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THE INFLUENCE OF GRAPHITE ADDITION ON TRIBOLOGICAL PROPERTIES OF POLYLACTIDE (PLA)

WPŁYW DODATKU GRAFITU NA WŁAŚCIWOŚCI TRIBOLOGICZNE POLILAKTYDU (PLA)

Key words: coefficient of friction, wear, temperature, biodegradable polymers.

Abstract: Plastics are widely used due to their numerous advantages. Unfortunately, most of their types do not decompose quickly in the natural environment, causing environmental pollution. In order to counteract the problem of waste, there is a growing interest in plastics that degrade under the influence of the natural environment. The authors of this article are trying to use biodegradable plastic for sliding elements. Polylactide (PLA) is a biodegradable, environmentally friendly polymer; however, it has a high wear and friction coefficient when working with steel. It was decided to check whether the addition of 10% graphite (a commonly used, environmentally friendly modifier) would improve the tribological properties of PLA. Using a pin-on-disc station, the coefficient of friction, wear and temperature of the sample were determined depending on the speed of cooperation and pressure. The addition of graphite significantly reduced the linear wear of the composite in a wide range of parameters (sliding speed and pressure), slightly decreased the coefficient of friction and slightly increased the temperature of the composite.

Słowa kluczowe: współczynnik tarcia, zużycie, temperatura, polimery biodegradowalne.

Streszczenie: Tworzywa sztuczne ze względu na liczne zalety są bardzo chętnie stosowane. Niestety większość ich rodzajów nie ulega szybkiemu rozkładowi w środowisku naturalnym, powodując zanieczyszczenie otoczenia. W celu przeciwdziałania problemowi odpadów rośnie zainteresowanie tworzywami, które ulegają degradacji pod wpływem oddziaływania środowiska naturalnego. Autorzy niniejszego artykułu prowadzą próby zastosowania biodegradowlnaego tworzywa sztucznego na elementy ślizgowe. Poliaktyd (PLA) jest biodegradowalnym polimerem przyjaznym środowisku, jednak podczas współpracy ze stalą ma duże zużycie i współczynnik tarcia. Postanowiono sprawdzić, czy dodatek 10% grafitu (powszechnie stosowany, nieszkodliwy dla środowiska modyfikator) poprawi właściwości tribologiczne PLA. Przy użyciu stanowiska typu *pin-on-disc* wyznaczono współczynnik tarcia, zużycie i temperaturę próbki w zależności od prędkości współpracy i nacisku. Dodatek grafitu znacząco zmniejszył zużycie liniowe kompozytu w szerokim zakresie parametrów (prędkości poślizgu i nacisku), a w niewielkim stopniu zmniejszył współczynnik tarcia oraz nieznacznie zwiększył temperaturę kompozytu.

INTRODUCTION

Plastics, due to their numerous advantages (e.g., relatively low price, ease of forming, corrosion resistance, low specific gravity), are widely used in industry, e.g., production of packaging, electronics, machine elements or medical equipment. **[L. 1]**. Sliding elements are also made of plastic. Their main advantage is that they do not require lubrication when working with metals, which makes them, among others, environmentally friendly.

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Unfortunately, the use of plastics is associated with the generation of waste harmful to the environment. Items made of polymeric materials need 100 to even 1000 years to decompose in the environment **[L. 2]**. Additionally, they are a source of microplastic that gets into human and animal organisms together with water **[L. 3]**. The annual sum of plastics produced in 2020 amounted to 367 million tonnes worldwide and 55 million tonnes in Europe **[L. 4]**. For this reason, work is being carried out to obtain "environmentally friendly" materials. Materials referred to as such are made of raw materials of natural origin or are more likely to be recycled. However, the most environmentally beneficial are biodegradable polymers.

Biodegradable polymeric materials degrade under the influence of atmospheric conditions or microorganisms to low-molecular products that do not pose a threat to the environment. Biodegradables are those polymers that are either formed in nature (natural polymers) or are analogues of natural polymers. Therefore, polymers made from renewable raw materials are generally biodegradable **[L. 5]**. Biodegradable polymers currently produced include TPS, PHA, PHB, PHO, PCL, PBS, PBAT and PLA **[L. 6]**.

Sliding elements made on the basis of biodegradable polymers would be very environmentally friendly. Firstly, there would be no need to use a lubricant in the node where they would be used, and it is estimated that in the European Union alone, approximately 600,000 tonnes of lubricants end up in the environment every year **[L. 7]**. Secondly, the unnecessary sliding element would not be waste lying in a landfill but would be biodegradable. Thirdly, consuming products would also biodegrade instead of being left in the environment.

