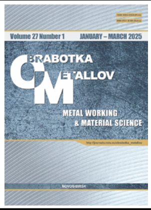
Obrabotka metallov (tekhnologiya, oborudovanie, instrumenty) = Metal Working and Material Science. 2025 vol. 27 no. 1 pp. 203–217
ISSN: 1994-6309 (print) / 2541-819X (online)
DOI: 10.17212/1994-6309-2025-27.1-203-217

NSTU NETI

Obrabotka metallov -
Metal Working and Material Science

Journal homepage: http://journals.nstu.ru/obrabotka_metallov



Wear behavior study of glass fiber and organic clay reinforced poly-phenylene-sulfide (PPS) composites material

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ARTICLE INFO

Article history:
Received: 10 January 2025
Revised: 23 January 2025
Accepted: 03 February 2025
Available online: 15 March 2025

Keywords:
Poly-phenylene-sulphide (*PPS*)
Glass fibre
Bentonite clay
Friction and wear
Tribological testing using the "pin-on-disc" method
Taguchi design
Wear behavior

ABSTRACT

Introduction. This study investigates the influence of key operating parameters (load, sliding velocity, and sliding distance) on the wear behavior of composites made of 40 % glass fiber and polyphenylene sulphide (*PPS*), with varying weight fractions of bentonite clay. The main purpose was to evaluate how different experimental conditions affect the wear characteristics. To achieve this, experiments were conducted using a *Taguchi L9* orthogonal array at three levels of complexity. The tribological tests were performed on a pin-on-disc setup, following *ASTM G99* standards, with six material samples containing different weight fractions of bentonite clay. The results show that wear of the original (virgin) sample increases with an increase in the applied average load. In contrast, samples containing bentonite clay exhibit a decrease in wear with increasing average load. Furthermore, an increase in the bentonite clay content leads to a significant reduction in wear, but a further increase to 7 % clay results in a noticeable increase in wear values. **Research Methods.** This study investigates the effect of load, sliding velocity, and weight fraction of bentonite clay on the wear and coefficient of friction (*COF*) of a composite material. Composite samples with varying clay content were tested using a pin-on-disc setup, and wear and *COF* were measured as dependent parameters. Scanning electron microscopy (*SEM*) was used to analyze the wear surfaces after testing to reveal the influence of independent parameters on wear mechanisms and surface morphology. The results revealed important trends in the friction and wear behavior under different conditions. Comparative analysis provided insights into optimizing the tribological performance of the material by balancing load, velocity, and clay content. **Result and Discussion.** This study investigates the effect of bentonite clay addition on the wear behaviour of *PPS* + *GF* composites. The findings reveal that wear decreases by up to 3 % with an increase in the weight percentage of bentonite clay, but increases again with a further increase in clay content. It is noted that a higher weight fraction of bentonite clay leads to an increase in the specific wear rate and a decrease in the coefficient of friction due to the manifestation of an abrasive wear mechanism caused by clay agglomeration. Conversely, a lower clay weight fraction promotes a reduction in the wear rate while increasing the coefficient of friction.This work intends to address the dual challenge of performance optimization and cost reduction in friction and wear applications. **The need of the work.** The purpose of this research is to develop an organic polymer composite that exhibits both high performance and cost-effectiveness. One of the key objectives is to create such a composite material using bentonite clay, an organic and readily available material that can be sourced at a low cost. This will enable the production of a competitively priced composite without compromising quality. Another goal of the research is to replace existing friction materials in brake and clutch systems with the newly developed composite, potentially improving its performance and durability. Furthermore, this work aims to create a composite material suitable for use in sliding bearings, particularly those operating in corrosive environments. Such a composite should possess increased resistance to chemical degradation, ensuring an extended lifespan and reliability under severe operating conditions.

For citation: Bhanavase V., Jogi B.F., Dama Y.B. Wear behavior study of glass fiber and organic clay reinforced poly-phenylene-sulfide (PPS) composites material. *Obrabotka metallov (tekhnologiya, oborudovanie, instrumenty) = Metal Working and Material Science*, 2025, vol. 27, no. 1, pp. 203–217. DOI: 10.17212/1994-6309-2025-27.1-203-217. (In Russian).

