of the coating are filled with precipitated phosphates. The maximum value of the protective ability, measured by the drop method using Akimov's reagent (PA), is 160 seconds with a process duration of 25 minutes, which is the optimal phosphating time at a given temperature.

It was found that at a phosphating solution temperature of $50\text{ }^\circ\text{C}$, phosphate layers are formed in a shorter time. In this solution, a layer with a specific gravity of 5 g/m^2 with the highest protective ability of 180 seconds is formed in 5 minutes.

Figure 1 shows the characteristics of coatings formed at the optimum process duration for each temperature in the solution temperature range from 22 to $50\text{ }^\circ\text{C}$. It can be seen from the figure that at different temperatures phosphate films of various thicknesses are formed, which can be used to form anticorrosive layers for oiling with a specific gravity of 7.5 g/m^2 and adhesion layers for painting with a specific gravity of up to 5.0 g/m^2 . Such masses comply with the recommendations for phosphating, including ensuring good physical and mechanical properties and increasing the corrosion resistance of paint and varnish coatings [14].

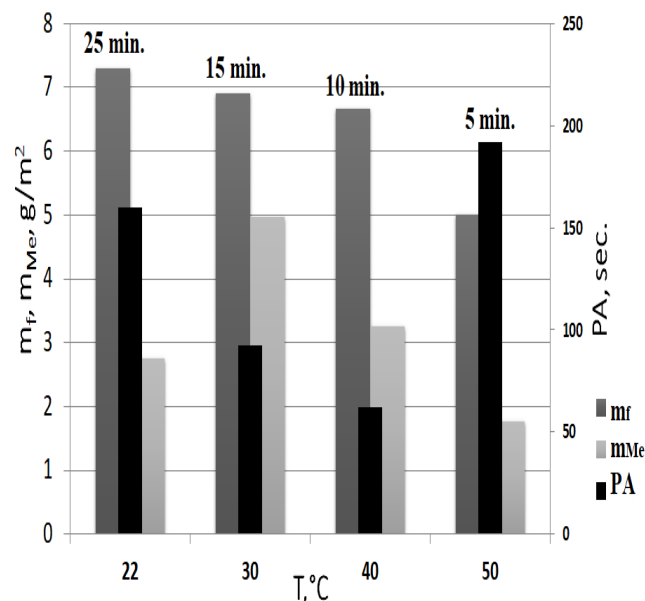


Fig. 1. Dependence of m_{ph} , m_{Me} and PA on the temperature of the phosphating solution at the optimal duration of the process

Thus, a phosphate film suitable for oiling is formed on the steel in the test solution at room temperature. The anticorrosive phosphate layers formed in the optimal mode were treated with I-20A straw oil. It was found that the PA of oil-treated samples increased up to 200 seconds, oil absorption is about 4 g/m².

Examination of the surface of the samples using a microscope have revealed an obvious difference between the coatings formed in solutions of different operating temperatures. It is confirmed that the film on a sample formed in a low-temperature solution is more coarse-crystalline and harsher, while the film on the sample formed at 50 °C has smaller crystals and is less harsh.

Corrosion tests carried out according to [15] showed that the investigated phosphate coatings do not corrode for 15 minutes in a sodium chloride solution, and oiled phosphate coatings formed in a low-temperature solution do not corrode for 2 hours. Thus, the layers formed in the test solution at room temperature can be used as independent anticorrosive coatings.

Corrosion tests of painted adhesive phosphate coated steel samples in a salt spray chamber for 240 hours showed that the phosphate layers deposited at 50 °C met the requirements of ASTM B117. It was found that painted phosphate coatings have exceptionally good adhesion (according to ASTM D3359) - class 0, which have not deteriorated even after corrosion tests.

4. Conclusion

As a result of the conducted studies of the process of low-temperature crystalline phosphating, we have obtained phosphate layers weighing 5.0-7.5 g/m² with an increased protective ability. The optimal conditions for the process of obtaining crystalline coatings for subsequent oiling and coloring have been determined.

The use of the developed process of low-temperature phosphating will reduce the energy costs of production, increase the protective properties of phosphate coatings, and reduce the toxicity of phosphating solutions.

«The work was carried out with the financial support of the Mendelev University of Chemical Technology. Project number X-2020-028».

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Efficiency of processing of recycle from porous PVC

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Abstract: The paper presents research on the physical processing properties and morphology of recycled plastics. The scope of work includes conducting the processing of primary PVC and recycled PVC and testing Melt Flow Index and structural investigation of the morphology of materials. In the studies polyvinyl chloride (PVC) Alfaviny GFM/4-31, was used in the study. This plastic has density of 1230 kg/m³ and hardness of 80 Sh° A. In the experiments, various types of chemical blowing agents were used: Expancel 950 MB80 manufactured by Akzo Nobel. Expancel is a mixture that contains 65 % microspheres in the copolymer of ethylene and vinyl acetate (EVA). The decomposition products of the applied blowing agents mainly include carbon dioxide CO₂, a small amount of water H₂O and nitrogen N₂. The technology for producing the recycled composition is based on the extrusion and compression technology of the compositions obtained. The study of selected physical properties was produced. The research on the structure of manufactured materials, melt flow index MFI, and macroscopic structure are presented.

Keywords: PVC, CHEMICAL BLOWING AGENT, RECYCLED PVC, MFI, MICROSTRUCTURE

1. Introduction

A growing interest in cellular plastics and the methods of their manufacture has led to the development of a new processing method of cellular extrusion and injection molding [1-5]. Cellular extrusion consists in introducing into a polymer plastic a blowing agent, which, under appropriate pressure and temperature conditions, decomposes, emitting gas [1, 3, 4, 6-8]. This gas dissolves in the polymer. Next, after the pressure drops, the gas begins to evolve from the polymer producing cellular structure, which has to be solidified by cooling the cellular product [8 -12]. A blowing agent (porophor) can be gaseous, solid or liquid. Gases and liquids are introduced into the input polymer under appropriate conditions – under pressure, using special devices which deliver them to the feed section of the plasticating system. Blowing agents used in the extrusion process can be divided into physical and chemical, however this classification is traditional and not exactly accurate [1, 3, 11-18]. Physical blowing agents (physical porophors) comprise substances which do not change their chemical structure during the extrusion process; they only change their state of aggregation [4, 5, 6, 12, 17-23]. Chemical blowing agents (chemical porophors) act similarly as physical porophors, yet gaseous products generated as a result of their decomposition and causing the cellular extrusion effect, remain inside the plastic material. Inert gases and low-boiling liquids are introduced under pressure by means of special dispensing devices directly into the feeding zone of the plasticizing unit of the extrusion machines during the extrusion process. The emerging cells may be filled with air or with any other gases, for instance CO₂ and N₂, yet later they are replaced by air as a result of diffusion [1, 3, 5, 7, 8, 24-29]. Research on the cellular extrusion of thermoplastics focuses on the process where product properties are modified by changing the conditions of extrusion and the properties of the processing line structural elements. The extrusion of products with the use of blowing agents results in new, modified physical and technological properties of cellular products.

The aim of the study is to analyze the physical processing properties and morphology of recycled plastics. The scope of work includes conducting the processing of primary PVC and recycled PVC. The scope of work also includes shrinkage testing of primary compacts and structural investigation of the morphology of materials.

2. Materials and methods

In the studies polyvinyl chloride (PVC) manufactured by Alfa sp.z.o.o. (Poland), Alfaviny GFM/4-31, was used in the study on cellular extrusion process. This plastic has density of 1230 kg/m³ and hardness of 80 Sh° A. PVC foams are distinguished from most other types of plastic foams by a broad range of unique characteristics. In the experiments described in the present paper, one type of blowing agents were used. Chemical blowing agents are

modifiers that generate gases through chemical reactions at elevated temperatures and the decomposition of organic or non-organic bonds, in effect producing a cellular structure due to affinity for polymer. In the experiments, various types of chemical blowing agents were used: Expancel 950 MB80 manufactured by Akzo Nobel. Expancel 950 MB80 is a blowing agent that has the form of spherical thermoplastic polymer capsules (microspheres) that contain a hydrocarbon gas. This is an endothermic blowing agent. Expancel microspheres do not bond because the capsules retain their blocking properties, which prevents release of the constrained gas. Expancel 950 MB 80 is a mixture that contains 65 % microspheres in the copolymer of ethylene and vinyl acetate (EVA). The decomposition products of the applied blowing agents mainly include carbon dioxide CO₂, a small amount of water H₂O and nitrogen N₂.

