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IN MEMORIAM OF PROFESSOR STANISŁAW PABIS

Professor Stanisław Pabis passed away suddenly on September 13, 2019, at the age of 94.

We lost an outstanding scientist in the field of Biosystems Engineering who founded the scientific school of methodology of empirical sciences and who made a significant contribution to the development of systems engineering in agriculture and food processing.

Professor Stanisław Pabis was born on 23 April 1925 in Krosno. He graduated from the Faculty of Mechanical Engineering of the Gdańsk University of Technology in 1952 and was employed by the newly created Institute for the



Development, Mechanization and Electrification of Agriculture (IBMER) in Warsaw in the same year. Stanisław Pabis continued to pursue an academic career during his employment at the IBMER. He was awarded a doctor's degree in Technical Sciences at the Faculty of Mechanical Engineering of the Warsaw.

University of Technology in 1960, the degree of habilitated doctor in Agricultural Sciences at the Faculty of Agriculture of the University of Agriculture in Lublin in 1966, the title of Associate Professor of Technical Sciences in 1971, and the title of Full Professor in 1983. During his employment in the IBMER in 1952-1980, Professor Pabis founded and managed the Agri-

cultural Drying Plant in the Institute's Branch in Kłudzienko (1954-1970). The plant featured the most advanced drying laboratory in Europe at the time. In 1971-1980, Professor Pabis founded and managed the Cybernetics Department of the IBMER in Warsaw. Between 1977 and 1995, Professor Pabis was a member of the academic staff of the Warsaw University of Life Sciences, where he was the Dean of the Faculty of Agricultural and Forest Technology (1981-1987) and the Head of the Department of Agricultural Process Engineering (1980-1995). He was a member of numerous Polish and international scientific associations and committees.

Professor Pabis' greatest scientific achievements were in the field of the drying of agricultural products. His research on the mathematical modeling of drying kinetics in agricultural products, conducted during a Rockefeller

scholarship program in 1960-1961, remains the cornerstone of scientific literature on agricultural drying methods, and his papers have been cited and referenced thousands of times. The results of his research into agricultural drying techniques were summarized in two books: Theory of Convective Drying of Agricultural Products (Teoria konwekcyjnego suszenia płodów rolnych), published by PWRiL in 1982, and a collective monograph entitled Grain Drying - Theory and Practice, published by John Wiley and Sons in New York in 1998. Professor Pabis also authored numerous articles in the leading scientific journals around the world. His scientific work also involved practical achievements, including many patents and designs of new drying equipment. Professor Pabis also conducted research into methods of empirical research, which gave rise to three books: *Methodology* and Methods of Empirical Sciences (Metodologia i metody nauk empirycznych), published by PWN in 1985, Methodology of Empirical Research – 12 Lectures (Metodologia nauk empirycznych – 12 wykładów) and Methodology of Empirical Research – 15 Lectures (Metodologia nauk empirycznych – 15 wykładów), published by the Koszalin University of Technology in 2007 and 2009, respectively.

Stanisław Pabis participated in several research placements and scientific internships, including at the Michigan State University in 1960 as a scholar of the Rockefeller Foundation, at the University of California in 1960-1961, and at the University of Manitoba, Canada in 1990-1991 as a scholar of the Natural Sciences and Engineering Research Council of Canada. He gave many lectures in research centers in the USA, Canada, Germany, the Netherlands, Great Britain, Hungary and other countries. In 2004, he was the first senior scientist in Poland to have received the NESTOR scholarship from the Foundation for Polish Science.

Professor Stanisław Pabis founded two academic education programs that were held each year in Poland. The first was the *School of Agricultural and Forest Engineering Systems* which was created in 1978 and managed by Professor Pabis for 25 years. The aim of this novel initiative was to disseminate knowledge on the latest research methods in the community of Polish agricultural engineers. The second project was the *School of Empirical Sciences Methodology* which opened in 1986 and was managed by Professor Pabis for 25 consecutive years. The school was established to promote the knowledge of research methodology among young scientists. Professor Pabis fervently supported the reintegration of the discipline of Agricultural Engineering into the field of Engineering and Technical Sciences, which ultimately took place during his career.

The main scientific and academic achievements relating to Professor Pabis' key research interests include 235 articles that were authored or co-authored by the Professor in Polish and foreign scientific journals (half of which are singleauthor papers), five books authored by the Professor, four books co-authored by the Professor, supervision of 23 doctoral students, reviews of 44 doctoral dissertations, 45 habilitation theses, and 26 evaluations of candidates for the title of professor. Professor Pabis also managed several dozen research projects.

Professor Stanisław Pabis was held in high esteem by the Polish community of biosystems and agricultural engineers. He was a respected authority whose achievements were widely recognized in the domestic and international scientific arena. He was a mentor and a teacher of many generations of scientists, doctoral students, and students of agricultural engineering. He received numerous awards and honors for his scientific, teaching and organizational achievements. He was awarded the following Polish state decorations: Silver Cross of Merit, Order of Polonia Restituta, and the Medal of the Commission of National Education. He was awarded on many occasions by the ministers of science and education, the Polish Academy of Sciences and the Rector of the Warsaw University of Life Sciences.

Professor Pabis retired in 1995 at the age of 70, but he continued to actively participate in scientific and academic life. Stanisław Pabis was a modest man who made ends meet on a small pension, but he never lost his passion for science. On 19 September 2019, Professor Pabis was scheduled to deliver a lecture on non-measurable sets during the Fourth Polish School of Biosystems Engineering. Unfortunately, his presentation never saw the light of day. We have lost a man of great passion (Professor Pabis was an avid mountain climber, swimmer, skier, photographer and gardener) and a scientist with many interdisciplinary interests (methodology of empirical sciences, astrophysics, physics, mathematics, philosophy, biology). Professor Pabis dedicated his life to the search for truth and the pursuit of ethics in science. He was a lenient tutor and supervisor to young scientists, and a great educator to many generations of undergraduate, graduate and postgraduate students.

When talking about Professor Stanisław Pabis, his co-workers always referred to him as "my Professor" or "our Professor", and this is how we will remember him.

Co-workers

Professor Małgorzata Jaros, PhD, Warsaw University of Life Science Professor Marek Markowski, PhD, University of Warmia and Mazury in Olsztyn Ryszard Myhan, Associate Professor, PhD, University of Warmia and Mazury in Olsztyn

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AN ANALYSIS OF THE INTERACTIONS BETWEEN INDIVIDUAL ROBOTS IN A DECENTRALIZED MULTI-ROBOT TRANSPORT SYSTEM DURING MOVEMENT INITIATION

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Key words: Mobile robot, decentralized multi-robot system, time delays, robotization of transport.

Abstract

The article analyzes the influence of control signal delays on the initiation of movement in a distributed group of robots and the implementation of the robots' programmed trajectory. A static analysis was carried out in a distributed group of robots, where the applied constraint was the connection of the robots to a transport pallet. The loads resulting from delayed transmission of the control signal were subjected to a dynamic analysis in MSC Adams View software.

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Introduction

Entrepreneurs are constantly faced with the challenge of increasing their production efficiency and reducing their operating costs in order to achieve success and strengthen their position on the market. This goal can be attained through the automation of in-house logistics. This solution is gaining widespread popularity because according to Industry 4.0 standards, internal transport is one of the most dynamically developing sectors of the economy. Transport operations generate losses due to increasing labor shortages and the growing costs of transport in the production process. Therefore, the costs associated with the internal transport of products have to be minimized by shortening product transport routes and times. Transport costs can be decreased by introducing autonomous robots which perform transport operations accurately, quickly and safely (Autonomiczne mobilne roboty... 2018). According to a report of the United Nations Conference on Trade and Development, robot-based transport facilitates and optimizes the work of factory employees and decreases production time by as much as 25% (JURCZAK 2018). Such a solution has been implemented in the Seat factory in Spain where mobile robots transport around 24,000 car parts daily (Fig. 1).



Fig. 1. Mobile transport robots at the Seat factory in Spain

Cooperating mobile robots for in-house transport are navigated based on independently created maps or previously uploaded building plans. The robots are equipped with cameras, built-in sensors and laser scanners to determine the location of the transported objects, avoid obstacles, respond to humans and other robots encountered on the route. A safety and navigation system that stops or activates the robot at the right moment is a very important element of the control algorithm.

An effective solution enabling the synchronous activation and deactivation of a group of transport robots is also required for managing robotized tasks. The factory environment usually consists of numerous rooms, production lines and devices that cause interference and delays in wireless transmission (SIWEK et al. 2018b). Even minor delays in the transmission of the control signal can cause considerable errors in the positioning of individual robots in a group (SIWEK et al. 2018a). The cooperation between mobile robots and industrial robots is particularly sensitive to positioning errors. A mobile robot has to assume a precisely controlled position to lift or put down an object (KACZMAREK et al. 2018). Various methods for controlling robot groups and eliminating positioning errors have been proposed in the literature. Such solutions include the Virtual Structure algorithm described by TAN and LEWIS (1996) and the optimal trajectory planning algorithm developed by CHENG et al. (2016). However, the time drift between individual robots is one of the key problems in transport operations involving distributed groups of robots. This problem has been discussed extensively and various solutions have been proposed in the literature (AUTEFAGE et al. 2015).