Despite the fact that biodegradable polymers have long been of interest to researchers **[L. 8]**, very little information was found on the tribological properties of biodegradable polymers. Běhálek and his team proved that adding fibres of natural origin to PLA reduces the value of the static and kinetic friction coefficient **[L. 9]**. Karalus et al. determined the tribological properties of polyurethanes with different structures and content of rigid segments **[L. 10]**. These materials are used in medicine (e.g. artificial heart components, intervertebral disc implants) due to their strength, elasticity, resistance to fatigue, biocompatibility and biotolerance. It has been shown that the increase in the content of rigid segments in PUR leads to an increase in density and hardness, adversely affecting the material's tribological properties.

Due to the lack of information on the tribological properties of biodegradable plastics, work began with polylactide (PLA), which is one of the most popular biodegradable plastics. Polylactide is a thermoplastic linear polyester **[L. 11]**. It is biocompatible, rigid, easy to process and obtained from renewable raw materials **[L. 12]**. Bottles made of PLA decompose after 75–80 days **[L. 11]**. In addition, it is very well suited for 3D printing using the FDM method (a very popular, cheap and uncomplicated incremental technology). Its disadvantage is the easy sorption of water, meaning it must be dried before processing. The disadvantage is also a relatively high density (1.25 g/cm^3) and high polarity, which does not allow good adhesion in multilayer structures to non-polar polymers (PE and PP) **[L. 11]**.

PLA, in cooperation with steel, has a high coefficient of friction and wear. In addition, this material has low-temperature resistance (glass transition temperature is only 55ºC) **[L. 13]**. Due to the need to improve the tribological properties, graphite was added **[L. 14]**. Due to its layered structure, graphite is used as a solid lubricant or, in powdered form, is an important component of plastic lubricants. Importantly, it is also environmentally friendly and compatible with nature-friendly PLA. Graphite has a very good thermal conductivity **[L. 15]**, so it was hoped that its addition would help dissipate heat from the friction junction.

The tested composite was patented **[L. 16]** and pre-tested. During the tests, it was observed that with the increase in graphite content in the composite, its consumption significantly decreased when working with C45 steel. Compared to unmodified polylactide, the addition of 1%, 5% and 10% of graphite reduced the wear of the composite by 26%, 33% and 65%, respectively. These results turned out to be so promising that it was decided to extend the research. This publication describes the results of this extension of friction and wear tests for different values of pressure and speed in the combination.

MATERIALS AND METHODS

The tests were carried out on a pin on the disc stand. The sliding pair consisted of a disc made of C45 steel with a hardness of 40 HRC and roughness (determined by the Ra parameter) in the range of 0.36 µm – 0.52 µm and a plastic/composite mandrel on its matrix. The temperature of the sample during the cooperation was measured on its side surface using a pyrometer.

The plastic used was polylactide (PLA), and graphite was used as an additive to PLA. The pins were produced on a 3D printer using the FDM method.

Due to the fact that the market did not offer the material for the printer (filament) in the form of PLA with the addition of graphite, the filament was made in-house. An amount of 900 g PLA Ingeo ™ 2003D was mixed with 100 g graphite with a grain size < 40 µm using a ZAMAK MERCATOR WG 150/280 laboratory mill to give a final masterbatch concentration of 10%.

The process was carried out at a temperature of 210°C for 12 minutes, obtaining the initial homogeneity of the master batch. Then, using the WANNER C17.26sv mill, the material was granulated. The obtained granulate was diluted during extrusion of the stream with re-granulation on a twin-screw extrusion line with a HAAKE Rheomex OS profiled head and then dried overnight at 40°C. From the obtained granulates, a filament with a diameter of 1.75 mm was extruded using a HAAKE Rheomex OS single-screw extruder. The extrusion process temperatures were 170°C/210°C/185°C/170°C from the feed zone to the die, respectively.

The samples were printed on the Zortrax M300 Dual printer with the parameters given in **Table 1**.

Table 1. Printing parameters of plastic pins
Tabela 1. Parametry wydruku próbek z tworzywa sztucznego

Nozzle diameter	0.4 mm
Printing temperature	200°C
Table temperature	60°C
Layer height	0.10 mm
Number of full layers top/bottom	16/16
Number of strokes	3
The style of the top and bottom layers	Concentric
Fill style	Rectilinear
Fill percentage	16%
Cooling	100%
Print speed	30 mm/s

When determining the motion characteristics of the sliding pairs, the rotational plan was used (**Table 2**). The experiment plan was defined for two variables (on five levels): pressure p (minimum value equal to 0.1 MPa and a maximum value equal to 0.3 MPa) and sliding velocity v (minimum value equal to 0.33 m/s and a maximum value equal to 1.5 m/s).