The polymer processing was carried out with the use of a single-screw extruder, type W-25 with the screw diameter of D=25 mm. The plasticizing unit was equipped with four heating zones. The process line was composed of the extrusion coating head, vacuum calibrator, the cooling bath and the remaining process line components.



Fig. 1 View of the extruded rod and fragments of the produced samples.

The process of grinding previously extruded samples was carried out on the Xiecheng XC-GP230 mill. It is a mill specially designed for grinding plastics materials with a capacity of 4 kW.



Fig. 2 Appearance of the shredder knives and PVC recycle.

The process of secondary extrusion of shredded PVC was carried out on the EHP 2x20 Sline twin screw extruder, the manufacturer of which is Zamak Mercator.

The extrusion line consists of the following elements: co-rotating extruder EHP 2x20 Sline, single-screw volumetric feeder, twin-screw volumetric feeder, extruder head, cooling bath, digital line control system.

Based on the adopted research program, the polymeric material for polymer processing molding modified in such a way that the blowing agents were fed into it during the mechanical mixing process.. The blowing agents used in the polymer processing were fed into the polymers being processed in the following quantities: 0.5%, 1.0% and 2.0% by mass (w/w). In order to obtain high process efficiency for the above blowing agents, the processing temperature range was 170-210°C.

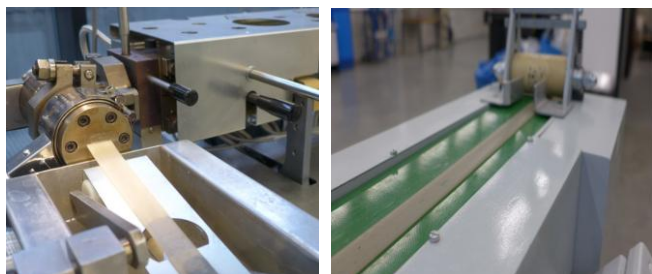


Fig. 3 View of technological line section for recycled plastics.

They are usually fed into the plasticizing unit using gravimetric or volumetric feeders. The polymer and blowing agent mixture then undergoes processing in the plasticizing unit and forming. In order to produce a favourable structure with small cells, the product should be cooled and solidified as quickly as possible.

3. Results and Discussion

The Melt Flow Index (MFI) is a measure of the ease of flow of the melt of a thermoplastic polymer. It is defined as the mass of polymer, in grams, flowing in ten minutes through a capillary of a specific diameter and length by a pressure applied via prescribed alternative gravimetric weights for alternative prescribed temperatures. The method is described in the similar standards ASTM D1238 and ISO 1133. MFI tests were carried out on the test stand equipped with a Ceast weight plastometer type 6542.00 with additional equipment and a laboratory balance.



Fig. 4 View of MFI instrument, Ceast 6542.0 and selected measurement samples

The result of the measurements carried out was the receipt of the MFI mass flow rate value for a PVC extrudate modified with a blowing agent. The range of obtained readings is from 2.27 g/10 min at 0% mass content of the blowing agent to 4.55 g / 10 min at 1.5% mass content of the blowing agent. For the MFI secondary tape test, results were found in the range of 0.64 g / 10 min at 0% mass content of the blowing agent to 1.70 g/10min at 1.0% mass content of the blowing agent in recycled material. The value of average values for individual materials with an indication of the content of the blowing agent was: 0.68; 1.28; 1.67; 1.37. The relationship of these values is presented in the chart below.

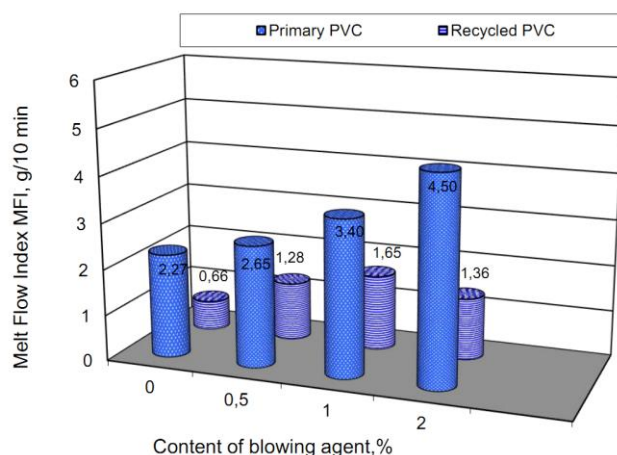


Fig. 5 Block diagrams illustrating the dependence of MFI on the blowing agent type and its content in primary and recycled PVC.

The investigation and analysis of the porous structure of the produced parts were conducted using a confocal microscope, type Nikon Eclipse LV100ND and copyright position of image analysis of porous structure and the author's stand for porous structure image analysis. The morphology shows are porous in whole cross section area. There is no solid outer layer visible, as in the case of injection molded samples. Obtained pores have different size and shape.

Microscopic studies show the effect of secondary extrusion on the structure of porous poly (vinyl chloride) depending on the amount of blowing agent added, which was added in primary extrusion in the amounts: 0%, 0.5%, 1.0% and 2.0% by mass in relation to the weight of the processed material.

Based on the obtained microscopic images, we can observe that after re-extrusion of porous PVC, pores still occur in the structure of the material, but their size has decreased and the quantity has decreased to such an extent that the extrudate no longer has a porous structure. The change in the number of pores and their surface quantity in the cross section of the molded parts can also depend on cooling intensity. Fast cooling hampers the occurrence and growth of the pores, especially of those located closer to the surface layer (Figs. 6-9).

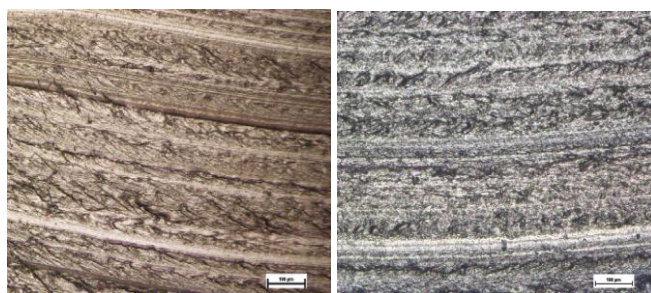


Fig. 6 Fragment of the cross section of the PVC and recycled PVC without microspheres.

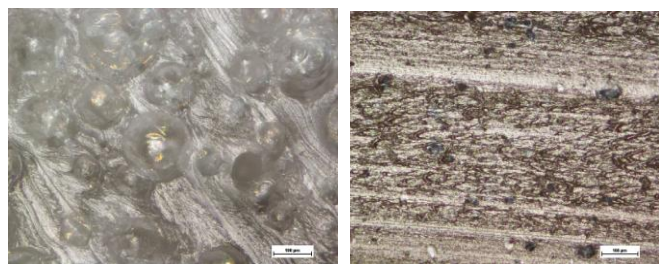


Fig. 7 Fragment of the cross section of the PVC and recycled PVC with 0.5% microspheres.

In the case of the porous parts produced using the blowing agent with the endothermic decomposition characteristics, the gas release in the course of processing comes to an end once the energy supply

is stopped. The obtained porous structure is uniform; the pores have a spherical or quasi spherical shape. The pores have similar sizes, irrespective of their location in the product.