The article analyzes the interactions between individual robots that transport cargo in a dispersed group. The circumstances under which the control signal is delayed were examined. When the control signal is delayed, the robot that is activated earlier exerts a load on stationary robots through the support platform (pallet, frame). The above can lead to undesirable changes in the position and orientation of the robot group before and after the set trajectory is completed. If such changes occur frequently, they can accelerate wear or cause damage to drive system components.

An analysis of the interactions between individual robots in a distributed multi-robot system

In a distributed multi-robot system, robots form a single network, but they interpret signals from the management center independently of each other and change their position or activity accordingly. The location coordinates of each robot in a distributed group are calculated independently, but relative to a common, stationary reference system. Each robot belongs to a group of robots that perform a specific task in a consistent manner. For the task to be carried out in a coordinated manner, the initial conditions (configuration of the robot group) have to be set, and the clocks of individual robots have to be synchronized (SIWEK et al. 2018a). In the process of controlling a distributed

group, attention should be paid to errors in the transmission of control data which cause delays in the control algorithm. Electromagnetic interference (caused by, for example, numerous wireless networks), power network interference, and hardware differences between the operating robots (network cards, processors) can impair the synchronized execution of the control algorithm and, consequently, lead to delays in task performance by individual robots. Incorrect or inefficient time synchronization will lead to operating errors in the control algorithm of the distributed group. In practice, the activation, maneuvering and deactivation of individual robots will proceed asynchronously. As a result, the task will not be correctly performed because robots will move along the wrong trajectory, they will fail to maintain formation, or they will collide with obstacles. In this article, the movement of a group of three mobile robots with differential drives was examined to determine the interactions between individual robots in the group (Fig. 2). The robots were tasked with transporting a load on a rigid transport pallet. They were connected to the pallet by rotary joints with one degree of freedom. The analyzed transport system is presented in Figure 2.



Fig. 2. View of the transport system: $1-{\rm transported}$ element, $2-{\rm pallet},$ $3-{\rm mobile}$ robot, $4-{\rm swivel}$ joint

A static analysis of loads in a distributed multi-robot system where the applied constraint is the connection of the robots to the pallets.

The initiation of movement in a group of transport robots was analyzed. It was assumed that two robots (Robot 1 and Robot 3) would be delayed (due to data transmission errors) relative to the third robot (Robot 2). The force with which Robot 2 pushes the pallet acts as a driving force on delayed Robots 1 and 3. The force would increase to the maximum value due to the robot's maximum driving moment. In practice, the above could lead to wheel boxing, engine burning or joint breakage. A simplified diagram of the transport system is shown in Figure 3, where: $1, \dots, 4$ – transport pallet joint number; d_1 , d_2 , d_3 – dimensions of the transport pallet; a_2 , a_3 – inclination angles of the outer beams of the pallet;



Fig. 3. Simplified diagram of the transport system, description in the text

0xy – global coordinate system; F – force with which Robot 2 pushes the pallet; R_1x , R_1y , R_3x , R_3y – joint reaction forces in Robots 1 and 3 according to the x and y axes of the global coordinate system 0xy; N_{ij} where i, j = 1, ..., 4 – internal forces in the structural elements of the pallet.

The applied force will elicit a reaction in the joints of Robots 1 and 3, and it will cause internal forces in the structural elements of the transport pallet. The transport pallet shown in Figure 3 can be regarded as a truss. If the loads are applied only to the joints, and the joints do not move relative to each other, the system can be considered static. Robots 1 and 3 act as fixed supports. The joints connecting the pallet to the robots can only be rotated in axes perpendicular to the ground plane xy. The balance of forces and moments in truss nodes was used to determine the reaction in supports and the internal forces acting on the structural elements of the frame. This task was accomplished by developing a system of equations (1), where M_1 , M_2 , M_3 are the total torques in truss joints, and Px and Py represent the sum of forces acting in the axis of the global coordinate system 0xy.

$$\sum M_{1} = -Fd_{3} - R_{3y}d_{1} + R_{3x}(d_{2} - d_{3}) = 0$$

$$\sum M_{2} = R_{3x}d_{2} - R_{1x}d_{3} + R_{1y}d_{1} = 0$$

$$\sum M_{3} = -Fd_{2} + R_{1x}(d_{2} - d_{3}) + R_{1y}d_{1} = 0$$

$$\sum P_{x} = -F + R_{1x} - R_{3x} = 0$$
(1)

An auxiliary equation (2) was also formulated to check equation (1) for errors:

$$\sum P_y = R_{1y} + R_{3y} = 0 \tag{2}$$

The above system of equations was transformed into a matrix (3) which was solved in MATLAB software. The dimensions of the truss (Tab. 1) were taken into account when determining the reaction of the supports. The value of the force was set at F = 250 N, and it corresponds to the value of the force generated by a robot with a fully charged battery. The values of support reactions are presented in Table 2.

$$\begin{bmatrix} Fd_3 \\ 0 \\ Fd_2 \\ F \end{bmatrix} = \begin{bmatrix} 0 & 0 & d_2 - d_3 & -d_1 \\ -d_1 & d_3 & d_2 & 0 \\ d_2 - d_3 & d_1 & 0 & 0 \\ 1 & 0 & -1 & 0 \end{bmatrix} \begin{bmatrix} R_{1x} \\ R_{1y} \\ R_{3x} \\ R_{3y} \end{bmatrix}$$
(3)

Table 1

r arameters of the transport system					
Parameter	$d_1 \mathrm{[m]}$	$d_2 \mathrm{[m]}$	$d_3 \mathrm{[m]}$	$a_2 [\mathrm{deg}]$	$a_3 [\mathrm{deg}]$
Value	1.0	1.2	0.7	60	75
					Table
		Support reac	tions		

Parameters of the transport system

$R_1 x$ [N]	$R_1 y$ [N]	$R_3 x$ [N]	R_{3y} [N]
-197.5	127.5	75	-75
	<i>R</i> ₁ <i>x</i> [N] -197.5	$\begin{array}{c c} R_{1}x [\mathrm{N}] & R_{1}y [\mathrm{N}] \\ \hline & -197.5 & 127.5 \end{array}$	$R_1 x$ [N] $R_1 y$ [N] $R_3 x$ [N] -197.5 127.5 75

The calculated values of support reactions were used to determine the internal forces acting on the structural elements of the frame. The reactions of joints 1, 2 and 3 were calculated with equations (4), (5), (6) in MATLAB software. The values of internal forces are presented in Table 3.

Joint 1

$$\sum_{x} P_{1x} = -R_{1x} + N_{13} \sin(90^\circ - \alpha_3) + N_{12} \cos(90^\circ - \alpha_2) + N_{14} = 0$$

$$\sum_{y} P_{1y} = R_{1y} + N_{12} \sin \alpha_2 - N_{13} \sin \alpha_3 = 0$$
(4)

Joint 2

$$\sum_{x} P_{2x} = -F - N_{12} \cos(90^\circ - \alpha_2) = 0$$

$$\sum_{y} P_{2y} = -N_{24} - N_{12} \sin \alpha_2 = 0$$
(5)

Joint 3

$$\sum P_{3x} = R_{3x} - N_{13} \sin(90^\circ - \alpha_3) = 0$$

$$\sum P_{3y} = -R_{3y} + N_{34} + N_{13} \sin \alpha_3 = 0$$
 (6)

Table 3

values of internal forces						
Parameter	$N_{12}[N]$	$N_{13} [{ m N}]$	N_{14} [N]	N_{24} [N]	$N_{34} [{ m N}]$	
Value	-287.35	288.46	-22.5	250	-351.92	

Values of internal forces

A dynamic analysis of loads resulting from control signal delays

The analysis was carried out using MSC Adams software. A simplified transport system was modeled (Fig. 4) to determine deviations in the positioning of delayed robots, caused by force F = 250 N. The deviations were measured over a time period equivalent to the delay in the transmission of the control signal. The initial conditions for the simulation were formulated in the first step. The initial position and the initial orientation of the robots in the global coordinate system were determined. A simplified model of a robot composed of a body and two drive wheels was adopted. Rotary joints controlled by drive torque were positioned on wheels. Rotary joints with free rotation were applied at the point of connection to the transport pallet. The transported load was rigidly connected to the pallet. The mass of all modeled components was equivalent to their real mass. The moment of inertia was determined in the CAD program relative to the center of gravity. The analysis accounted only for the movements along the ground plane, and the appropriate constrains were implemented in the model.



Fig. 4. Simplified simulation model of the transport system in MSC Adams software

Displacements along x and y axes of the frame at the points of connection to the robots were determined during the simulation of movement in the transport system. The delays in the transmission of control signals ranged from 0 ms to 200 ms. The changes in the positional errors of individual robots during group movement, caused by three delays in the transmission of control signals of Robot 1 and Robot 3 relative to Robot 2 (0, 50 and 200 ms), are presented in Figures 5, 6 and 7.



Fig. 5. Trajectory of Robot 1 resulting from the delay of control signals



Fig. 6. Trajectory of Robot 2 resulting from the delay of control signals



Fig. 7. Trajectory of Robot 3 resulting from the delay of control signals

An analysis of the trajectories presented in Figures 5-7 reveals errors in the robot's final position caused by the time delay. These errors can be eliminated by formulating a control law that accounts for the delays caused by the time-varying force acting on the robots. Therefore, the differences in the forces between robots should be minimized in the process of formulating the goal function.