Table 2. Research program obtained by means of a rotational plan
Tabela 2. Program badań uzyskany za pomocą planu rotalnego

No.	p [MPa]	V [m/s]
1	0.100	0.915
2	0.129	0.501
3	0.129	1.329
4	0.200	0.330
5	0.200	0.915
6	0.200	0.915
7	0.200	0.915
8	0.200	0.915
9	0.200	0.915
10	0.200	1.500
11	0.271	0.501
12	0.271	1.329
13	0.300	0.915

RESULTS

The obtained results were used to calculate the non-linear regression function. Using the obtained non-linear regression equations, surface plots were created. Due to the fact that the graphs are generated from the regression equation, friction and wear achieved unrealistic results in some areas (e.g., coefficient of friction 1.2 or wear 0).

In the case of regression for the sample's intensity of wear and temperature, a satisfactory fit was obtained for the measurement results. Changing the form of the function, e.g. to a higher degree polynomial, slightly increased the value of the coefficient of determination R^2 and significantly complicated the regression equation. For this reason, a second-degree polynomial was chosen for the regression equations. Most likely, the dependence of wear intensity and sample temperature on speed and pressure is more complex and cannot be well described by mathematical functions. Even though the degree of fit was not more than satisfactory, the graphs obtained on the basis of regression were sufficient to achieve the purpose of the research. They made it possible to determine whether the addition of graphite affects the intensity of wear and the temperature of the sample in a wide range of parameters – speed and pressure.

The tests measured the coefficient of friction, wear and temperature, so the results section is presented in three sections.

Coefficient of friction

The results of the friction coefficient tests are shown in **Fig. 1**. To improve the readability of the graphs, the axis of pressures is presented so that larger values are closer to the reader and smaller ones are further away. The obtained regression equations were in the form of second-degree polynomials; for such functions, the values of the coefficient of determination R^2 were obtained: 0.97 for the PLA-steel pair and 0.95 for the PLA+C-steel pair. Based on the R^2 value, it can be assessed that the model was very well matched to the results obtained experimentally. The obtained regression equations had the following form:

For PLA-steel pair: $\mu = 1.81 - 2.24p - 1.66v + 3.33p^2 + 0.61v^2$

For PLA+C-steel pair: $\mu = 1.23 - 1.52p - 1.12v + 2.56p^2 + 0.42v^2$

where:

μ – coefficient of friction, p – pressure [MPa] and v – speed [m/s].

When comparing the graphs with the influence of the sliding speed v on the friction coefficient μ, it was observed that from the lowest sliding speed, the friction coefficient is lower in the pair PLA+10%C-steel. This statement is true for the entire range of tested pressures.