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Abbreviations

PPS – Polyphenylene Sulphide
GF – Glass Fibre
COF – Coefficient of Friction
SEM – Scanning Electron Microscopy
FRP – Fibre-Reinforced Polymer
MMT – Montmorillonite
POM – Polyoxymethylene
PTFE – Poly-Tetra-Fluoro-Ethylene
EDS – Energy-Dispersive Spectroscopy
HRC – *Rockwell C* scale hardness
OMMT – Organically Modified Montmorillonite
SAXS – Small Angle X-ray Scattering

Introduction

The problem of pollution associated with particulate emissions from ceramic, semi-metallic, and metallic brake pads has stimulated research into their replacement with alternative materials based on natural fibers, such as flax, hemp, and sisal. These organic fibers offer advantages in terms of cost-effectiveness, biodegradability, and low weight. Concurrently, synthetic fiber-reinforced polymer (*FRP*) composites are finding widespread application in various engineering fields, including aerospace, automotive, and civil industries, due to their high specific properties (modulus of elasticity and strength), biodegradability (in specific cases), corrosion resistance, and long service life.

A key role in determining the properties of *FRP* composites is played by the fiber-matrix interphase, through which shear stresses are transferred from the matrix to the reinforcing fiber, influencing both the short-term and long-term characteristics of the material. This paper presents a review of the structure and properties of the fiber-matrix interphase [1–3]. It is shown that the characteristics of the interphase between the reinforcing fiber and the polymer matrix have a significant impact on the mechanical and tribological properties of the composite. Using the example of *GFF*/*PPS* (glass fiber/polyphenylene sulfide) composites, it is demonstrated that an optimal composition containing 80 wt. % *GFF* (~70 vol. %) provides the best mechanical properties and wettability. The high mechanical performance of *PPS* composites with ultra-high *GFF* content is attributed to the increased thickness of the interphase layer and the effect of fiber interlocking.

In the context of environmental friendliness, the use of biodegradable reinforcing fibers, such as clay, can raise questions when applied to carbon fiber / clay / *POM* (polyoxymethylene) based composites. Experiments aimed at studying the mechanical and tribological properties of such composites have shown that adding clay contributes to an increase in tensile modulus and strength. It has been established that the adding of carbon fiber into *POM* composites improves their mechanical properties and wear resistance. A carbon fiber, clay, and *POM*-based composite demonstrated minimal specific wear rate and coefficient of friction values. Polymer composites modified with nanoclays exhibit enhanced mechanical properties, such as tensile strength, yield strength, modulus of elasticity, fracture toughness, and fatigue strength, compared to unmodified polymers. Nevertheless, data on the wear resistance and surface mechanical properties (hardness and scratch resistance) of such materials remain limited. It has been shown that optimizing the content (wt. %) of nanoclay helps to improve the interfacial interaction between the fiber, polymer matrix, and nanoclay, which opens up prospects for increasing the effectiveness of nanocomposite applications in structural applications [4–6].

To evaluate the tribological characteristics of composite materials, the wear rate and coefficient of friction were determined. Experimental studies have shown that polymer composites containing carbon fibers, graphite, and polytetrafluoroethylene in a polyphenylene sulfide matrix exhibit good wear resistance performance under operating conditions. It is noted that surface treatment of montmorillonite (*MMT*) clay leads to improved adhesion and interaction with reinforcing components, which positively affects the strength characteristics of the composites (tensile and flexural strength) and complements the functionality provided by natural fibers. Surface modification imparts hydrophobic properties to hydrophilic *MMT*, enhancing its compatibility with the organic polymer matrix.

It should be noted that the interfacial interaction between the matrix and the reinforcing clay fibers has a significant influence on the process of local strain formation (as confirmed by the Digital Image Correlation method), as well as on the processes of initiation and propagation of damage in carbon fiber reinforced polyphenylene sulfide (*PPS*) samples (*5HS*) during tensile testing at angles of ± 45° and 0°. Interfacial adhesion has a substantial impact on the failure mode: samples with strong interfacial bonding exhibit cohesive failure, while samples with weak interfacial bonding are characterized by intensive delamination [7–9].