Results and Discussion

The article discusses the practical applications of mobile robots working in a dispersed group to transport large-sized and irregularly shaped objects. A review of the literature revealed that delays in the transmission of control signals to the robot system compromise the system's positioning accuracy and constitute an important problem in robotized transport operations. The results of the simulation tests conducted in this study indicate that:

- The delays in the transmission of control signals lead to non-synchronous activation of robots in a dispersed group;

 During non-synchronous initiation of movement, the force with which a mobile robot pushes the transported load elicits a reaction in the motionless robots. These reactions affect the service life and the accuracy of the robots' propulsion systems;

- The resulting forces lead to displacement and changes in the orientation of the transport system, which, consequently, causes positional and orientation errors relative to the initial configuration.

The above problems can be resolved by formulating a control law that accounts for the dynamic synchronization of robots in a dispersed group and by minimizing differences in the forces between robots in the process of formulating the goal function.

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INFLUENCE OF RUBBER POWDER MASS FLOW RATE ON PLASMA PYROLYSIS

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Keywords: plasma pyrolysis, rubber waste, thermal utilization, absorption spectroscopy.

Abstract

The paper describes an experimental examination of the thermal utilization of used rubber. The research was carried out to examine the influence of rubber powder mass flow rate on the plasma pyrolysis of rubber. An arc plasma generator was applied. Ar and a mixture of Ar and H₂ were used as plasma gases. The composition of gaseous products was analyzed by infrared absorption spectroscopy. All of the rubber introduced to the plasma jet was decomposed. The outgoing gas did not contain any toxic chemical compounds such as NO_x or HCN.

Introduction

Even though, that there have been applied numerous methods to utilize rubber wastes (LIANG et al. 2020, SOVJAK et al. 2019, HUILIN et al. 2019) the problem of used rubber has not been solved satisfactorily yet (WOJCIECHOWSKI, DOLIŃSKI 2014). Almost every landfill in Poland has a special spot covered with used tires well beyond the landfill capacity. This situation seems to be proved by devastating fires of the landfills in 2018 in Skawina, Siemianowice Śląskie,

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Trzebinia (PAWŁOWSKA 2018) and many others. Criminal issue of setting fires is involved to cover cases of used tires illegally brought to Poland. The fires are set out to cut investigations and lawsuits. The examples multiply in the whole country.

In year 2018 in Poland almost 190 thousand (BIAŁASZ 2018) tons of tires were brought out to the domestic market (Fig. 1). The weigh of used tires annually seems to grow almost linearly. It has to be highlighted that used tires make up approximately 75% of all the mass of rubber waste. Additionally, close 20% of tires get worn out during regular exploitation (BIAŁASZ 2018).



Fig. 1. Estimated annual amount of used tires in Poland

Thermal utilization of rubber waste can be not only a solution to the environmental problem but it might be a profitable activity as well. Decomposition of rubber in the plasma jet may be a source of hydrocarbons and syngas which exquisitely can reduce costs of utilization process of rubber by using those gases as fuel. Moreover, plasma pyrolysis of rubber might reduce amount of side products comparing to other thermal utilization methods of rubber waste.

Amongst the physical and thermal methods of utilization of rubber waste plasma pyrolysis brings a special attention and interest. That kind of pyrolysis is applied more seldom than thermal pyrolysis (maximum process temperature – 1,000 K). Plasma pyrolysis has much bigger potential though (WIELGOSIŃSKI 2011, TANG, HUANG 2004). Plasma pyrolysis offers very high process temperature, which can range from 6,000 K to 18,000 K (MAJEWSKI, DĘBSKI 2012). So high process temperature provides plasma pyrolysis with huge advantage over thermal pyrolysis or ordinary incineration, especially considering decomposition of any substance or purity of gaseous products of rubber thermal utilization. That high temperature is able to decompose almost every substance and material.

The process of plasma pyrolysis has already been examined (SZUSZKIEWICZ et al. 2001) considering plasmatron power influence on the course of plasma pyrolysis of rubber. However, a dependence of rubber powder mass flow rate on plasma pyrolysis of rubber has not been investigated yet.

The aim of the research described in the paper was to determine the influence of amount of rubber delivered to the plasma jet on the composition of the gaseous products in the process during plasma pyrolysis. The secondary aim of the research was to investigate the influence of mass of rubber on concentration changes of the gaseous products as a potential source of energy.

Experimental set-up

The research on plasma pyrolysis of rubber has been carried out using a plasmatron (Fig. 2). The source of the heat is a plasma jet generated with plasma gas ionized by electric arc in the plasmatron (MAJEWSKI 2011, MIKOŚ 1987). The maximum electric power of the plasmatron in all the experiments equalled up to 30 kW.



Fig. 2. Scheme of test - stand of plasma pyrolysis of rubber waste

Ar was used as a primary plasma gas. It is an inert gas and so it does not influence composition of the gaseous products. Also, H_2 was used as a secondary plasma gas. On the basis of the additional experiment (SZUSZKIEWICZ 2007) it was found that in the mixture of plasma gas H_2 should not exceed rate of 3.6%. Higher amount of H_2 in the plasma gas could be dangerous. H_2 is a molecular gas and it plays the role of a gas stabilizing the plasma jet. Additionally, it increases the power of a plasmatron.

The plasma generator is a PN-120 type plasmatron (Fig. 3). It has been produced by the Institute of Nuclear Research in Świerk, Poland. The cathode (3)



Fig. 3. Cross-section of PN-120 plasmatron 1 – casing, 2 – anode-nozzle,
3 – cathode tungsten rod, 4 – cathode holder, 5 – cathode core, 6 – water drainage; positiver pole of electric supply circuit, 7 – water supply; negativer pole of electric supply circuit, 8 – intake of plasma gas, 9 – isolator

of the plasmatron is made of tungsten. The anode (2) is made of pure copper. The anode also serves as a nozzle, which shapes the plasma jet. The plasma generator is electrically DC supplied. That construction is the most popular amongst all the plasmatrons. The DC electric supply (6, 7) simplifies the electric arc initiation and makes the plasmatron construction easier, less expensive and more dependable.

Since the temperature in the axis of the plasma jet is very high and equals up to 18,000 K (CHAMOLLO et al. 2018), both electrodes of the plasmatron have to be cooled intensively. This task is obtained by the water cooling system (6, 7).

To ensure that the reactions of decomposition and synthesis processes of the rubber are not influenced by the ambient air, the plasmatron was placed in the reactor (Fig. 2). The reactor is an open tank, so the outgoing gas will not cause the overpressure inside of it. The temperature of the reactor is lowered by the separate water cooling system (Fig. 2). The other task of the reactor cooling system is to provide intensive quenching inside of it.

The source of the examined rubber was used tires. The rubber was obtained from the vulcanization plant. The exact chemical composition of rubber mixtures has never been revealed by the tires company. It always is kept as a secret. Nevertheless, CHANG et al. (1996) revealed the elemental composition of rubber mixture used in the USA: C – 86.84%, H₂ – 7.17%, incinerated mineral compounds – 3.78%, S – 1.89%, N₂ – 0.3%, O₂ – 0.02%.

The rubber was delivered to the plasma jet by a fluidal feeder (Fig. 2). The size of the rubber particles have to be amounting up to hundreds micrometers to get entirely decomposed. Granulation of rubber powder was investigated in SZUSZKIEWICZ et al. (2007).



Fig. 4. Absorption spectrum of outgoing gas for pyrolysis of rubber powder in Ar plasma (P = 21.45 kW)

The investigation of plasma pyrolysis of rubber was carried out in two separate series of experiments. In the first experiment plasma pyrolysis was carried out in the Ar plasma gas, at constant plasmatron power and constant plasma gas flow rate (Tab. 1). The rubber powder mass flow rate was a variable and ranged from 0.05 kg/h to 8.04 kg/h. In the second experiment plasma pyrolysis was carried out in the Ar + 3.6% H₂ plasma gas (Tab. 2). Also the rubber powder mass flow rate was a variable and ranged from 0.05 kg/h to 8.04 kg/h.

	Parameters of pyrolysis of rubber powder in Ar plasma						
Sample	Voltage U	Current I	Plasmatron power P	Plasma gas flow rate <i>Q</i>	Rubber powder mass flow rate g		
	[V]	[A]	[kW]	[l/h]	[kg/h]		
1	39	550	21.45	5,325	0.05		
2	39	550	21.45	5,325	0.16		
3	39	550	21.45	5,325	1.8		
4	39	550	21.45	5,325	2.29		
5	39	550	21.45	5,325	3.46		
6	39	550	21.45	5,325	4.39		
7	39	550	21.45	5,325	5.76		
8	39	550	21.45	5,325	8.04		

Table 2

Table 1

Sample	Voltage U	Current I	Plasmatron power P	Plasma gas flow rate Q	Rubber powder mass flow rate g
	[V]	[A]	[kW]	[l/h]	[kg/h]
1	49	550	26.95	5,453	0.05
2	49	550	26.95	5,453	0.16
3	49	550	26.95	5,453	1.8
4	49	550	26.95	5,453	2.29
5	49	550	26.95	5,453	3.46
6	49	550	26.95	5,453	4.39
7	49	550	26.95	5,453	5.75
8	49	550	26.95	5,453	8.04

Results and discussion

The rubber powder was decomposed in two different plasma gases: in pure Ar and in mixture of Ar and 3.6% H₂. Plasma pyrolysis was carried out in the function of the rubber powder mass flow rate.