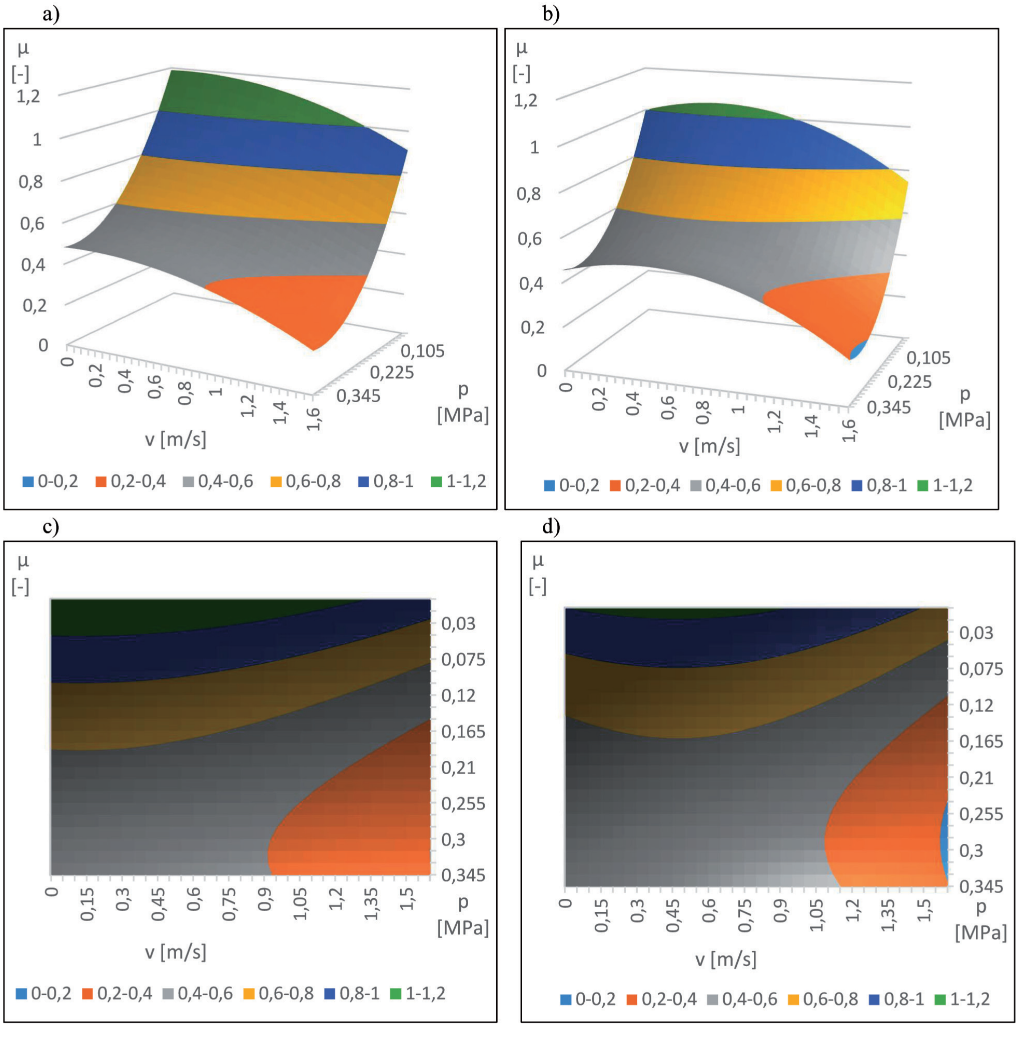

Fig. 1. Dependence of the coefficient of friction μ on the speed v and pressure p in the sliding pair: a) and c) PLA-steel C45, b) and d) PLA+10%C-steel C45

Rys. 1. Zależność współczynnika tarcia μ od prędkości v i nacisku p w parze ślizgowej: a) i c) PLA-stal C45, b) i d) PLA+10%C--stal C45

In the case of the PLA-steel pair, increasing the sliding speed first causes a minimal change in the friction coefficient, only increasing the sliding speed above approx. 0.8 m/s is associated with a noticeable decrease in the friction coefficient. The highest coefficient of friction occurs at the lowest pressure (0.1 MPa) and the lowest sliding speed, and the lowest value occurs at the highest pressure (0.3 MPa) and the highest sliding speed (1.5 m/s).

In the case of the PLA+10%C-steel pair, regardless of the pressure value, initially increasing the sliding speed causes a slight increase in the value of the friction coefficient. After exceeding the speed of approx. 0.3 m/s, a further increase in the sliding speed causes a decrease in the friction coefficient (for the entire range of the tested pressure). The highest coefficient of friction occurs at the lowest pressure (0.1 MPa) and the sliding speed of approx. 0.3 m/s and the lowest coefficient of friction occurs at the highest pressure (0.3 MPa) and the highest sliding speed (1.5 m/s).

For both tested materials, the friction coefficient μ decreased with the increase of the sliding speed v and the pressure p. The most significant friction coefficient reduction was observed in the pressure range of 0-0.2 MPa.

Wear intensity

The wear intensity test results are shown in **Fig. 2**. The obtained regression equations were in