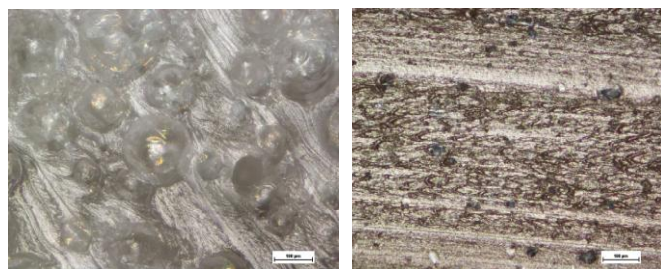


Fig. 8 Fragment of the cross section of the PVC and recycled PVC with 1.0% microspheres.

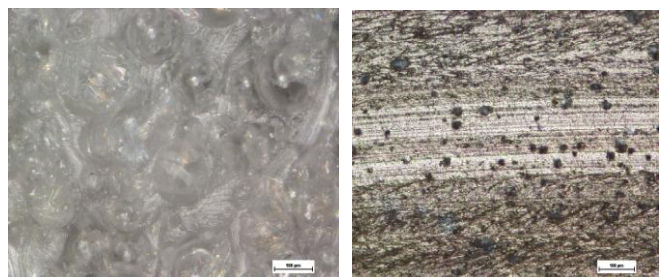


Fig. 9 Fragment of the cross section of the PVC and recycled PVC with 2.0% microspheres.

The difference between the structure of primary PVC and recycled PVC, where a blowing agent was added during the porous processing, is significant. Secondary extruded poly (vinyl chloride) samples have a solid structure with single pores appearing in sizes much smaller than in the case of primary samples. It is noticeable that the number of pores of the secondary samples depends on the amount of blowing agent added during the primary extrusion process. Namely, the smallest number of pores occurs when the blowing agent in the primary extrusion was added in an amount of 0.5% by mass based on the weight of the processed material, and the largest number of pores occurs when the blowing agent was added in the amount of 2.0% by weight based on the weight recycled plastic.

4. Conclusions

Appropriate selection of methods and technological lines allows the processing of secondary plastics with high efficiency and good quality of secondary products. The purpose of secondary plastics processing is to obtain products with repeatable technological properties. After testing for selected strength indicators, it results that in the case of PVC without the content of blowing agent, its value significantly decreases, for secondary plastics, compared to primary plastics.

The porous structure is an advantage of plastics parts produced in the cellular processing, as it results in a decreased amount of the polymeric composite needed in their production. Owing to the use of chemical blowing agents, porous parts have, among others, lower weight, enhanced damping properties, and lower processing shrinkage. The cellularity determines the gaseous phase content in a cellular product, determining at the same time the density decrease value of this product. Such content of the blowing agent in the polymer determines both the coating continuity over the whole cross section and the uniform distribution of pores and their similar sizes.

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Study of temperature changes in ceramic cavity walls of beehives

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Abstract: Subject of the present paper is the measurement of temperature changes in the ceramic cavity walls of Dadant Blatt beehives. Using a FLIR P640 infrared camera and two different heat sources, 80 thermograms of ceramic cavity walls were obtained. The infrared images of the studied objects are divided into rectangular zones. The results of the study are presented tabularly and graphically.

Keywords: CERAMIC CAVITY WALL, INFRARED CAMERA, INFRARED IMAGES.

1. Introduction

More than 80 infrared images were obtained at a temperature reading interval of 2 minutes using an infrared camera brand FLIR P640 [1], placed at a distance of 1m from the studied objects. The emissivity ϵ was selected based on paint catalog data, since the studied parts (hive and radiator walls) is applied a layer of paint. The selected value is $\epsilon = 0.96$ [2]. The study was performed in two stages using two different heat sources - an oil radiator at the first stage and a plate at the second stage.

The highest ground surface temperatures recorded on Earth are usually in the range from 50° to 70°C, in some desert areas up to 80°C. For certain soils, the ground surface temperature can reach 90°C. The highest measured temperatures are 57.8°C for the air and 93.9°C for the ground surface [3, 4]. The highest air temperature measured in Bulgaria is 45.2°C [5].

2. Experimental part and discussion

Stage 1

At stage 1, an oil radiator preheated to an average temperature of 161.5°C was used as the heat source (Fig. 1). The recorded temperature of the radiator is in its area of contact with the ceramic cavity walls of the beehive. This zone is marked in Fig.1 with Bx1 (the designation Bx is an abbreviation of Box).

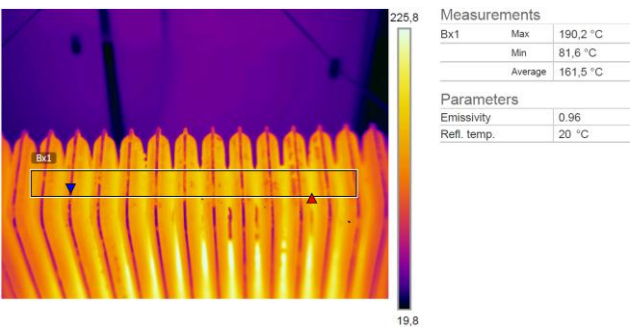


Fig. 1. Heat source – an oil radiator during the examination of a ceramic cavity wall.

Once the temperature of the radiator was set, the object under examination—a ceramic cavity wall of a beehive – was positioned on top of it. When compiling the thermograms, the infrared images were divided into several rectangular zones designated as Bx1, Bx2,...,Bxn. Thermograms were obtained and a thermographic analysis of a total of 15 infrared images was performed. The initially measured temperatures of the test object are shown in Fig. 2. The highest temperatures, which were recorded at the edges of the object, are marked with red triangles. The minimum temperatures are marked with inverted blue triangles. The average temperature of all zones is 22.55°C.

As the temperature rises, areas with uneven temperature distribution begin to appear. In Fig. 3 is shown a thermogram from the 14th minute of the experiment. As can be seen, in zones Bx1 and Bx8 are observed sections that are colder than the other zones. Compared to zone Bx 5, their average temperatures are lower by 1.7°C for Bx1 and 2.1°C for Bx8. A visual inspection shows a change in the upper surface of the beehive wall zone Bx5, where a section higher than the rest of the wall can be observed.

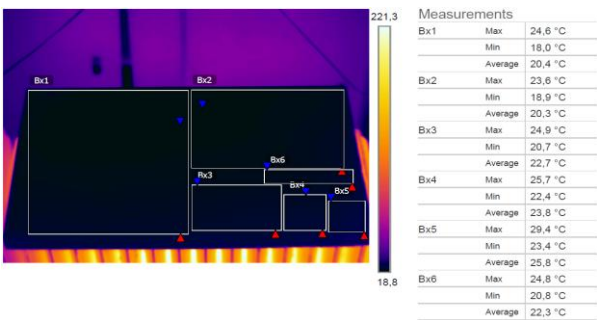


Fig. 2. Initially measured temperatures on the wall of the beehive.



Fig. 3. Temperature distribution on the wall of the beehive after the 14th minute of the experiment.

At the end of the experiment, colder zones continued to be visible in the area on the left side of the studied object, as shown in Fig. 4. After removing the beehive wall from the radiator, another measurement was taken of the average radiator temperature. An increase in its average temperature by 13°C (174.5°C) was observed, as shown in Fig. 5.

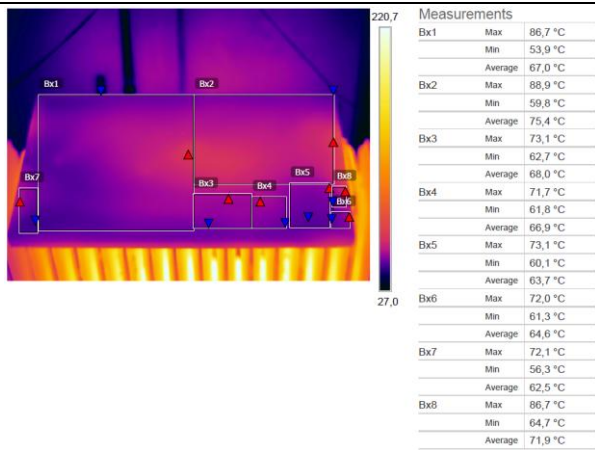


Fig. 4. Last thermogram obtained at the end of the first stage of the study of the ceramic cavity wall.