The exemplary absorption spectrum for pyrolysis of the rubber in the Ar plasma was presented in the Figure 4. There were identified infrared bands of $\rm C_2H_2,\, \rm CH_4,\, \rm CO,\, \rm CO_2$ and the common band of $\rm C_2H_2$ and $\rm CH_4.$ The band of H₂O is also visible in the absorption spectrum but it is only a residue of water vapor present in the reactor prior to the experiment.

The presence of H_2 in the outgoing gas could not be verified because of the technical specification of the absorption spectrometer. Although, according to the literature (CHANG et al. 1996), H_2 is present in the outgoing gas.

No bands of NO_x are present in the absorption spectra. No bands characteristic for HCN were found in the spectra, neither. The analysis of the spectra (Fig. 4) confirms that there are no toxic chemical compounds. It means that pyrolysis of rubber in the Ar plasma brings about no undesirable compounds in the outgoing gas. So, there is no need to install any filtration or cleaning systems in the test-stand set-up.

The analysis of the gaseous products in the outgoing gas lets find out that the concentration of C_2H_2 is increasing as the rubber powder mass flow rate grows (Fig. 5). The concentration function of C_2H_2 is monotonically increasing.



Fig. 5. C_2H_2 in outgoing gas for pyrolysis of rubber in Ar plasma in the function of rubber powder mass flow rate (P = 21.45 kW)

The concentration functions of the other identified gaseous products $(CH_4, CO \text{ and } CO_2)$ of pyrolysis of the rubber in the Ar plasma (Figs. 6, 7, 8) are not monotonic.

The analysis of the absorption spectra of pyrolysis of the rubber in the Ar + 3.6% H₂ plasma shows off more numerous products of the process. Except for C_2H_2 , CH_4 , CO and CO_2 , identified also in the spectra of pyrolysis of the rubber in the Ar plasma, also C_3H_8 (propane) and C_4H_{10} (butane) were found for pyrolysis in the Ar + 3.6% H₂ plasma (Fig. 9). Obviously, presence of H₂ in the plasma gas stimulated synthesis of bigger number of hydrocarbons.

According to the analysis of the spectra (Fig. 9), neither toxic nor harmful gases have been identified in the outgoing gas for pyrolysis of the rubber in the Ar + 3.6% H₂ plasma.



Fig. 6. CH_4 in outgoing gas for pyrolysis of rubber in Ar plasma in the function of rubber powder mass flow rate (P = 21.45 kW)



Fig. 7. CO in outgoing gas for pyrolysis of rubber in Ar plasma in the function of rubber powder mass flow rate (P = 21.45 kW); CO concentration is proportional to measured absorbance



Fig. 8. CO_2 in outgoing gas for pyrolysis of rubber in Ar plasma in the function of rubber powder mass flow rate (P = 21.45 kW); CO_2 concentration is proportional to measured absorbance



Fig. 9. Exemplary absorption spectrum of outgoing gas for pyrolysis of rubber powder in Ar + 3.6% $\rm H_2$ plasma (P=26.95 kW)

During the experiment the rubber powder mass flow rate was being changed in the range from 0.05 kg/h up to 8.04 kg/h. The increase of the rubber powder mass flow rate caused the concentration of C_2H_2 , C_3H_8 and C_4H_{10} was increasing (Figs. 10, 11, 12). The concentration functions of the three gases were monotonic.

While the rubber powder mass flow rate was increasing the concentration functions of CH_4 , CO and CO₂ were changing non-monotonically (Figs. 13, 14, 15).



Fig. 10. C_2H_2 in outgoing gas for pyrolysis of rubber in Ar + 3.6% H₂ plasma in the function of rubber powder mass flow rate (P = 26.95 kW)



Fig. 11. C_3H_8 in outgoing gas for pyrolysis of rubber in Ar + 3.6% H_2 plasma in the function of rubber powder mass flow rate (P = 26.95 kW); concentration of propane is proportional to measured absorbance







Fig. 13. $\rm CH_4$ in outgoing gas for pyrolysis of rubber in Ar + 3.6% $\rm H_2$ plasma in the function of rubber powder mass flow rate (P = 26.95 kW)



Fig. 14. CO in outgoing gas for pyrolysis of rubber in Ar + 3.6% H_2 plasma in the function of rubber powder mass flow rate (P = 26.95 kW); CO concentration is proportional to measured absorbance





Vast majority of the products of plasma pyrolysis of rubber is gas (over 99%) (SZUSZKIEWICZ et al. 2001). The analysis of the solid state products has been done by the atomic absorption spectrometry, flame photometry and absorption spectroscopy FTIR. The analysis revealed that solid state products were mainly soot. It contained: chemical compounds with SO₂ group and twelve elements, namely Pb, Zn, Cu, Fe, Mn, Cd, Cr, Ni, Ca, Mg, Na and K.

Conclusions

The carried out research positively verified all the assumed aims. Plasma pyrolysis seems to be a successful method for utilization of rubber waste. Generally, the increase of the rubber powder mass flow rate brings positive effect concerning the increase of production of most of the gaseous products, especially hydrocarbons.

The detailed conclusions according to the experimental research reveal all the benefits of the plasma pyrolysis of rubber. All the rubber powder introduced to the plasma jet was entirely decomposed. As the products of plasma pyrolysis mainly gas and small amount of soot were obtained.

Despite no filters had been applied no toxic compounds, like NO_x , HCN or SO_2 , were identified in the gaseous products of plasma pyrolysis of the rubber.

Application of $\rm H_2$ as a secondary plasma gas had not only a chemical importance for plasma pyrolysis. $\rm H_2$ contained in the plasma gas influenced the increase of the electric current in the plasma jet. Also, $\rm H_2$ stimulated numerical amount and amount of hydrocarbons coming into being during plasma pyrolysis. Concentration of the gaseous products for the Ar + H_2 plasma is bigger than for the Ar plasma.

The increase of the rubber powder mass flow rate did not increase the production of all the gaseous products. The increase of the rubber powder mass flow rate for the Ar plasma made the concentration of C_2H_2 monotonically increased. The concentration of all the other identified products of plasma pyrolysis changed non monotonically.

The increase of the rubber powder mass flow rate in the Ar + H_2 plasma resulted in the monotonic increase of the concentration of C_2H_2 , C_3H_8 and C_4H_{10} and non-monotonic concentration change of all the other identified gaseous products of plasma pyrolysis.

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CHANGES IN THE QUALITY CHARACTERISTICS OF RASPBERRY FRUIT DUE TO MECHANICAL VIBRATIONS AND STORAGE CONDITIONS

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Abstract

The paper presents the results of a study investigating a mechanical impact, in the form of vibrations, on the overall quality of raspberry fruits after harvest, in view of the conditions of their transport to the consumer and the processing plant. The degree of compaction of the raspberry fruit layer was determined during a test carried out under laboratory conditions, and the amount of juice leakage, resulting from the vibrations to which the raspberries were subjected, was measured. It was demonstrated that the quantity (mass) of juice leakage was considerably affected by the mechanical impact, in the form of vibrations, exerted on a layer of raspberry fruits. The leakage was smaller in cooled fruits, and significantly greater in fruits that were not subjected to cooling immediately after harvest, and were stored under ambient conditions.

Introduction

Raspberry fruits are characterized by low durability and rather loose cell/ tissue structure, combined with a high water content. This results in the fact that there are many factors influencing the decrease in durability of raspberry fruits after harvest (GOŁACKI, ROWIŃSKI 2006, HAFFNER et al. 2002, PERKINS--VEAZIE, NONNECKE 1992, ROBBINS, SJULIN 1986). The most important include: the fruit maturity at harvest, time, temperature and other conditions of the post-harvest storage, as well as conditions of the transport to the final consumer or processing plant. Inappropriate handling at the mentioned stages reduces the overall post-harvest quality, which is manifested by appearance of mould, fungi and other micro-organisms. Decay processes take place immediately after harvesting, when the raspberries are already in the packaging, as well as during a shorter or longer transport to the place of final use (consumer, cold store, processing plant) (IDASZEWSKA, BIEŃCZAK 2013, HEIBERG 1988, JAKUBCZAK, UZIAK 2005, RAMSAY 1983). High rating of raspberry fruits for consumption and those intended for freezing or processing depends mainly on the average size (dimensions) of the berries, which is determined in various ways, on their attractive red colour, and on mechanical properties (DOBRZAŃSKI et al. 1994, DOBRZAŃSKI, RYBCZYŃSKI 1995, GOŁACKI, ROWIŃSKI 2006, KUCZYŃSKI, RYBCZYŃSKI 2004, RAPCEWICZ, DANEK 2010, RAMSAY 1983). Earlier observations of the fruit at plantations show a gradual decrease in firmness as well as colour change (darkening) (KUCZYŃSKI et al. 1994, RAMSAY 1983, ROBBINS, SJULIN, 1986, SJULIN, ROBBINS 1987). Raspberry fruits are among the group of most delicate soft fruits, which are sensitive to any mechanical damage associated, among other things, with changing conditions of their storage and transport. At the time of harvest and after it, various biochemical processes take place in the fruits from this group, and, additionally, changes occur in the organoleptic characteristics and intracellular structure during transport to the final consumer