In addition to the interface, the frequency of motion affects tribological characteristics: the coefficient of friction decreases with increasing frequency. Conversely, increasing the sliding distance leads to an increase in the coefficient of friction. Typical load does not significantly affect wear. The dependence of wear on the distance traveled is complex: it initially decreases and then increases. Thus, the coefficient of friction and wear rate depend on the load and frequency of reciprocating motion. The network structure of dispersed silicate layers in nanocomposites, and therefore the viscosity properties of nanocomposites, are largely determined by the concentration of added *OMMT* fibers [10–12]. This paper presents a novel small-angle X-ray scattering (*SAXS*) method for analyzing the degree of dispersion of silicate layers in a polymer matrix. *SAXS* and *STEM* results showed that an *OMMT* content of 5 wt. % is a threshold, initiating the formation of a strong flocculated structure of dispersed silicate layers. Further increasing the concentration of *OMMT* significantly alters the viscosity properties of the nanocomposite containing 5 wt. % *OMMT* [13].

A composite coating of carbon fiber-reinforced *PPS* applied to stainless steel and operating under water lubrication condition exhibits significantly higher wear resistance than under dry friction conditions [14–15]. The tribological behavior of the composite coating under water lubrication depends on both sliding speed and load. At low speed (0.43 m/s), a steady increase in friction is observed, which transitions to a gradual decrease with increasing load. The effect of sliding speed on the wear rate of the composite coating is less pronounced than the effect of load on the coefficient of friction, which rapidly increases under pressure. The coefficient of friction gradually increases with increasing load; at high speeds (0.86 m/s), this effect is amplified [16–18].

A study of the effect of dry and water lubrication on the tribological characteristics of carbon fiber-reinforced polyphenylene sulfide coatings shows that the fluctuations of the coefficient of friction over time are more stable when using water lubrication than with dry friction. The composite coating operating under water lubrication exhibits higher wear resistance than under dry friction conditions. Studies [19–20] present data devoted to the study of the tribological and mechanical properties of a composite material consisting of 70 % polyamide-66 (*PA66*) and 30 % polyphenylene sulfide (*PPS*) modified with various contents of polytetrafluoroethylene (*PTFE*).

The authors express their sincere gratitude to the Mechanical Engineering Department of Dr. Babasaheb Ambedkar Technological University, Lonere; D. N. Polymers, Chinchwad; Vishwakarma Institute of Information Technology, Kondhwa, Pune; DUTECH India Laboratories, Pune; and Agharkar Research Institute, Pune, for their support and invaluable contributions to this work. The authors are also grateful to the listed organizations for their contributions to the research.

Experimental procedure

Analysis has shown that adding *PTFE* to the *PA66*/*PPS* blend negatively affects the properties of the latter but significantly reduces the coefficient of friction and increases wear resistance. A *PPS*-based composite with 40 % glass fiber and varying concentrations of bentonite clay additives, obtained by thermal-compression bonding, is to some extent environmentally friendly. This study investigated the influence of load, sliding speed, and composition on the tribological characteristics of *PPS* reinforced with 40 % glass fiber and containing various concentrations of clay. The results show that increasing the content of clay particles and their distribution in the *PPS* matrix contributes to the accumulation of wear debris. This debris is formed as a result of adhesive wear at the contact interface between the composite surface and asperities on the steel disk, which act as abrasive particles. The tribological behavior of this wear debris is determined by the relative height of the asperities on the surface of the steel counterface. It was found that the minimum coefficient of friction corresponds to the composite containing no clay. The addition of 2 % clay leads to an increase in the coefficient of friction, while a further increase to 5 % causes a decrease in the coefficient of friction. Detailed information about the composition of the investigated materials is presented in Table 1.

Table 1

Materials and Method

Sr. No.	Sample	*PPS* [wt. %]	*GF* [wt. %]	Clay [wt. %]
1.	*PGB0*	62	30	0
2.	*PGB1*	60	30	2
3.	*PGB2*	55	30	5
4.	*PGB3*	50	30	9