TEMPERATURE IN TIME

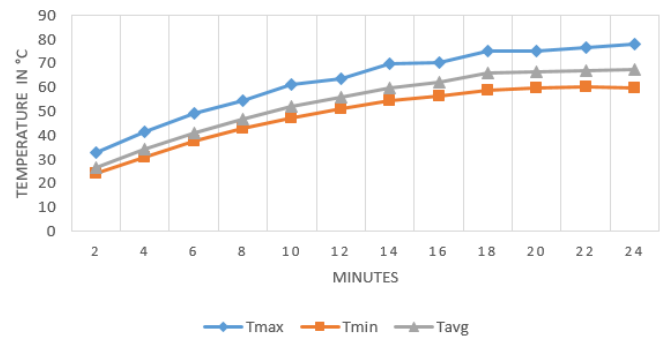


Fig. 6. Change of the temperature of the ceramic cavity wall as a function of time.

The study was repeated with the same parameters using another detail from the beehive – its front wall with a clear opening in the middle, as shown in Fig. 7.



Fig. 7. Detail from the beehive – front wall with a clear opening.

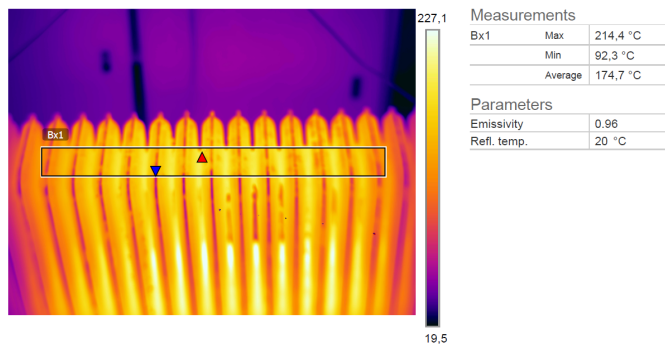


Fig. 5. Average temperature of the oil radiator reported after removal of the ceramic cavity

The experimental data are shown in Table 1, where Tmax is the maximum average temperature, Tmin is the minimum average temperature, and Tavg is the average temperature of all zones of the hive wall. The graph compiled from the data in the table is shown in Fig. 6.

Table 1. Temperature data from the study of ceramic cavity wall.

No	Time [min]	Tmax [°C]	Tmin [°C]	Tavg [°C]
1	2	32,8	24,5	26,9
2	4	41,7	31	34,2
3	6	49,1	37,7	41,3
4	8	54,8	42,9	47
5	10	61,4	47,5	52,1
6	12	63,7	51	56,3
7	14	69,8	54,4	59,9
8	16	70,7	56,4	62,3
9	18	75,2	59	66,2
10	20	75,3	59,9	66,4
11	22	76,6	60,4	67,3
12	24	78	60,1	67,5

The heat source is preheated to an average temperature of 169.5°C (Fig. 8). The recorded temperature of the radiator is in its area of contact with the wall of the beehive. The zone is marked on the image as Bx1.

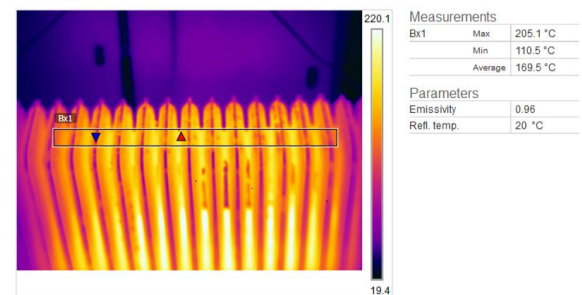


Fig. 8. Heat source – oil radiator, when examining a front wall with a clear opening.

In the thermograms of the images from the second case, the same temperature scale is set (from 24°C to 140°C). It is positioned on the right in Fig. 9. The fixed values on the scale are for the purpose of easier registration of zones with different temperatures. No visible defects (warmer areas) are observed.

In Fig. 9 is shown the temperature distribution at the initial moment – immediately after the studied part has been positioned on the heat source. The temperature of the clear opening, which is marked as zone Bx6, has also been measured.

In Table 2 has 3 additional columns recording the data from the clear opening temperature measurement. The angle at which the infrared images are taken does not register the heat source, yet the reported values for zone Bx6 raise the total temperature of the studied part in the range from 0° to 8°C. The graph based on this data in Table 2 is shown in Fig. 10. It registers elevated temperatures when the maximum and average values of the temperature in the clear opening zone are read. There is no significant difference in the values of the reported minimum temperatures in the clear opening zone, as the zones around the clear opening influence the measurement results. The change in the temperature values as a function of time for zone Bx6 are shown in Table 3. Fig. 11 shows the curve constructed based on the data in the table.

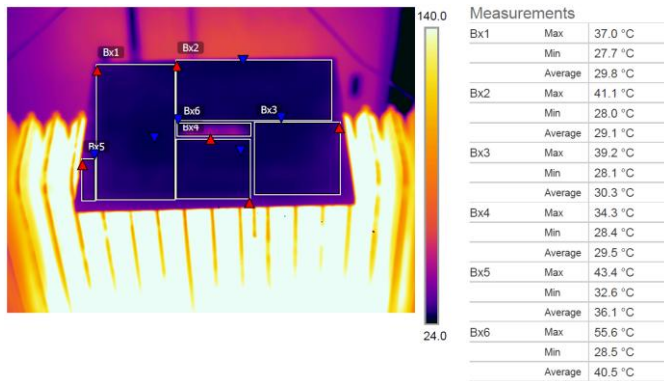


Fig. 9. Initial temperature distribution on a front wall with a clear opening.

Table 2. Temperature data from the study of a front wall with a clear opening.

No	Time [min]	Tmax [°C]	Tmin [°C]	Tavg [°C]	Tmax + Bx6 [°C]	Tmin + Bx6 [°C]	Tavg + Bx6 [°C]
1	0	39	28,9	31	41,8	28,9	32,3
2	2	49,6	35,2	38,7	54,1	35,7	41,6
3	4	58,5	41,7	46,8	64,2	42,6	50,3
4	6	69	47,6	54,3	75,1	49,1	58,2
5	8	76,5	52,8	60,4	83,1	54,6	64,8
6	10	79,2	55,9	64	86,5	58,3	69
7	12	82,8	58,7	67,2	90,3	61,4	72
8	14	85,5	60,9	63,9	93,2	63,8	70,2
9	16	86,7	62,7	72	94,8	65,5	76,8
10	18	89,8	64	73,5	97,8	67,3	78,5

Table 3. Temperature data from the study of zone Bx6.

No	Time [min]	Tmax Bx6 [°C]	Tmin Bx6 [°C]	Tavg Bx6 [°C]
1	0	55,6	28,5	40,5
2	2	76,6	38	55,9
3	4	92,9	47,1	67,8
4	6	105,3	56,5	78,1
5	8	116,3	63,7	86,8
6	10	123,1	70	93,7
7	12	128,1	74,9	95,9
8	14	132	78,4	101,5
9	16	135,3	79,1	100,8
10	18	137,5	83,8	103,4

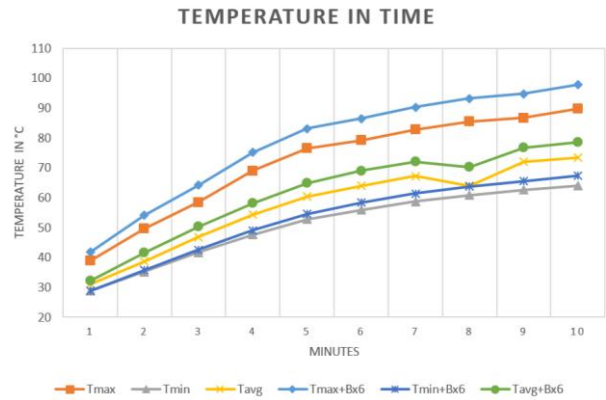


Fig. 10. Dependence on time of the temperature of the front wall with a clear opening.