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or processing plant. They are reflected, inter alia, in the destruction of the original shape and dimensions of the fruits, as well as an increased compaction of the layer of fruits, which, in turn, leads to juice leakage, thus lowering the overall consumption and processing value. In the context of preserving the highest possible quality of these fruits particularly their transport is one of the biggest logistical challenges. Inadequate transport conditions, viz. high temperature, too high or low humidity, extended time of transport, and unsuitable means thereof affect the overall perceived quality of the transported raspberry fruits. Changes in the shape and dimensions of both single fruits and the entire volume of the layer, reduced turgor pressure and mechanical strength, as well as compromised flavour, are among possible detriments. It can be assumed that transport of raspberries is a mobile variation of their storage, and, therefore, the same parameters that act on them during storage are responsible for the changes in the transported fruits. In the literature of the subject, be it national or international, there is relatively little information about the influence of vibrations generated during transport on the transported product, including raspberries. The data from literature concern mainly the quantitative losses estimated after the transport process, and therefore the final outcome (RYBCZYŃSKI et al. 2001, THOMPSON 2010). However, during the transport of the fruits minimal displacement of the products takes place as well as their mechanical interactions resulting from shock and vibration of a transport box. During transport raspberries might experience forced influence of mechanical phenomena and accelerated deterioration of their quality, including a reduction of the amount of certain valuable substances. Therefore, it can be assumed that the shocks and vibrations experienced during transport should be recognized as negative factors that may cause acceleration of the overall deterioration process of raspberry fruits (BARRITT et al. 1980, IDASZEWSKA et al. 2014, ROBBINS, SJULIN 1986, ROBBINS, SJULIN 1989).

The aim of the study was to determine the amount of raspberry juice leakage depending on storage conditions after harvest and the intensity of mechanical vibrations. The research was conducted in terms of changes in qualitative and technological features that may occur in the conditions of fruit transport to the consumer and processing plant. The degree of shaking of the raspberry layer was determined in laboratory conditions, determined in various ranges of mechanical vibrations.

Material and methods

Studies were carried out on the fruits of an autumn variety of raspberries, i.e. Polka, collected at a private plantation in the area of Ostrów Lubelski. Raspberries harvest took place in the second half of August. Immediately after harvest, fresh raspberry fruits were placed in a container into layer with the thickness



Fig. 1. The view of a laboratory vibrating shaker

of a = 50 mm, and then subject to a shake for the time of $t_w = 15$ min, using a laboratory vibratory shaker (Fig. 1). Vibratory shaker ANALYSETTE (AS 400 Retsch GmbH) is equipped with an optical amplitude control with an amplitude value label allowing to read amplitude value. Two vibration amplitudes 1 and 2 mm were used in relation to the gravitational acceleration at a vibration frequency of 50 Hz.

The scope of the experiment included the use of various parameters of the process of mechanical vibration impact on the raspberry fruit layer. The total vibration time was 15 minutes, with varying vibration intervals ($t_{id} = 20, 40, 60, 80$), conducted at 3-second intervals. Two groups of fruits were used in the study, viz. raspberries

stored for 24 hours at the temperature of 25°C and those stored for the same period of time in cold store condition at T = 5°C. In the particular stages of research the degree of compaction u (decrease in the thickness of the raw material layer before and after shaking) in mm was determined and quantity (mass) of the juice leakage m_s in g was measured (Fig. 2).

A statistical analysis of the obtained results was carried out in order to obtain a mathematical representation of the change in the thickness of the material layer and juice leakage as a result of the vibrations used.



Fig. 2. Research scheme

Research results and their analysis

On the basis of the results obtained, it can be concluded that in the case of raspberry fruits temperature and the remaining environmental conditions in which the raspberries are stored immediately after harvesting are very important factors affecting the overall consumer-processing quality. This is confirmed by the data on the degree of compaction of the two layers of raspberries, i.e. fruits stored after harvesting at the temperature of 25°C and fruits stored in a cold store (Figs. 3, 4).







Fig. 4. Research results illustrating the degree of compaction of the layer of raspberry fruits cooled immediately after harvesting and stored at the temperature $T = 5^{\circ}$ C along with statistical deviation. The initial thickness of the fruit layer $a = 50 \text{ mm} (\pm 1 \text{ mm})$

The tests carried out in the laboratory are an approximate representation of the vibrations that occur in various forms during the final transport of raspberries to the recipient. The analysis of the research results showed that rapid cooling of raspberries after harvesting determine their increased resistance to mechanical stress in the form of multi-planar shocks, vibrations, and mini displacements.

By comparing the degree of compaction (compression) of the layer of cooled raspberry fruits and that of stored at ambient temperature the differences of the height, when compared with the original height of a = 50 mm, ranged from 25.76% to 64.44% (for $T = 25^{\circ}$ C) and from 14.4% to 41.2% in the case of cooled fruits ($T = 5^{\circ}$ C). The average difference of the value of this parameter between the two test groups of the fruits ranged from 11.3% to 25.9%. With regard to the parameters of vibration to which the test samples were subjected to, the greatest degree of compaction of the layer of fruits was recorded for the longest intervals t_{id} equal to 60 and 80 s, regardless of the vibration amplitude. The data obtained confirm susceptibility of raspberry fruits to mechanical influence, with fruits stored immediately after harvesting at ambient temperature being particularly sensitive in this regard.

Figures 5 and 6 summarize the results of studies concerning the increase of juice leakage from the layer consisting of two respective groups of raspberries, viz. cooled ones and stored at the temperature $T = 5^{\circ}$ C and those stored at the temperature $T = 25^{\circ}$ C, both groups for the period of 24 hours.

The analysis of the obtained data clearly demonstrate that mechanical stress in the form of vibration exerted on raspberry fruits has a large impact on the amount of juice leakage. This effect is smaller in the case of cooled fruits, and much larger in the case of fruits not cooled immediately after harvesting and stored at ambient conditions. The average values of juice leakage, regardless of the parameters of vibration process, oscillate approximately between 1.7 and 4.3%. From the point of view of processing usefulness these are big losses that significantly decrease the desired value and technological quality of raspberries.

With respect to the quality and value of raspberries intended for direct consumption such scale of losses which may arise due to inappropriate storage conditions immediately after harvesting and during transport is not acceptable.

The juice leakage increased in direct proportion to the vibration intervals set up for the experiment. Its greatest value of 370.88 g (as referred to 1 kg of fruits) was obtained for the layer of fruits stored at 25°C and shook with the amplitude of vibration equal to 1 mm. In the case of cooled raspberries (5°C) the value of the leakage was equal to 345.42 g. The difference in respect of these data was 6.86% (Figs. 5, 6). The analysis of data shows that regardless of the test parameters the difference in the amount of leakage from the layer of raspberries cooled after harvesting and those not cooled ranges from 4.92% to 10.05%. A similar trend was noted in relation to the degree of compaction.









The layer of cooled raspberry fruits was compacted to a lesser extent, regardless of the applied vibration, i.e. the frequency and amplitude.

On the basis of the results obtained, mathematical models describing the loss of the thickness of the raw material layer and juice leakage during simulated transport were developed (Tab. 1, 2).

In view of the delicacy of the structure and susceptibility of raspberry fruits to shocks, which were confirmed in the research, several practical principles should be recommended that are necessary to maintain high consumer and processing quality of the fruits. It is therefore recommended to:

 – cool fruits as soon as possible after harvesting and deliver them fast to the final consumer, cold store, or another processing establishment;

 adjust the means of transport to suit demands of the soft fruit group, including raspberries; – make technical improvements to the means of transport through the use of suspended chassis, transport boxes with limited capacity, and minimize the distance over which fruits are transported;

- use partitioned fruit crates, which allow to reduce the thickness of the fruit layer in the box (Fig. 7 – Utility model W.126925) (appropriate patent pending).

Table 1

The regression equations illustrating the nature of changes in the height	
of raspberry fruit layer resulting from varied intervals of the vibration	

	Fruit temperature $T = 25^{\circ}$ C				
$\begin{array}{c} \text{Amplitude of vibration} \\ [\text{mm} \cdot \text{g}^{\text{-1}}] \end{array}$	equation	R^2 (coefficient of determination)			
1	$a = -0.166 \cdot t_{id} + 40.395$	0.999			
2	$a = -0.312 \cdot t_{id} + 42.517$	0.995			
	Fruit temperature $T = 5^{\circ}C$				
$\begin{array}{c} \text{Amplitude of vibration} \\ [\text{mm} \cdot \text{g}^{\text{-}1}] \end{array}$	equation	R^2 (coefficient of determination)			
1	$a = 0.427 \cdot t_{id} + 334.74$	0.960			
2	$a = 0.469 \cdot t_{id} + 321.99$	0.928			

a – height of the fruit layer [mm],

 t_{id} – vibration interval [s].