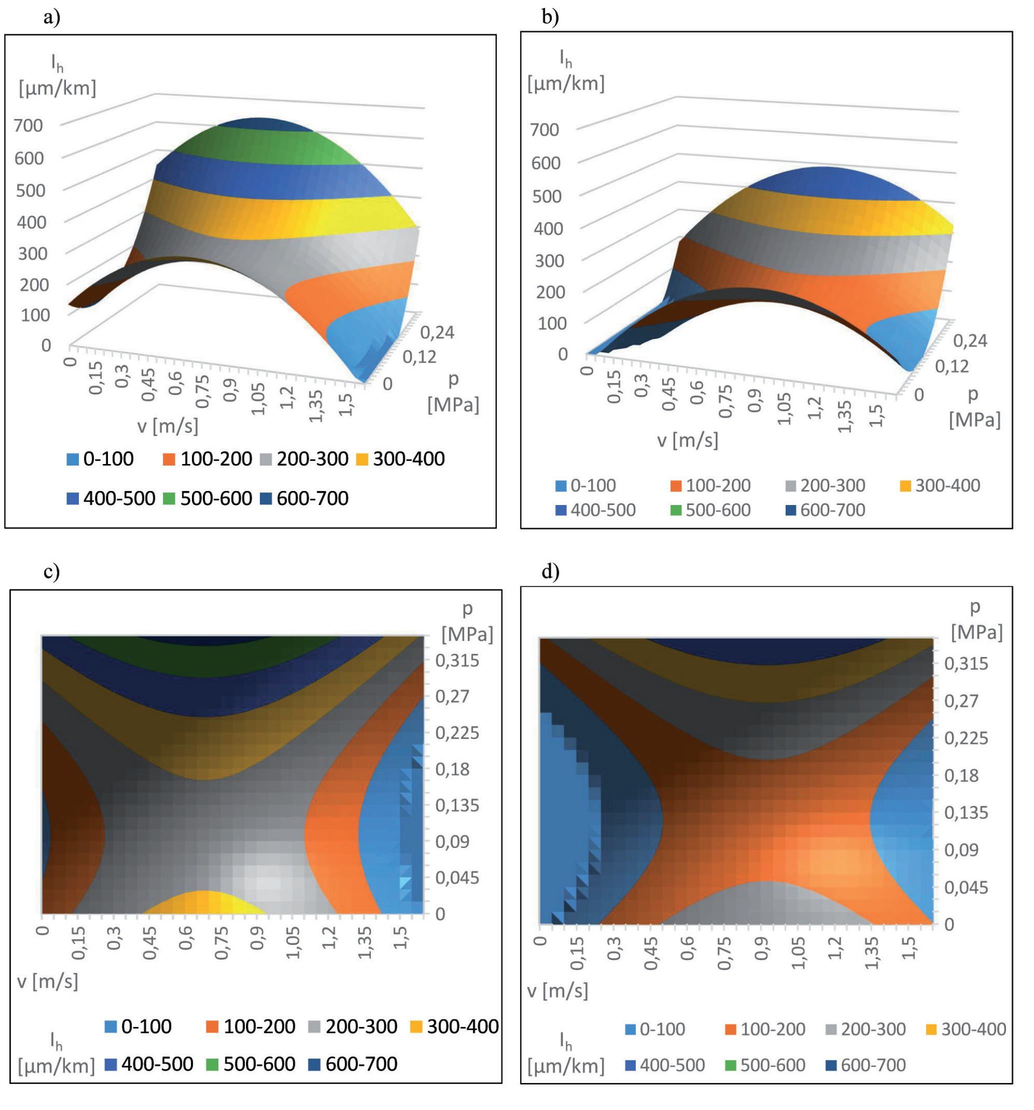

Fig. 2. Dependence of linear wear intensity I_h on velocity v and pressure p in the sliding pair: a) and c) PLA-C45 steel, b) and d) PLA+10%C-steel C45

Rys. 2. Zależność intensywności zużycia liniowego I_h od prędkości v i nacisku p w parze ślizgowej: a) i c) PLA-stal C45, b) i d) PLA+10%C-stal C45 the form of second-degree polynomials; for such functions, the coefficients of determination R^2 were obtained: 0.68 for the PLA-steel pair and 0.59 for the PLA+C-steel pair. Based on the value of R^2, it can be assessed that the model matched the results obtained experimentally to a satisfactory degree. The obtained regression equations had the following form:

For PLA-steel pair: $I_h = 4.39 - 674.78p + 777.27v + 5387.49p^2 + 577.47v^2$
For PLA+C-steel pair: $I_h = -211.15 - 809.55p + 914.29v + 5136.67p^2 - 558.01v^2$

where:
I_h – linear wear intensity [μm/km], p – pressure [MPa] and v – speed [m/s].

Comparing the graphs showed that the wear of the PLA+10%C composite is generally lower, regardless of the sliding speed and pressure. The wear of the PLA+10%C composite does not exceed 500 μm/km, and for unmodified PLA, it exceeds 500 μm/km in a wide range of sliding speeds and even reaches 630 μm/km.

In the case of the PLA-steel pair, increasing the sliding speed, starting from 0 m/s, causes an increase in wear (for the entire range of the tested pressure p). Maximum wear occurs at the sliding speed of approx. 0.7 m/s, and the maximum pressure (0.3 MPa) amounts to approx. 630 μm/km. Above this value, an increase in the sliding speed is associated with a decrease in wear. The smallest wear occurs at the highest sliding speed and pressures of 0–0.15 MPa and amounts to approx. 30 μm/km.

In the case of the PLA+10% C-steel pair, the course of the graph is very similar to that obtained for the PLA steel pair, but the consumption values are significantly lower. Increasing the sliding speed, starting from 0 m/s, also causes an increase in wear (for the entire range of tested pressure p), but at sliding speeds up to 0.05 m/s and pressures of 0–0.24 MPa, the wear does not exceed 30 μm/km, i.e. it is lower by approx. 100 μm/km, up to approx. 200 μm/km, compared to the PLA-steel pair with similar parameters of cooperation. The maximum wear occurs at the sliding speed of approx. 1.0 m/s and the pressure of 0.3 MPa and is approx. 490 μm/km, above this value, the increase in the sliding speed is associated with a decrease in linear wear. The smallest wear occurs for the sliding speed of up to 0.05 m/s and at low pressures (0–0.24 MPa) and is approx. 30 μm/km, and at the sliding speed of 1.5 m/s and the pressure of approx. 0.12 MPa and is about 50 μm/km.