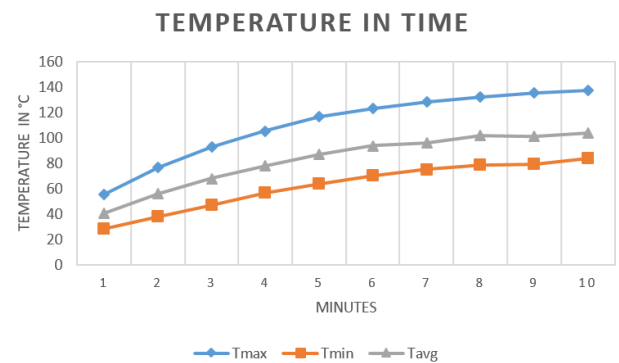


Fig. 11. Dependence on time of the temperature of the front wall clear opening.

Stage 2

In stage 2 was used a heater, preheated to an average temperature of 52°C, with a maximum reading in some areas of 63°C. The area of the heater (shown in Fig. 12) is 0.09 m². The heated part has a measured area of 0.10 m², and on its right-hand side there is an area of 0.01 m² (zone Bx11) which does not come into direct contact with the heater. This is the reason why lower temperatures are observed in zone Bx11. After obtaining the thermograms, the resulting data is presented in two tables. Table 4 shows the maximum, minimum and average temperature for the area of the beehive wall in contact with the heater. Table 5 shows the maximum, minimum and average temperature in zone Bx11.

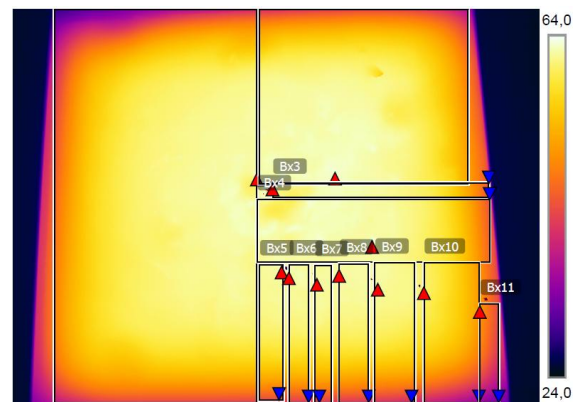


Fig. 12. Heater, preheated to an average temperature of 52°C.

The difference in temperatures is visible, starting from the second minute of the experiment as shown in Fig. 13, where the difference is small (below 1°C). At the end of the test, the difference reaches 9.5°C, as shown in Fig. 14. The scale of the

thermograms is set in the range from 24° to 64°C. At the 8th minute of the study a defect (a warmer section), appears on the wall of the beehive and at the 16th minute it is already clearly expressed, with the presence of a crack, as shown with a line contour on Fig. 15.

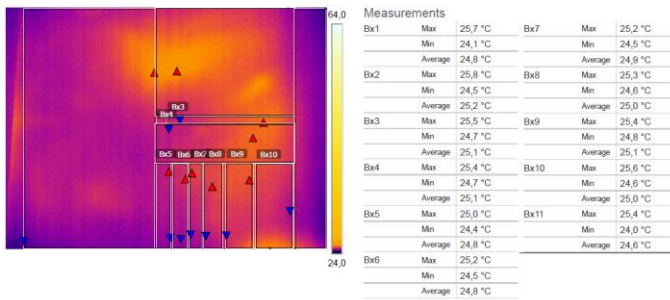


Fig. 13. Thermogram for the 2nd minute of the study.

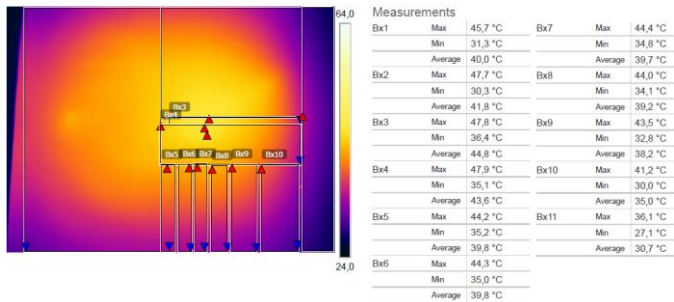


Fig. 14. Thermogram for the last 98th minute of the test.



Fig. 15. Presence of a defect (warmer section) on the wall of the beehive.

Table 4. Data with temperatures obtained in the study for the area of the wall of the beehive contacting with the heater.

Nº	Time [min]	Tmax [°C]	Tmin [°C]	Tavg [°C]
1	0	25,1	24,5	24,8
2	2	25,4	24,5	25
3	4	27,2	25,1	26,2
4	6	29,4	26	27,9
5	8	31,5	26,6	29,6
6	10	33,6	27,4	31,1
7	12	35,4	28,3	32,6
8	14	37	29	33,8
9	16	38,2	29,8	34,9
10	18	39,4	30,2	35,8
11	20	40,6	31,2	36,9
12	22	41,2	31,2	37,3
13	24	41,8	31,5	37,6
14	26	42,4	31,8	38,1
15	28	42,9	32,2	38,6
16	30	43,1	32,2	38,7

17	32	43,4	32,3	38,9
18	34	43,6	32,5	39,1
19	36	44	32,7	39,4
20	38	44,1	32,9	39,5
21	40	44,1	32,8	39,5
22	42	44	32,8	39,4
23	44	44,1	33	39,5
24	46	44,3	33	39,7
25	48	44,4	32,7	39,7
26	50	44,6	33,1	39,8
27	52	44,7	32,9	39,9
28	54	44,7	33,2	39,9
29	56	44,7	33,2	39,9
30	58	44,8	33,3	40
31	60	44,8	33,3	40
32	62	44,8	33,3	39,9
33	64	44,8	33,2	39,9
34	66	44,8	33,2	39,9
35	68	44,8	33,2	39,9
36	70	44,9	33,2	40
37	72	45	33,2	40
38	74	44,9	33,2	40
39	76	44,7	33,2	39,9
40	78	44,6	33,1	39,8
41	80	44,6	33,1	39,8
42	82	44,6	33,3	39,9
43	84	44,7	33,3	40
44	86	44,8	33,2	40
45	88	45	33,3	40,1
46	90	45	33,3	40,1
47	92	44,9	33,2	40
48	94	45	33,4	40,1
49	96	45,1	33,4	40,2
50	98	45,1	33,5	40,2

Table 5. Data with temperatures obtained in the study for zone Bx11 of the wall of the beehive.

Nº	Time [min]	Tmax Bx11 [°C]	Tmin Bx11 [°C]	Tavg Bx11 [°C]
1	0	25,5	24,2	24,6
2	2	25,4	24	24,6
3	4	26,2	24,1	24,9
4	6	27,4	24,2	25,5
5	8	28,5	24,4	26
6	10	29,7	24,3	26,5
7	12	30,8	24,6	27,1
8	14	31,7	24,8	27,7
9	16	32,7	25,2	28,3
10	18	33	25,4	28,6
11	20	34,1	25,9	29,4
12	22	33,9	25,9	29,2
13	24	34,4	25,9	29,4
14	26	34,5	26	29,6
15	28	35	26,2	29,9
16	30	35,1	26,3	30
17	32	35	26,4	30
18	34	35,3	26,5	30,2
19	36	35,8	26,6	30,4
20	38	36	26,6	30,5
21	40	35,5	26,6	30,4
22	42	35,4	26,6	30,3
23	44	36	26,8	30,6
24	46	36	26,7	30,6
25	48	35,6	26,8	30,5
26	50	35,8	26,7	30,6
27	52	35,8	26,8	30,6
28	54	36	27	30,6