Table 2

The regression equations illustrating the nature of changes in the amount of raspberry fruits juice leakage resulting from varying vibration intervals

Amplitude of vibration [mm · g ⁻¹]	equation	R^2 (coefficient of determination)
1	$m_s = -0.222 \cdot t_{id} + 48.138$	0.968
2	$m_s = -0.337 \cdot t_{id} + 34.817$	0.661
	Fruit temperature $T = 5^{\circ}C$	
Amplitude of vibration [mm · g ⁻¹]	equation	R^2 (coefficient of determination)
1	$m_s = 0.5451 \cdot t_{id} + 295.4$	0.989
2	$m_s = 0.3936 \cdot t_{id} + 312.6$	0.909

 m_s – amount (mass) of juice [g],

 t_{id} – vibration intervals [s].



Fig. 7. Proposed partitioned box for harvesting, storage and transport of soft fruits, including raspberries; 1, 2, 3 – box walls, 4 – box bottom, 5 – pull-out partitions, 6 – protruding partition handles, 7 – slat supports, 8 – partition mounting slots, 9 – recesses for handles, 10 – limiters

Conclusions

On the basis of the conducted research it can be concluded that:

1. In the case of raspberry fruits very important factors determining the overall consumer-processing quality thereof are the temperature (5 and 25°C) and other environmental conditions (vibration amplitudes 1 and 2 mm, vibration frequency of 50 Hz) in which they are stored immediately after harvesting.

2. When comparing the degree of compaction (compression) u of the layer of raspberry fruits cooled after harvesting and those stored at ambient temperature the differences in the height of fruit layer with respect to the initial height a ranged from 25.76% to 64.44% (for $T = 25^{\circ}$ C) and from 14.4% to 41.2% in the case of the cooled fruits ($T = 5^{\circ}$ C).

3. With regard to the parameters of the vibration used, the greatest degree of compaction of the fruit layer, regardless of the amplitude of vibration (vibration amplitudes 1 and 2 mm, vibration frequency of 50 Hz), was observed for the longest intervals t_{id} equal to 60 and 80 seconds respectively.

4. Mechanical impact on the fruits in the form of vibration has a significant impact on the quantity (mass) of the juice leakage. This effect is smaller in the case of cooled fruits, while in the case of the fruits not cooled after harvesting and stored at ambient conditions the impact is much larger.

5. The average values of juice leakage (recognized as loss), regardless of the parameters of the shaking process, range from 30 to 35%.

6. The test results confirmed that from the point of view of the general quality and technological value of raspberry fruit, it is advisable to cool them as soon as possible after harvesting and storage in a thin (less than 5 cm) layer, which is possible of the proposed box structure. In addition, it is recommended to use flexible and cushioned chassis means of transport, guaranteeing the greatest possible reduction of mechanical vibration when transporting fruit to the consumer or processing plant.

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RESEARCH ON INNOVATIVE FOILS FOR AGRICULTURAL APPLICATIONS

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Abstract

The paper presents the results of a study investigating the impact of reduced graphene oxide (RGO) on selected mechanical and functional properties of LDPE foils. The foils were made by blow extrusion, with different amounts of RGO added to the granules prior to the extrusion process. The mechanical properties of foil samples were assessed in a static tensile test, and their bacterial resistance was tested. The impact of RGO on antibacterial interactions and the desirable mechanical properties of the foils was analyzed. The results of this study supported the selection of the most advantageous solution for industrial applications.

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Introduction

Currently, due to EU requirements and the generally dynamic development of polymer processing, entrepreneurs producing plastic foils are looking for increasingly unusual and innovative solutions for these products (SIWEK et al. 2010, DUDA 2003). As an example, can be given materials with the content of additives that change their mechanical, physicochemical, structural properties, various types of blends with the addition of compatibilizers, polymers enriched with antimicrobial additives, or affecting their easier and faster degradation, and many others (WENDA et al. 2016, WOLSKA et al. 2017, JAŁBRZYKOWSKI et al. 2018b, LIYA et al. 2013, ŁOPACKA 2013. MALINOWSKA-PAŃCZYK et al. 2010).

One of the important activities in the field of packaging foils is the production of products with antimicrobial properties, while maintaining their maximally favorable mechanical characteristics (FALKIEWICZ-DULIK, KOWALCZYK 2016, JURASZEK, GRZESIAK 2008, ZIELECKA et al. 2012, GNIAZDOWSKA et al. 2015). In order to obtain antimicrobial properties, the material is enriched with e.g. gold, silver or copper nanoparticles (JALBRZYKOWSKI et al. 2018a, LIYA et al. 2013, ŁOPACKA 2013, MALINOWSKA-PAŃCZYK et al. 2010, MALINOWSKI 2015, JALBRZYKOWSKI et al. 2018b, JALBRZYKOWSKI 2019).

Currently, in the context of antimicrobial interactions, is observed a large interest of researchers in graphene oxide (HEBDA, ŁOPATA 2012, XU et al. 2014, YU et al. 2014, YANG et al. 2012, KIM et al. 2010). As it is commonly known today, graphene oxide is characterized by, among others extremely favorable mechanical (high mechanical resistance, tensile strength, considerable flexibility), electrical, conductive and bactericidal properties, biocompatibility etc. It is worth noting that this is a compound that combines many beneficial features and useful properties not found in such a rich combination, for any other substance. Certainly, this fact has contributed to many scientific works and a number of research and application experiments involving this substance (LI, KANER 2008, STANKOVICH et al. 2006, BAI et al. 2010, BLAKE et al. 2007, SCHWIERZ 2010)

Taking the above mentioned into consideration, this paper describes selected studies of polyethylene foils enriched with reduced graphene oxide (RGO). RGO was added to the polymer to check its effect on antimicrobial interactions and change of the mechanical properties of the foil produced.

Materials and research methodology

LDPE granules and reduced RGO graphene oxide were used for the tests (commercially available). Three-layered foils were produced by blow extrusion before testing. Due to the fact that the main purpose of the research was to assess the impact of RGO concentration on the analyzed characteristics, plastic foils were made with the same parameters of the extrusion process, treating the values of these parameters as a constant quantity, having no impact on interesting issues. The following materials were prepared for testing: clean LDPE foil (without the addition of graphene oxide – Fig. 1*a*), foil with the addition of 2 g/l (Fig. 1*b*) and 3 g/l (g of graphene oxide / liter of granulate) (Fig. 1*c*). All foils were 40 μ m thick (5 μ m skin layers, 30 μ m core, each layer was built the same).



Fig. 1. View of prepared foils: a – without RGO, b – with 2 g/l of RGO, c – with 3 g/l of RGO

The main purpose of the study was to evaluate the foils in terms of selected mechanical and microbiological properties. The assessment of mechanical properties was carried out using a Zwick/Roell testing machine in accordance with the methodology for testing tensile properties of plastic foils (ASTM D882). *Escherichia coli* (*E. coli*) was used to assess bactericidal properties. Before testing, the foil samples were sterilized, weighed and then exposed to *Escherichia coli* culture. It consisted of immersing them in BHI broth (from Oxoid) in which the *E. coli* bacterial strain with 10^5 CFU/ml inoculum (CFU – bacterial colony forming unit) was suspended. Then they were placed in an incubator (temperature 37° C) under aerobic conditions. The samples were exposed for 72 hours to the bacterial strain. Bacteria were identified and propagated in accordance with applicable standards in microbiological diagnostics (Cryobank Mast Diagnostica). During the process, the number of bacterial cells was measured after 24, 48 and 72 hours. Tests were repeated three times.

Research results and discussion

Figure 2 presents selected results of studies on the impact of incubation time and RGO concentration on bacterial colony development. The obtained results indicate that irrespective of the RGO concentration, a longer incubation time of the bacterial colony means its larger growth (Fig. 2*a*). At the same time,



Fig. 2. The number of bacterial cells depending on: a – colony incubation time (general trends for all samples), b – type of foil tested (incubation time 72 hours)

the higher the RGO concentration, the lower the bacterial colony growth for samples incubated for the same amount of time (Fig. 2b). Generally, the results obtained confirm the beneficial effect of graphene oxide on the antibacterial properties of LDPE. This is a very beneficial information, especially since this type of foil is a common material for packaging food products. It should also be remembered that despite the antimicrobial nature of such material, its beneficial effect is probably effective for a limited time. The point is that the longer the material is exposed to *E. coli* strains, the larger the clusters of these bacteria colonize on its surface. On the other hand, if *E. coli* settled on pure LDPE, the growth of bacterial colonies would be even greater. Generalizing this paragraph of the work, it can be stated that the addition of RGO to foil material certainly brings benefits in the form of increased resistance to bacterial effects, e.g. packaging for food products.