Temperature

The temperature test results are shown in **Fig. 3**. The obtained regression equations were in the form of second-degree polynomials; for such functions, the values of the coefficient of determination R^2 were obtained: 0.68 for the PLA-steel pair and 0.73 for the PLA+C-steel pair. Based on the value of R^2, it can be assessed that the model was satisfactorily matched to the results obtained experimentally. The obtained regression equations had the following form:

For PLA-steel pair: $T = 20.79 + 55.61p - 0.61v - 123.75p^2 + 1.79v^2$
For PLA+C-steel pair: $T = 25.04 - 19.80p + 8.31v + 79.75p^2 - 4.09v^2$

where:
T – sample temperature [°C], p – pressure [MPa] and v – velocity [m/s].

When analysing the graphs, it was noticed that the temperature of the sample in the combination did not exceed 33ºC. This seems to be a small value, but it should be noted that the heat deflection temperature (HDT) for PLA is only 55ºC **[L. 17]**. Therefore, the sample was already approaching the temperature threatening the correct operation of the combination.

For the PLA-steel sliding pair, increasing the sliding speed has little effect on the temperature of the plastic sample. For the entire range of the tested pressure, the temperature difference between cooperation at a sliding speed close to 0 m/s and a sliding speed of 1.5 m/s does not exceed 5°C. Also, changing the pressure has little effect on the temperature of the sample. With increasing pressure, starting from 0 MPa, the temperature increased, and after exceeding the pressure of approx. 0.2 MPa, the temperature decreased. The lowest temperature occurs at sliding speeds close to 0 m/s and pressure close to 0 MPa and is approx. 21°C (a value close to the ambient temperature). The highest temperature occurs at a sliding speed of 1.5 m/s and a pressure of approx. 0.12 MPa and is approx. 31°C.

In the case of the PLA+10%C-steel sliding pair, it can be said that the addition of graphite increased