29	56	35,8	26,9	30,5
30	58	35,8	27,1	30,6
31	60	36	26,9	30,6
32	62	35,7	27	30,5
33	64	36,2	27	30,7
34	66	35,8	27	30,6
35	68	35,8	27	30,7
36	70	35,6	27,1	30,6
37	72	36	27,1	30,7
38	74	35,8	27,1	30,6
39	76	36,2	27,2	30,8
40	78	35,8	27	30,6
41	80	35,7	27,1	30,6
42	82	36	27,1	30,8
43	84	35,9	27,2	30,7
44	86	35,8	27,1	30,7
45	88	35,8	27,2	30,7
46	90	35,9	27,1	30,7
47	92	35,8	27,1	30,7
48	94	36	27,1	30,7
49	96	36,1	27,1	30,7
50	98	36,1	27,1	30,7

Two graphs are constructed based on the data included in the tables. The graph shown in Fig. 16 uses the data in Table 4. There is an exponential increase in the temperature from the beginning up to the 12th minute of the study. After that, the rate of increase slows down until the 20th minute, when there begins a smooth rise with minimal differences of the order of tenths of a degree. From the 36th to the 98th minute, the temperature rises by 1°C. The graph in Fig. 17, which based on the data from Table 5, also shows an exponential increase in the initial minutes. The temperature rise then follows a decreasing rate, which is due to the absence of a direct heat source under zone Bx11.

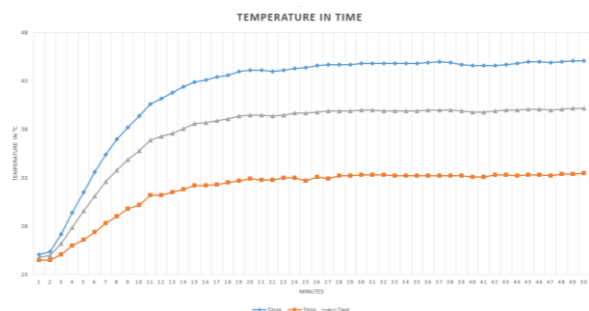


Fig. 16. Dependence on time of the temperatures of the area of the wall of the beehive, which is in contact with the heater.

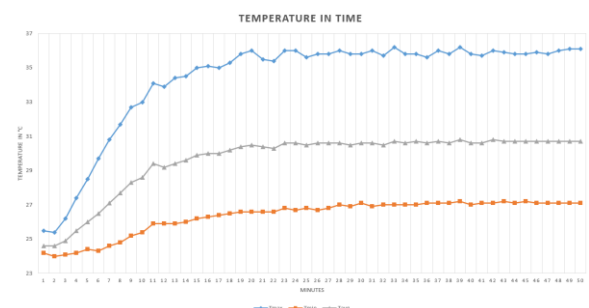


Fig. 17. Dependence on time of the temperatures of the area of the wall of the beehive without direct contact with the heater (zone Bx11).

3. Conclusions

The thermographic analyses of the two stages of the study point to the thermal insulating properties of the two beehive ceramic cavity walls. At stage 1, the set temperatures were higher than those reached under normal atmospheric conditions. A defect (an area with higher temperature) was observed. At stage 2, the set temperatures were close to the actual ones reached and recorded on Earth. At this stage, a defect was also recorded. During the measurement, a temperature difference of 11.8°C was observed between the heater and the average temperature of the upper surface of the beehive wall, as reached within 58 minutes. After the 58th minute, the average temperature fluctuates for 40 minutes in the range between 40°C and 40.2°C. In the area that does not come into direct contact with the heater, an average temperature of 30°C is reached at the 30th minute of the study, with average temperature fluctuations starting from the 44th to the 98th minute in the range from 30.6°C to 30.8°C.

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Current state of research in the field of measures regarding the prevention and combat of the freeze-thaw phenomenon of roads from the patent literature

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Abstract: Patents for inventions in the field of measures for preventing and combating the freeze-thaw phenomenon of roads address a topic related to the evolution of technology. Of particular importance is the stabilization of the substrate of the road transport route. An invention (J. Horneegger, N. Zafir, M. Alupoaie, 2012) relates to a mixture used for the substrate of access roads and / or platforms. This invention relates to another invention (Y. Kadono, T. Nakazato, E. Miura, T. Kitamura, 1975) about a paved roadbed composed of a lower roadbed layer, an intermediate layer made of sulfur, an upper roadbed layer and a surface top layer. Another invention (D. Lee, K. Hong-joo, 2016) relates to a repairing and reinforcing ultrarapid hardening mortar composition, and to a method for repairing and reinforcing a road surface. Another invention (M. Radu, F. Radu, L. V. Pasăre, I. A. Buzuriiu, P. Părvu, C. G. Mudrețchi, C. Răcănel, A. Burlacu, V. Tiron, 2018) relates to a process for preparing ecological composite asphaltic mixtures to be used as road structures.

Keywords: ROAD, FROST, FREEZE, THAW, DEFROST, PATENT, INVENTION

1. Introduction

Road complexes are affected by the freeze-thaw phenomenon. At temperatures of 0°C free water passes from liquid to solid state, through the phenomenon of freezing. Freezing of water from the pores leads to changes in the physical and mechanical properties of the road layers, among them we can mention: important volume variations, increases of mechanical strengths (by increasing cohesion) during frost, decrease of permeability and decrease of mechanical strengths during thawing. These changes can be explained by the increase in water volume of approx. 9% by frost. By increasing the volume of water by freezing there is a swelling of the bituminous coatings due to ice crystals that tend to dislodge the particles from the initial position, destroying the structure of the road layer.

In the case of the non-rigid road system, the following effects may occur due to the freeze-thaw phenomenon:

- irregular swellings (bellows), possibly accompanied by cracks and fissures, of the bituminous coatings;
- cracks, fissures, faience, cracks and local deformations during the thawing period.

Patents in the field of measures to prevent and combat the phenomenon of freeze-thaw roads address a multiple and complex issue, closely related to the current evolution of technology. The following are examples of some of the most representative patents in this field.

2. Results and discussion

Of particular importance is the stabilization of the substrate of the road transport route. The substrate of the track is that layer of non-cohesive material placed between the track prism and the upper face of the embankment. An invention (J. Horneegger, N. Zafir, M. Alupoaie, 2012) [1] relates to a mixture used for the substrate of access roads and / or platforms. The mixture for the track substrate which is the subject of the invention is intended to replace the ballast distribution layer at the access roads, providing protection to the track bed. The mixture according to the invention consists of 70% recycled crushed stone or new crushed stone, crushed and sieved, and 30% natural aggregates, such as gravel and sand.

The technical problem solved by the invention consists in protecting the track bed by increasing the frost resistance and improving the distribution of the efforts from permanent loads and from payloads. The mixture for the track substrate that is constituted in proportion of minimum 30% natural aggregates and maximum 70% recycled crushed stone, crushed and sifted, the mixture having a well determined granulometry, a homogeneous composition with a content of fine particles with diameter $d < 0.05$ mm less than 3% and the organic matter content less than 1% by weight.

The mixture for the track substrate has a particle size determined by sieving through sieves of different sizes, shown in Table 1.

Table 1: Granulometry of the roadway substrate.

Sieves diameter [mm]	Mixture of recycled crushed stone and natural aggregates [%]
63	100
45	80-100
32	70-95
16	50-73
8	37-60
4	26-48
2	18-40
1	14-32
0,5	8-25
0,25	4-18
0,12	0-10
0,06	0-3
0,02	-

The mixture for the track substrate according to the invention consists of a mixture of 11 natural aggregates having the non-uniformity coefficient $U_n > 15$, the equivalent diameter $d_{85} > 10$ mm, the permeability coefficient $K < 1 \times 10^{-6}$ m/s for the degree of compaction Proctor D = 95%.

By applying the invention, the following advantages are obtained:

- the mixture offers protection to the track bed, being made of frost-resistant granular materials;
- provides a good distribution of efforts from permanent loads and payloads;
- creates a good load-bearing capacity;
- achieves a uniform elastic environment on the entire road platform.