Bentonite clay (aluminum phyllosilicate) is a common component used in combination with *PPS* and 40 % glass fiber to create an environmentally safe *PPS* composite. Wear tests were carried out on a *DUCOM TR-20-M26* friction machine using a pin-on-disk configuration, providing continuous contact between the sample (pin) and a rotating disk. The experiments used a cylindrical pin with a height of 40 mm and a diameter of 10 mm, in contact with a flat surface of a steel disk with a diameter of 300 mm and a thickness of 12 mm. The disks were made of 41MoCr11 steel with a hardness in the range of 55–58 HRC. The steel pins were made of composite material with carefully selected compositions. The surfaces of the pin and disk were cleaned with a tension-activated operator before each test. Each test was repeated five times using new pins and disks while maintaining constant parameters. The tests were carried out under dry friction conditions to maintain a constant temperature throughout a sliding distance of 33,085.26 m. The experiments used three sliding speeds: 2.0423 m/s, 4.0846 m/s, and 6.1269 m/s, as well as three levels of contact load between the pin and disk. The values of the test parameters for each level are presented in Table 2. The experimental design was developed in accordance with the '*Taguchi L9*' array, which involved conducting nine tests for each material composition.

The purpose of this study was to determine the wear rate and coefficient of friction of six different *PPS*-based composites with 40 % glass fiber and varying clay contents. The materials were provided by DN Polymers, located in Chinchwad, Pune. Before and after testing, the samples (pins and disks) were weighed on precision analytical balance. Morphological studies of the worn surface were performed using scanning electron microscopy (*SEM*).

Table 2

Test parameters

Sliding Speed v [m/s] ± 5 %	Pressure $\boldsymbol{p}$ [N/mm^2] ± 5 %	Load $\boldsymbol{N}$ [N] ± 5 %	Time [min]
2.045	0.27	30	20 min
4.085	0.52	50	50 min
6.127	0.78	70	80 min

Results and Discussion

Investigation of the wear process of PGB0 as a function of load

This study investigates the influence of the average load on the wear of *PPS* + 40% *GF* composite at a constant sliding speed. The *Taguchi L9* array was employed to analyze three operating parameters at three levels, as detailed in Table 3. This approach aims to systematically investigate the effect of load on wear. The results, presented in Fig. 1, show that the *PGB0* composite generally exhibits lower wear compared to base *PPS*. It is noted that the wear of *PGB0* increases with increasing load at a sliding speed of 2.0423 m/s (Fig. 1). This can be attributed to the increase in heat generation in the contact zone under load, which leads to softening of the polymer matrix and a reduction in shear resistance required to remove material from the sample surface.

Table 3

Wear investigation of *PGB0*

Sr. No.	Load [N]	Speed [rpm]	Time [min]	Avg. Wear [μm]
1.	30	200	20	5.58
2.	30	500	50	123.52
3.	30	800	80	142.25
4.	50	200	20	163.22
5.	50	500	50	189.42
6.	50	800	80	192.87
7.	70	200	20	419.12
8.	70	500	50	822.53
9.	70	800	80	825.15