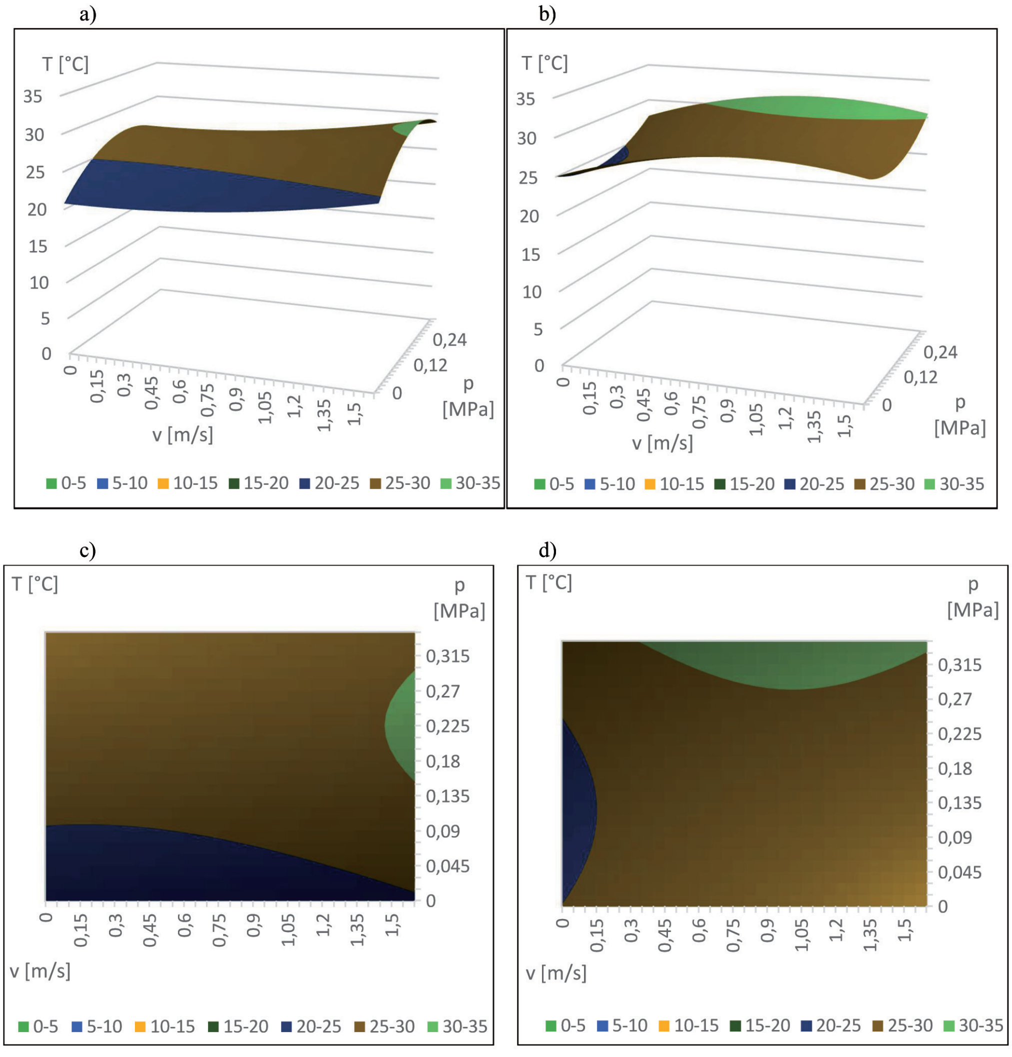

Fig. 3. Dependence of the temperature of a plastic sample on the velocity v and pressure p in the sliding pair: a) and c) PLA-steel C45, b) and d) PLA+10%C-steel C45

Rys. 3. Zależność temperatury próbki z tworzywa sztucznego od prędkości v i nacisku p w parze ślizgowej: a) i c) PLA-stal C45, b) i d) PLA+10%C-stal C45

the temperature of the sample. The temperature above 30°C appeared in a wider range than in the case of unmodified polylactide. Increasing the sliding speed has little effect on increasing the temperature at the point of cooperation (similarly to the previously discussed unmodified PLA). For the entire range of the tested pressure, the temperature difference between cooperation at a sliding speed close to 0 m/s and a sliding speed of 1.5 m/s did not exceed 5°C.

Also, pressure had little effect on temperature. With increasing pressure from 0 MPa, the temperature decreased after exceeding the pressure of approx. 0.12 MPa, the temperature increased. The lowest temperature occurred at the sliding speed close to 0 m/s and the pressure of approx. 0.12 MPa and was approx. 24°C. The highest temperature occurred at the sliding speed of 1.0 m/s and the pressure of approx. 0.3 MPa and was approx. 32°C.

With v and p values of 1.6 m/s and 0.4 MPa, the sample was deformed as a result of too high temperature. With these parameters, the material of the samples softened enough to flow in the direction of cooperation (**Fig. 4a** and **Fig. 4b**), creating a "flash" behind the sample. On the dial with the unaided eye, one could see the "rubbing, smearing" of the material (**Fig. 4c**).

It should be noted that with the v and p parameter values of 1.6 m/s and 0.4 MPa, emergency wear appeared. This was most likely related to the generation of a temperature above 50ºC in the contact. The temperature of the PLA+10%C sample measured with a pyrometer did not exceed 40ºC; however, one should be aware that the temperature shown in **Fig. 3** does not reflect the temperature at the contact but at the surface of the sample.

a) b) c)

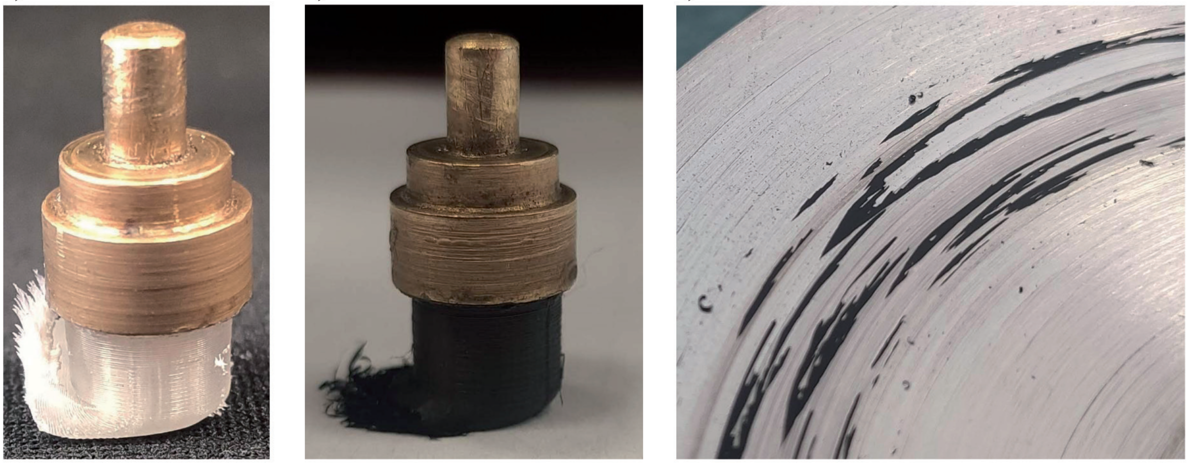

Fig. 4. Samples and counter-sample after cooperation with the speed v = 1.6 m/s and pressure p = 0.4 MPa: a) unmodified PLA, b) PLA+10%C, c) "rubbing, smearing" the composite on the surface of the steel disc

Rys. 4. Próbki i przeciwpróbka po współpracy z prędkością v = 1,6 m/s i naciskiem p = 0,4 MPa: a) PLA niemodyfikowane, b) PLA+10%C, c) „roztracia, rozsmarowania” kompozytu na powierzchni stalowej tarczy

DISCUSSION

The addition of graphite reduced PLA's coefficient of friction and wear. The decrease in wear was significant and visible almost in the entire range of speeds v and pressure p, while the coefficient of friction decreased to a lesser extent. The addition of graphite slightly increased the temperature of the sample during cooperation with steel.