This invention relates to another invention (Y. Kadono, T. Nakazato, E. Miura, T. Kitamura, 1975) [2] about a paved roadbed composed of a lower roadbed layer, an intermediate layer made of sulfur, an upper roadbed layer and a surface top layer, the overall thickness of the upper roadbed layer and surface top layer being greater than about 8 inches and the sulfur layer being positioned atop the existing water table and being about 4-12 inches thick.

Fig. 1 represents a longitudinal sectional view illustrating an example of a paved roadbed according to the present invention.

The roadbed comprises a lower roadbed layer **1** which consists of a roadbed base **2** formed by leveling the natural ground and an overlying layer **3** of any conventional roadbed material such as sand. As an alternative, the layer **3** may be formed by rolling directly the roadbed base **2** without using a roadbed material.

Atop the lower roadbed layer there is a layer **4** formed substantially of sulfur. Sulfur used therein is solid sulfur such as

grain or flake-shape. The sulfur layer 4 is not always percent sulfur, but may be made substantially of sulfur.

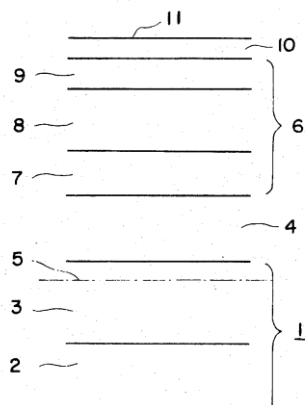


Fig. 1 Longitudinal sectional view of a paved roadbed.

The sulfur layer 4 is positioned atop the water table or underground water level 5.

Atop the sulfur layer 4 there is provided an upper roadbed layer 6. It is preferable that the upper roadbed layer 6 be formed of conventional roadbed material, or a material of small particles.

Over the layers 7, 8 and 9 there is a surface layer 10, the upper surface of which corresponds to the so-called road surface 11.

This invention contemplates therefore an improvement in paved road surface, by utilizing two known but heretofore unexploited properties of sulfur. It has been proven through experiments that the paved roadbed according to this invention undergoes no adverse influence under the severe weather condition existing in the extremely cold regions, and no frost heaving phenomenon, with the result of a notably longer service life.

Another invention (H. Sang-il, J. J. Woo, 2017) [3] relates to an asphalt concrete admixture for road pavement which is improved in the effect of suppressing ice formation in the asphalt concrete mixture used for road pavement by mixing the same.

This invention relates to an asphalt concrete admixture for road pavement which is capable of reducing the risk of freezing, and an asphalt concrete admixture containing the same. The slip accident can be prevented by reducing the phenomenon of freezing of the road surface due to snow or rain during the winter season. In addition, it does not contain chloride as an additive, so it prevents corrosion of metal and concrete due to chloride ion (Cl^-), and can prevent environmental pollution such as soil pollution and water pollution. The present invention also provides an asphalt concrete improved in slip resistance performance while maintaining the inherent physical properties of asphalt concrete even when the asphalt concrete admixture for road pavement, in which the inorganic salt, the porous body and the organic acid in the asphalt concrete mixture are embedded.

This invention proposes a multifunctional road pavement coating material which is excellent in absorbing power of snow removing agent, effectively prevents freezing of the road pavement surface and temperature rise, and has high ultraviolet stability.

The present invention relates to an asphalt concrete additive for road pavement which is improved in the effect of suppressing ice formation. The additive may include 4 to 17 parts by weight of the porous material and 7 to 23 parts by weight of the organic acid based on 100 parts by weight of the inorganic salt.

Another invention (D. Lee, K. Hong-joo, 2016) [4] relates to a repairing and reinforcing ultrarapid hardening mortar composition, and to a method for repairing and reinforcing a road surface, a runway surface, and an L-shaped side gutter deteriorated part by using the same. More specifically, when the surfaces of a road, a runway, and the like and a deteriorated part and a damaged part of a road side gutter are repaired and reinforced, durability and adhesive strength are improved, so repairing and reinforcing effects can be

maintained for a long time. At the same time, repairing and reinforcement can be stably completed in a short time, so excellent economical feasibility can be ensured. Also, the repairing and reinforcing ultrarapid hardening mortar composition can be directly used on the spot, so usage convenience is excellent. Moreover, excellent resistance is ensured against freezing, thawing, and salt damage.

The repair or reinforcement composition according to the present invention comprises the following six components:

a) A mixture composed of 65 to 80 wt% blast furnace slag having a specific surface area of 6000 to 7000 cm^2/g , 10 to 20 wt% of lime, 5 to 15 wt% of anhydrous gypsum or high, 50 to 100 parts by weight of a mixture having a specific surface area of 6,500 to 7,500 cm^2/g through a mechanochemical activation treatment by mixing and pulverizing, 0,5 to 10 parts by weight of clinker, 0,5 to 10 parts by weight of plaster, to 10 parts by weight of silica fume, 0,1 to 5 parts by weight of fumed silica, 0,01 to 5 parts by weight of fly ash, 0,1 to 5 parts by weight of red mud, 0,01 to 10 parts by weight of calcined pomolane and 0,01 to 10 parts by weight of micro silica, 10 to 70% by weight of a component; 0,05 to 0,2 parts by weight of a viscosity enhancer; 0,3 to 1,1 parts by weight of a fluidizing agent; 0,05 to 0,3 parts by weight of a curing accelerator; 0,1 to 0,4 parts by weight of a retarding agent; 2 to 7 parts by weight of a siliceous waterproofing agent; 1,5 to 3 parts by weight of a CSA, and 42 to 64 parts by weight of silica sand and 30 to 90% by weight of a second powder component comprising 100 parts by weight of the powder component.

b) One or more powder or liquid rubber selected from ethylene-vinyl acetate (EVA) resin, NR (natural rubber) resin, NBR (natural rubber-butadiene rubber) resin and SBR 1 to 20 parts by weight of a modified latex component obtained by mixing carbon black and fibers in a resin;

c) 1 to 7 parts by weight of methyl methacrylate, 5 to 20 parts by weight of styrene monomer, 1 to 10 parts by weight of n-butyl acrylate, 0,1 to 10 parts by weight of methyl acrylate, 0,1 to 10 parts by weight of isobornyl acrylate, 0,05 to 5 parts by weight of an initiator and 0,05 to 5 parts by weight of an emulsifier;

d) 1 to 10 parts by weight of a hydrophilic polyvinyl alcohol short fiber component;

e) 1 to 5 parts by weight of an additive component comprising a mixture of a preservative, an antifoaming agent and a humectant;

f) 10 to 50 parts by weight of water.

Another invention (A. V. Lăzărescu, H. Szilagyi, C. Baeră, A.C. Hegyi, V. Meită, 2021) [5] relates to a process for making an alkaline-activated geopolymer microconcrete without cement content and to prefabricated elements such as pavers, slabs and street curbs made with this microconcrete, these being used for arranging pedestrian roads, bicycle lanes, parking lots, roads, highways, industrial spaces, docks and ports, airstrips and the like.

The process according to the invention for making the geopolymer microconcrete consists in mixing the following raw materials: maximum 40% class F power plant ash existing in Romania, with fineness characterized as a residue on a 0,045 mm sieve, an alkaline activator based on Na_2SiO_3 and NaOH and natural aggregates of river with a maximum granulation of 8 mm, the mass ratio between the Na_2SiO_3 solution and the NaOH solution used for the preparation of the alkaline activator being between 0,5 - 2,5, and the mass ratio between the alkaline activator and the power plant ash is between 0,5 - 1,0. The pavers according to the invention have a length between 200 - 250 mm $\pm$ 2 mm, a total width of 80 - 110 mm $\pm$ 2 mm, a height of 60 - 80 mm $\pm$ 3 mm and have a strength after 7 days of casting at splitting of at least 3,6 MPa. The tiles according to the invention have a length between 200 - 1000 mm $\pm$ 5 mm, a variable width, a height between 30 - 100 mm $\pm$ 5 mm and have after 7 days from casting a tensile strength by bending of minimum 3,5 MPa. The edges according to the invention have a length between 500 - 1000 mm $\pm$ 3 mm, a width of at least 200 mm $\pm$ 3 mm, a height of at least 200 mm $\pm$ 3 mm, and have a tensile strength at 7 days after casting, bending of at least 3,5 MPa, all these prefabricated pavement elements having a density of 1600 - 1900 Kg/m^3 , water absorption of max. 6%, Bohme wear resistance

of maximum 20000 mm³/5000 mm² and mass loss after freeze-thaw of max. 1 Kg/m².