Fig. 3. Breaking force value depending on the type of foil tested: 1 - LDPE, 2 - LDPE + 2 g/l, 3 - LDPE + 3 g/l

Figure 3 shows the impact of RGO on the mechanical characteristics of LDPE. The graph presented illustrates the overall positive effect of graphene oxide on foil behavior during stretching. However, in the case of foils with RGO content of 2 g/l, a significant increase in relative elongation was obtained, while in the case of 3 g/l contents, a slightly smaller increase in elongation, but an increase in the value of the force causing the foil to break. This means that RGO generally improves the mechanical properties of the foil. In addition, it is likely possible to manipulate the ranges of individual features. This aspect also has its significance, because in addition to the fact that while maintaining the current thickness of the foil it is possibile to get more durable packaging and also produce thinner foils basing on the fact that thanks to RGO it has better mechanical characteristics. This creates the possibility of more economical foil production by producing thinner products. Economical production means higher sales profits. In addition, due to more economical use of the base material, we affect the environmental protection as a result of reduced exploitation of fossil sources being a raw material for production, among others technical plastics.

Conclusions

The paper presents selected results of testing the LDPE foils useful properties. The foils were enriched with reduced graphene oxide to improve its mechanical properties and assess the impact of RGO on the antibacterial properties of the foil. The research and analysis of the results obtained allowed to formulate the following general conclusions:

1. A favorable effect of RGO content on the examined features of LDPE foil was found. In general, it can be seen that the higher the RGO concentration, the better the antibacterial resistance and better mechanical characteristics of the foil.

2. In relation to studies on the impact of RGO on antibacterial interactions, it can be seen that the higher the RGO concentration, the smaller the bacterial $(E. \ coli)$ colony growth, the longer the foil is exposed to bacterial interactions, the unfortunately the larger the bacterial colony growth. However, the presence of graphene in the film is no doubt a barrier for the bacteria.

3. In relation to testing the mechanical properties of the RGO enriched foil, it can be stated that the prepared samples have revealed the situation when it is possible to achieve either greater material elongation with slightly lower tensile strength and breaking strength, or less elongation with general improvement of the material's mechanical properties. This means that with a properly prepared polymer composition, it is possible to control the ranges of selected mechanical properties of the foil.

4. By improving the mechanical properties of the foil, in this case, by enriching it with an RGO nanoadditive, the possibility of producing packaging materials with increased mechanical properties arises. At the same time, it is possible to produce foils with similar mechanical properties, but thinner (however, this has not been studied). The production of thinner films saves the polymer raw material, and thus the technology is more environmentally friendly, because it reduces the burden on natural deposits, which are the basis for the production of, among others technical polymers.

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METALLIC ALLOY WITH SHAPE MEMORY – SELECTED PROPERTIES AND ENGINEERING ASPECTS

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Abstract

In the case of shape memory alloys (SMA), a form to which a material is expected to return during heating can be repeatedly programmed, whereas other related properties can be individually adjusted. It was found that most producers of commercial assortment based on SMA as well as traders are seldom willing to lift the veil of secrecy on this topic. In the context of own experimental studies, the authors made a reference to the technical aspects of some post-treatments of a Ni-Ti alloy with a view to further practical application, e.g. the design and construction of machinery and structures with the involvement of SMA. For these purposes, high-temperature shape setting trials were carried out using various parameters of heat treatment with no secrecy surrounding the procedures applied. Some of the tested parameters proved effective, whereas some were less useful. Following the activation of the reverse transformation by heating, a somewhat different behaviour was observed, and simultaneously one of the crucial material temperatures was determined. The paper as a whole is reported from a specifically engineering/technical point of view, which is continuously emphasized in the content of the presented article.

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Introduction

In modern engineering shape memory materials (SMM) constitute the most important type of materials among the so-called intelligent materials, which in turn fall into a broader subgroup of active/multi-functional materials. It is noteworthy than in contrast to typical construction materials, the functional materials play specific roles. In this case, other than the ability to transfer mechanical loads and stresses. A material can only be considered as a SMART material if it changes its properties, parameters or behaviour under the influence of a specific of external, non-mechanical factors (KAMILA 2013, KAUSHAL et al. 2016). The reaction inside the material is reversible.

It has been found that functional materials happen to be very advanced materials developed to provide a specific property or function. In some cases these materials exhibit a combination of different features within one structure. Metallic nickel-titanium (Ni-Ti) alloys with shape memory effect (SME) reveal the following main properties: free shape change associated with the one-way and two-way shape memory effect (OWSME and TWSME) and the phenomenon of pseudo-elasticity (PE). These phenomena can be revealed owing to different paths of thermo-mechanical loads that these materials are exposed to (VAN HUMBEECK 2001, MOHD JANI et al. 2014, NARESH et al. 2016). Other related functional properties include: restrained shape recovery, shape recovery inhibited by an opposing force during heating, high damping capacity and fatigue resistance in the martensitic state, as well as biocompatibility (VAN HUMBEECK 2001, KUCHARCZYK et al. 2011, MOHD JANI et al. 2014).

A vast and interesting array of their properties creates a platform for possible industrial applications. These materials available in a wide assortment (e.g. plates, wires, rods, tubes, films) are used, among others, in electronics, telecommunication, industry, security, sports, clothing, home technology and medicine. In some applications, such materials can act simultaneously as a sensor and an actuator or the actuator itself. The structure of SMM can be porous or nanometric. Furthermore, such materials are used to produce very small components applied in microscopic devices-mechanisms known as microelectromechanical systems. The manufacturing processes of products involving SMM are already based on 3D printing technologies.

An important thing to note is, especially from a practical perspective, that some characteristics related to the SME in shape memory alloys (SMA) can be adjustable (WANG et al. 2004, SADIQ et al. 2010, KUŚ, KŁYSZ 2012, BRAZ FERNANDES et al. 2013, VILLA 2015). Moreover, the shape to which the material is expected to return during its heating can be repeatedly programmed, depending on the application purposes/needs. For these purposes one of the so-called post-/final treatments can be applied to the SMA. An appropriate selection of material deformation parameters and subsequent annealing, in particular, give the possibility to influence transformation temperatures and their sequences. It is of primary importance to understand the structure, as well as the properties of SMA, are additionally influenced by other factors like e.g. alloy composition, repeated cycling (DUERIG, PELTON 1994, KUŚ 2010, SADIQ et al. 2010, MOHD JANI et al. 2014).

Although various ready-made SMA based products are already commercially available, there is usually a need to change (or tune) certain material characteristics to a specific property or function, e.g. in the case of individual projects and construction of miscellaneous elements, devices or structures with the involvement of SMA. Unfortunately, most producers of SMA-assortment or traders are seldom willing to lift the veil of secrecy on this topic. The authors of the paper experienced this state of affairs on numerous occasions, not to mention the manufacturers keep the processing history in the materials sold undisclosed. Thus, it comes to the conclusion that some issues related to final processing remain unclear and deserve to be more systematically reported. The questions that pose themselves are as follows: how to fix the desired shape of the material – is it a matter of appropriate heat treatment, should the temperatures of structural transformations be obtained, should the sequences be altered? There are not many literature positions currently available, especially in the category of complete engineering technological information about the above-mentioned steps. A contribution to a different extent in this field was made by some researchers (SUZUKI 1998, LIU et al. 2008, RAO et al. 2015, HEIDARI et al. 2016, JAHANBAZI ASL et al. 2019). Because SMM are still classified as engineering materials with high innovation potential, the authors of this work imply that this sort of insightful data can be useful for machine designers, various examples of mechanism employing SMA in fields such as mechanics, mechatronics and robotics, as well as for students or enthusiasts.

In the context of own experimental research, the authors made reference to technical aspects of some post-treatments of a Ni-Ti alloy with a view to further practical application, e.g. design and construction of machinery and structures with the involvement of SMM. For this purpose, attempts concerning the high-temperature shape setting were conducted using various parameters of annealing treatment. The paper as a whole is reported from a specifically engineering/technical point of view, which is continuously emphasized in the content of the presented article.

Materials and methods

Commercially available Ni-Ti arc wires were used throughout the experimental works (Fig. 1). It was determined that the temperature A_{f} defined as the end of the reverse transformation from martensite to austenite, falls well below



Fig. 1. Shape and as-received state (PE behaviour at room and lower temperature)

the ambient temperature. Thus, the materials in an as-received state exhibited PE at a room temperature and below.

As already mentioned above, the shape to which the material is expected to return during the SME can be programmed. Then, depending on the purpose, SMM may have different final shapes (e.g. straight elements, products in the form of springs or other profiles). The aforementioned information is the reason behind the implementation of annealing treatment, which applicable temperature range varies between 300 and 600°C, at adequate time intervals (SUZUKI 1998, LIU et al. 2008, RAO et al. 2015, STORTIERO 2015, HABERLAND, ELAHIMIA 2016, HEIDARI et al. 2016). The use of higher temperatures, during the heat treatment, is reported by JAHANBAZI ASL et al. (2019). Moreover, a contoured form-platform (either metallic or ceramic) is necessary to give shape that is to be desired. Apart from shaping up a material, the platform's main task is to block expanding or shrinking materials during heating from the martensitic state.

The shape setting form depicted in Figure 2 was used to perform the experiments. As shown in the picture, its circumference bears a resemblance to a fish, which was achieved by profiling a wire and then squeezing it between the nuts of the screws attached to a steel sheet. Then the hand-made platform was placed in a furnace.