The obtained dependencies can be interpreted differently, i.e. depending on what is more important. PLA+10%C during operating conditions at which it achieves the lowest coefficient of friction, it does not achieve the lowest wear, and vice versa – with the lowest linear wear, it does not achieve the lowest coefficient of friction. Regardless of the operating conditions, the addition of graphite is beneficial. Apart from a slight increase in temperature, there are no contraindications to adding graphite.

The addition of graphite slightly increased the temperature of the sample during cooperation with steel. The expected result was that thanks to graphite, which is a very good conductor of heat, it will be better dissipated from the association. The graphite conglomerates may be so arranged in the composite that they have no connection. There is PLA, which as an insulator prevents efficient heat dissipation between them.

On the other hand, it is possible that the heat generated by the loss was conducted through the graphite to the sample's interior, but there was a problem with its dissipation to the environment. This is most likely related to the decrease in PLA emissivity after adding graphite.

The addition of graphite to polystyrene reduces the material's emissivity, and this effect is used in the production of expanded polystyrene (styrofoam). With the addition of graphite, styrofoam is finally a better insulator than expanded polystyrene without the addition. A similar phenomenon may occur for PLA, and PLA with the addition of graphite is characterised by a lower emissivity, which makes it difficult to dissipate heat from the association.

Unfortunately, PLA is characterised by low resistance to increased temperature. For this reason, the lack of improvement in heat dissipation after adding graphite is a big disappointment. The problem associated with the use of the PLA+10%C composite is the significant value of the friction coefficient (for pressure less than 0.2 MPa, it is over 0.5). The tests showed that the coefficient of friction in the pair PLA + 10% C steel decreases for higher pressures and speeds of cooperation (its value is close to 0.2 for the maximum value of the tested pressure and speed). The decrease in the value of the friction coefficient with increasing pressure and speed may be a factor protecting the sliding element. As already mentioned, PLA is characterised by low resistance to increased temperature. Therefore, any increase in load or speed in the combination can cause damage to the sliding element in a short time. Due to the fact that for the combination PLA+10%C steel, a decrease in the coefficient of friction was observed with increasing pressure and speed, short-term emergency overloads may be compensated by reducing the coefficient of friction. Thanks to the reduced coefficient of friction in the connection, less heat will be generated, which will allow the material to survive a moment of overload.

CONCLUSIONS

The main purpose of the research was to compare unmodified PLA to PLA with 10% graphite. Therefore, the following conclusions for PLA+10%C are made in comparison to unmodified PLA:

1. With the addition of 10% graphite, PLA was characterised by significantly lower wear in a wide range of cooperation parameters (velocity v and pressure p). This confirms that the use of graphite as an additive to PLA is beneficial, especially since it has been previously proven that the addition of graphite does not impair other essential properties of PLA **[L. 14]**.
2. Significant differences in wear are clearly visible at low speeds and pressures. This is important because the PLA+10%C composite is designed to work with steel at lower pressures and speeds, and this is due to the low-strength properties of PLA and its very limited temperature resistance.
3. The addition of graphite only slightly decreased the coefficient of friction of the PLA+10%C composite.
4. The dependence of the coefficient of friction and wear is similar for PLA and PLA+10%C. What differs from the obtained graphs are the values of the measured quantities.
5. The addition of graphite slightly increased the temperature of the sample when working with steel.
6. The sliding element made of PLA+10%C composite is recommended to work at a pressure of about p=0.15 MPa. For such a pressure value, the smallest wear was observed, and the coefficient of friction did not exceed 0.5 (in the entire range of the tested cooperation speeds). If possible, the cooperation speed should be close to 1.5 m/s because the lowest wear and lowest coefficient of friction were recorded for this value.
7. If the priority is to obtain the lowest coefficient of friction, a pressure of about 0.3 MPa should be used. With such pressure, the coefficient of friction in the PLA+10%C pair of C45 steel does not exceed 0.2. Unfortunately, as a result of increasing pressure, wear also increases significantly.
8. Pressure and speed higher than p=0.3 MPa and v=1.5 m/s should not be used because emergency wear occurs after these values are exceeded.

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