Another invention (M. Radu, F. Radu, L. V. Pasăre, I. A. Buzuriu, P. Pârnu, C. G. Mudrețchi, C. Răcănel, A. Burlacu, V. Tiron, 2018) [6] relates to a process for preparing ecological composite asphaltic mixtures to be used as road structures.

According to the invention the process consists in that it is carried out at a temperature of 165 - 170°C by mixing the mineral aggregates bitumen and additions resulting from industrial wastes by using a mobile preparation station placed close to the place of using to finally obtain a mixture having a water-absorption level of 0,18 - 0,57% an optimal behaviour at the freezing phenomenon and stability within the thermal range of use from -40°C up to 70°C.

The technology which is the object of the invention represents a succession of operations optimized in terms of the materials used, the duration of these operations, the temperature at which the process takes place and the working parameters of the equipment used, as follows.

It starts with the preparation of the materials that make up the composition of a batch of mixture. The natural aggregates used are dosed according to the following particle size composition:

- Chip 16-25 mm: 27,0 – 30,7 %
- Chisel 8-16 mm: 9,0 – 11,5 %
- Chippings 4-8 mm: 10,0 – 19,1 %
- Crushed sand: 15,0 – 16,3 %
- Natural sand: 14,0 – 15,3 %
- Filler: 2,7 – 2,9 %

Except for the filler (which is added directly to the mixer), these aggregates are placed in an oven for drying and heating to a temperature of 165 - 170 °C, the process being carried out in a continuous flow, in a mobile station. The heated aggregates are then transferred with a conveyor belt to the mixer of the mixture preparation station, batchwise, in the amount corresponding to a batch. A quantity of hot bitumen is introduced into the mixer, in a proportion of 4,2 % in total (of which about 1 % will be used for melting and homogenizing high density polyethylene and sulfur required for crosslinking) in the case of mixtures intended laying the connecting layers of road pavement and in proportion of 6% in total in the case of mixtures prepared for the wear layer (of which about 1 % will be used in this case for melting and homogenization of high density polyethylene and sulfur needed for crosslinking).

Granulometric sieve sorters can be partially replaced with recycled steel slag, which is cheaper and has superior mechanical strength. Their weight, cumulated for the three particle size classes, is optimally in the range of 15,0 – 18,0 %, a higher weight of slag leading to high water absorption coefficients, requiring additional additions of high density polyethylene, with correspondingly higher costs.

Of the amount of bitumen prepared for introduction into the mixer, a part (about 1 %) is used for melting and homogenizing the thermoplastic addition consisting of high density polyethylene in the form of granules, in a proportion of 0,1 % to the mass of a batch, as well as for the dispersion of sulfur in proportion of 0,0005% to the mass of a batch. Said process, lasting one hour, is carried out in a reaction vessel provided with a stirrer, at a temperature of 165 - 170 °C, the sulfur powder being introduced 10 minutes before being introduced into the mixer. A bitumen pump is used to transfer the mixture of bitumen, polyethylene and sulfur to the mixer. In addition to the main role in the road pavement in which the asphalt mixture (waterproofing and elastomer) will be integrated, the addition of high density polyethylene also has the secondary role of ensuring an optimal viscosity of the mixture, by crosslinking with all components, especially with synthetic rubber powder (butadiene-a-methylstyrene copolymer), recycled from used tires, with a grain size of 0 – 0,5 mm and with steel slag, which makes it less absorbent for water, by its external filming.

Rubber powder plays a key role in ensuring the elasticity of asphalt pavement. It is pneumatically dosed and inserted directly into the mixer, in a proportion of 0,2 – 0,3 % of the mass of a batch. The crosslinking between the thermoplastic component consisting of high density polyethylene and the elastomeric component,

consisting of powder, is performed using a small amount of technical sulfur 99,5 % used in the vulcanization mixtures of elastomers, in proportion of 0,0005 % to the mass of a charges.

Other additives are also introduced in the mixer to improve the performance of asphalt pavements. Thus, in a proportion of 0,1 - 0,5% compared to the mass of a batch, polyacrylonitrile fibers with dimensions of 4 - 20 mm and fineness of 20 - 200 Tex are added, to reinforce the composition of the mixture, having as effects the increase of stability. road layer and its compressive strength, but also increased water absorption, to counteract which increases the proportion of high density polyethylene to the value of 0,15 – 0,2%, 0,18 % cationic polyelectrolytes are also added to increase adhesion and elastic recovery.

Kneading takes about 10 minutes. After the complete homogenization of the asphalt mixture, it is transferred, using dump trucks, in the equipment provided for its asphaltting in order to achieve some layers of wear and connection of the road pavements, for all categories of roads. The sizing and compaction technology is the classic one.

The invention has the following advantages:

- it is energy efficient, by substantially shortening the period in which the bitumen and then the asphalt mixture are heated and maintained at the process temperature, as well as by reducing this temperature to the value of 165 - 170 °C;

- it is economically efficient, in addition to reducing energy consumption and reducing costs, by using additives prepared from cheap raw materials, obtained from waste and by avoiding long-distance transport of mixture and mixture components, as well as by avoiding considerable time deaths and, in this way, the increase of labor productivity;

- ensures a superior quality of the asphalt mixture, primarily in terms of mechanical strength and durability in operation, of about 15 years, due to the fact that all interactions between components take place at a temperature (165 - 170 °C) at which the fluidity the homogeneity is also optimal, avoiding some separations in time of the crosslinked components, with unfavorable effects on the characteristics of the asphalt pavement;

- the technology ensures a mixture whose water permeability is considerably reduced, having as an effect an optimal behavior to the phenomenon of frost and the increase of the thermal range of safe use (from - 40 °C to + 70 °C), due to the addition high density polyethylene and rubber powder;

- the technology is environmentally friendly, without the use of solvents that, by evaporation, lead to harmful emissions;

- does not require a special expensive installation, such as a colloidal mill;

- the costs and energy consumptions for the modification of the bitumen before the preparation of the mixture are avoided, in order to improve its performances.

Another invention (J. Jeong, 2002) [7] relates to an ice melt injection device for preventing freezing of roads.

This invention provides a meteorological observation unit, a central control unit for receiving an output value of the meteorological observation unit by wire or wireless, comparing the reference value with a reference value, and outputting a control signal according to the output value.

A pump station having a control panel for operating a pump connected to a melt liquid storage tank for pressurizing the melt liquid in accordance with an output signal while connected to the output side of the central control unit. It consists of a melt ice injection section installed on both sides of the road surface, and a solenoid valve provided in the pipeline connecting the pump station and the melt liquid injection section, so that the frozen road surface can be thawed in winter to prevent traffic accidents caused by slipping. In summer, by reducing the temperature of the road surface to reduce the shrinkage and inflation length of roads, roads, bridges, etc. due to thermal expansion, to prevent damage, clean the road surface to provide a clean road environment will be.

3. Conclusion

Research for patenting in the field of preventing and combating the freeze-thaw phenomenon of roads includes the following steps: identification, selection of technology or application of technology.

Technology transfer is a process that is influenced by the development and changes of science-based technologies, with references in the fields of advanced technologies. Depending on the intention of the technology provider, this transfer falls into two categories: unintentional transfer (in which the innovator has no profit) and intentional transfer (in which the provider has benefits).

Patents for preventing and combating the freeze-thaw phenomenon of roads are brought to the attention of those interested in this field and through popularization in international scientific conferences and congresses, as well as the publication of articles and papers belonging to the technical and scientific literature.

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