The experiments consisted in carrying out heat treatment on SMA at six different temperatures over different periods of time. This paper presents the results obtained at two selected temperatures: T=400 and 475°C; for times: t=15 and 45 min. For this purpose, a laboratory muffle furnace (produced by Czylok Company, Poland) was used. After the heat treatment processes were concluded, the authors proceeded to check the accuracy with which the shape was mapped in relation to the set shape of the platform. The wire samples were



Fig. 2. Shape setting form used in experiments

allowed to cool in the open air of the laboratory, once removed from the chamber of the furnace. The assortment had been prepared accordingly, so a new wire was always available for a new heat treatment regime. The simultaneous stage of the experiments conducted involved the implementation of SME as the result of preliminary cooling of the wire material, then deformation and finally its heating. It was an attempt to determine the range of crucial temperatures, in which the tested material behaved differently (displaying OWSME, PE and TWSME). The shape recovery process was of primary interest, hence thoroughly monitored and an attempt was made to establish A_f

There are obviously more advanced laboratory methods for assessing the properties of SMM, most notably their metallic subgroup (MOHD JANI et al. 2014, TURABI et al. 2016). Differential scanning calorimetry (DSC) is extensively used to characterize the thermal features of SMA. Currently access to this type of apparatus is rather wide enough, and cost performance measurement – not that high. Thermal properties, including the phase transformation temperatures, play a crucial role in determining the areas, in which basic SMA phenomena occur. During controlled heating up or cooling down of a material sample in DSC method the heat flow is measured. In our study, phase transformation from the low-temperature martensite to high-temperature austenite was complementary analyzed by DSC on the device Netzsch DSC 204 F1 Phoenix. In this case, for comparison purposes, determination of the A_f temperature was of interest. A small piece of sample, namely 3.5 mg in mass, was prepared for DSC testing, whereas the typical heating and cooling rates were 10 K/min.

Results and discussion

Figures 3 and 4 show the state and behaviour of the material after annealing at 400°C for 15 min. As seen below, after removing from the experimental platform the fixed shape of the material differs from the one set initially. It follows that the heat treatment parameters used proved to be inappropriate for the purposes of preserving the shape defined by the platform. Nonetheless, the high-temperature shape obtained was further employed to characterize the material during the implementation of the SME.



Fig. 3. Material shape fixed after annealing at 400° C for 15 min



Fig. 4. Material behaviour after annealing at 400° C for 15 min

Note that when a new shape is prepared for fixation, an Ni-Ti alloy should initially consists of low-temperature martensite phase. Then it gets easilydeformable, owing to the presence of martensite – its state resembles plasticine or a soft solder wire. In the opposite case (austenite as the starting phase in a material), it is still possible to shape a material on the form. However, it becomes more difficult.

The experiments have shown that in the cooling temperature range from +5 to -10°C the material is in a martensitic state, which makes it highly ductile. The heating range through the temperature (80÷21.5°C) revealed full recovery of the preserved shape (OWSME). The dynamics of the recovery reaction significantly dropped above the threshold of 24°C, while it was found that the material exhibited PE at ambient temperature.

Figures 5, 6 and 7 show the shape and behaviour of the material after annealing at 475°C for 15 min. As can be seen below, after removing from the experimental form the shape of the material is compatible with the one previously set.

The experiments have shown that in the cooling temperature range from +20 to -5° C the material is in a martensitic state. The heating rate through the temperature ($75\div35^{\circ}$ C) revealed full recovery of the fixed shape. Below the threshold of 34°C only partial recovery of the preserved shape was observed, whereas the temperatures below roughly 27° C inhibited the material from returning to its high-temperature shape (Fig. 7).



Fig. 5. Material shape fixed after annealing at $475^{\rm o}{\rm C}$ for 15 min



Fig. 6. Material behaviour after annealing 475°C for 15 min



Fig. 7. Detailed views in material behaviour after annealing at 475°C for 15 min

While annealing at 400°C for 15 minutes may be inefficient to fully preserve the desired shape, an increase by 45 minutes may be sufficient to facilitate this. Figures 8, 9, 10 show the state and behaviour of the material after annealing at 400°C for an extended period of time. As evidenced, after its removal from the platform, the shape of the material seems to be much more compatible with what was intended, especially in direct comparison with what was observed during 15 minutes of heating (Fig. 3).



Fig. 8. Material shape fixed after annealing at $400^{\rm o}\mathrm{C}$ for $45~\mathrm{min}$



Fig. 9. Material behaviour after annealing at $400^{\circ}\mathrm{C}$ for $45~\mathrm{min}$



Fig. 10. Detailed views in material behaviour after annealing at 400° C for 45 min

Analogically, the high-temperature shape obtained was further employed to characterize the material during the implementation of the SME. Unlike the previous case, the material no longer displayed PE behaviour at room temperature (in this case estimated at 27.5°C). Following the deformation at this temperature, only partial (incomplete) recovery of shape memory can be observed (Fig. 9).

Figure 10 shows in detail the behaviour of the material in the context of the implementation of SME. The experiments have shown that in the cooling temperature range from +10.5 to -4.5°C the material is in a martensitic state. The heating rate through the temperature ($70 \div 42.3$ °C) revealed full recovery of the fixed shape. Around the threshold of 40°C only partial recovery of the preserved shape was observed, whereas the temperatures below roughly 31.5°C inhibited the material from returning to its high-temperature shape.

While carrying out the experiments related to the phenomenon of shape recovery at 400°C over the course of 45 min, the authors noticed that upon completion of several full cycles of transformations, the material began to show the first symptoms of the TWSME (Fig. 11). This scientific phenomenon is predicated upon the fact that the changes in the shape of the material (which in this case are less noticeable than for the OWSME) can be observed both during heating and cooling. The literature sources available to the authors link the above-mentioned observation with the introduction of well-thought-out modifications to the material microstructure during the SME cycles.



Fig. 11. Material behaviour after annealing at 400° C for 45 min. It shows symptoms of TWSME

In reference to the behaviour of SMA during SME, the phenomenon of phase transformation temperature hysteresis occurs and should be taken into consideration. The experiments proved that it is possible to modify the reaction temperatures of a material to suit its specific purpose (in a controlled manner). This is the reason why fairly simple heat treatment is carried out with different parameters. The work presents the results obtained for two selected temperatures, 400 and 475°C; for times, 15 and 45 min, whereas it is important to note that the heat treatment processes were carried out for other values of temperature between 400 and 535°C. Some of the tested parameters proved effective, whereas some were less useful. The microstructure of the material examined changed in the aftermath, especially if pre-deformed.

As was mentioned earlier, phase transformation from the low-temperature martensite to high-temperature austenite was analyzed by DSC method. Thus, for confirmation purposes, determination of the A_f temperature was of interest. Figure 12 presents the data received from the calorimetric measurement for a sample during its heating cycle, which was previously annealed at 475°C for 15 min.



Fig. 12. DSC heating curve obtained for material after annealing at 475° C for 15 min

As shown in above figure, one endothermic peak was recorded after heating regime what clearly means that during heating the reverse transformation from martensite to austenite occurred. From the obtained DSC profile the phase transformation temperatures can be deduced – by means of the method of tangent lines and their intersections. This approach is generally accepted in the scientific community dealing with SMA as being sufficiently accurate. The discrepancy between the values of temperature A_f in relation to its previous behavior during the implementation of the SME can be explained by the lack of external load as the calorimetric measurements were done (Memry Corporation).

Summary

SMM, including metals and their alloys, fall into the category of SMART materials. According to one of the available definitions, these materials are featured by the ability to change their properties, parameters or behaviour under the influence of external factors. Among many SMA, binary Ni-Ti present an interesting set of functional properties. They create big possibilities for new and creative practical applications.

Seemingly uncomplicated heat treatment of Ni-Ti based SMA gives an opportunity to influence selected characteristics associated with the SME and the shape to which the material is expected to return. In the context of own experimental research, the authors made reference to selected technical aspects of some final treatments of a Ni-Ti alloy with a view to further practical application, e.g. design and construction of machinery and structures with the involvement of SMM. In this case, usually there is a need to regulate (or modify) certain material characteristics to a specific function and SMM make it possible. This interesting feature distinguishes such materials from conventional structural materials with precisely predefined properties, to which the engineers must adapt to. It is important to note that while it is desirable for the above-mentioned needs, instability of SMA functional properties (e.g. transformation temperatures) in some cases can prove to be a problem.

The authors of the paper are convinced that the available literature sources providing data on the above-mentioned topics are insufficient, especially regarding the access to engineering technological information, including procedures in details, practical advices and tips from measurements, or observations. Also, there are rather not many reports that contribute to this field. On the downside, these articles are sometimes heavy-going for an ordinary person interested in SMM. Furthermore, such people hardly every have expertise in the topic and expect more professional guidance. The authors believe the information they provided could prove to be useful for machine designers, various examples of mechanism employing SMA in fields such as mechanics, mechatronics and robotics, as well as enthusiasts.

It turns out that there are already many sources commercially offering SMART materials including SMA. For the moment, the most accessible way to purchase such materials is via foreign online platforms (and then process them on one's own). Their offer usually includes wires or springs of different sizes. Therefore, if a reader takes an interest in the topic discussed, our work could serve as guidance, which justifies the purpose of this research. Finally, it is worth to emphasize that the popularity of SMM is not in decline, despite being on the market for many years, considering both research and utilization potential (MOHD JANI et al. 2014, NARESH et al. 2016).

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