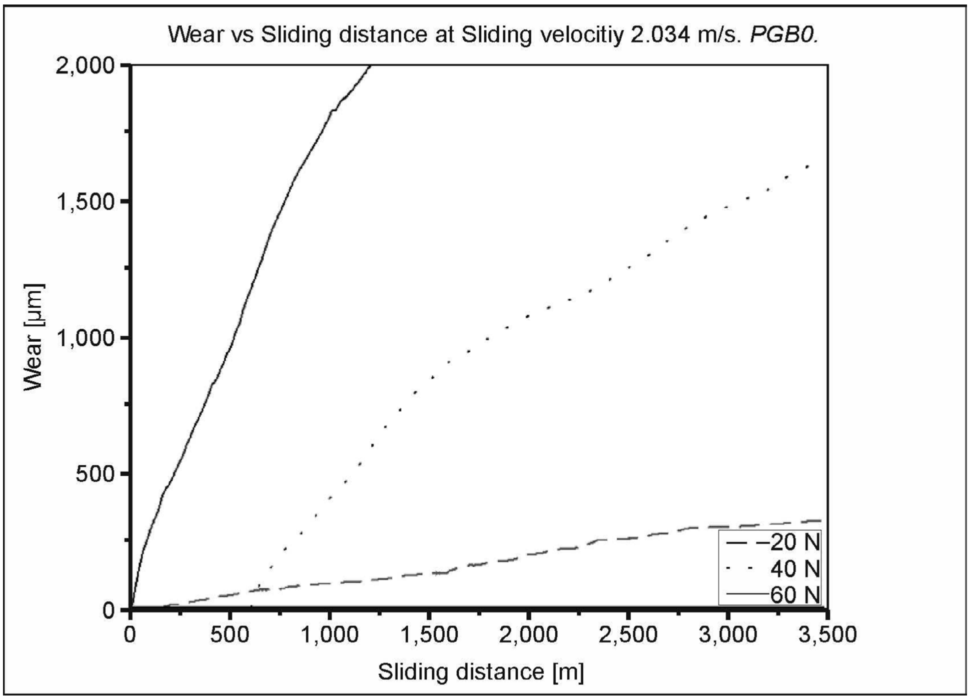


Fig. 1. Wear vs Sliding Distance. *PGB0*

Investigation of the wear process of PGB1 as a function of load

This section focuses on investigating the influence of 1 wt% bentonite clay on the wear resistance of the composite. As indicated in Table 4, the experiment aimed to assess wear as a function of sliding distance at a constant sliding speed of 2.0434 m/s and various contact load values. The objective of the analysis was to understand the impact of adding 1 % bentonite clay on the wear resistance of the composite under various loads.

The results, presented in Fig. 2, show that wear increases with increasing contact load. This trend was observed for all the investigated samples. Comparison with the *PGB0* composite (Fig. 2) demonstrates that the addition of 1 % clay leads to a reduction in wear, which is likely due to the action of clay particles as a solid lubricant and an increase in wear resistance under similar operating conditions. However, with increasing load, an increase in the wear of the *PGB1* composite is observed.

Influence of sliding speed on wear at a constant load of 20 N

This section analyzes the influence of sliding speed on wear at a fixed contact load of 20 N. The results, presented in Fig. 3, indicate significant abrasive wear of the material. Wear increases with increasing sliding

Table 4

Wear investigation for *PGB1*

Sr. No.	Load [N]	Speed [rpm]	Time [min]	Avg. Wear [µm]
1.	30	200	20	3.54
2.	30	500	50	42.35
3.	30	800	80	46.89
4.	50	200	20	57.35
5.	50	500	50	98.96
6.	50	800	80	100.78
7.	70	200	20	105.42
8.	70	500	50	110.54
9.	70	800	80	117.67

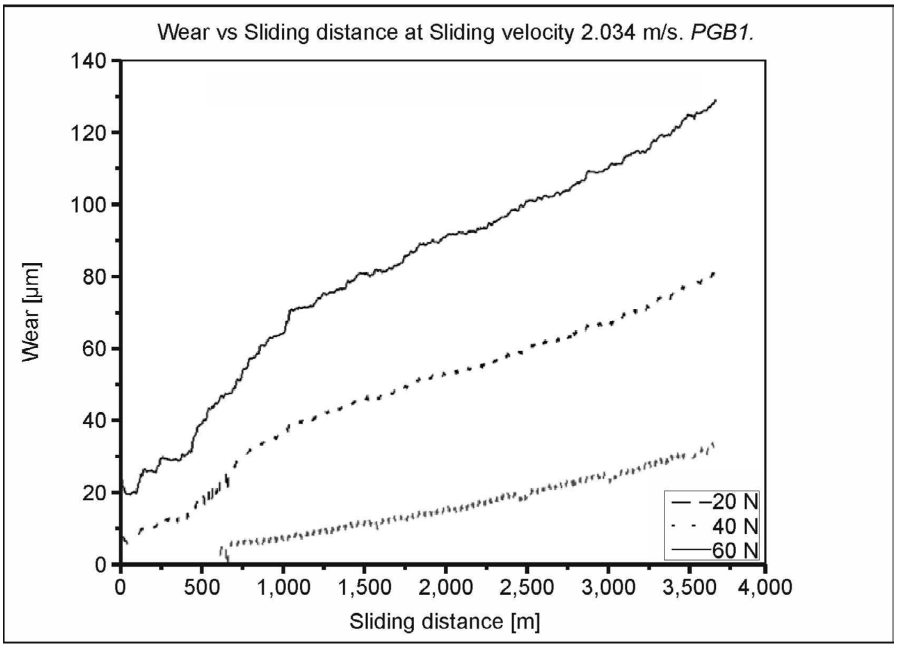


Fig. 2. Wear vs Sliding distance. *PGB1*

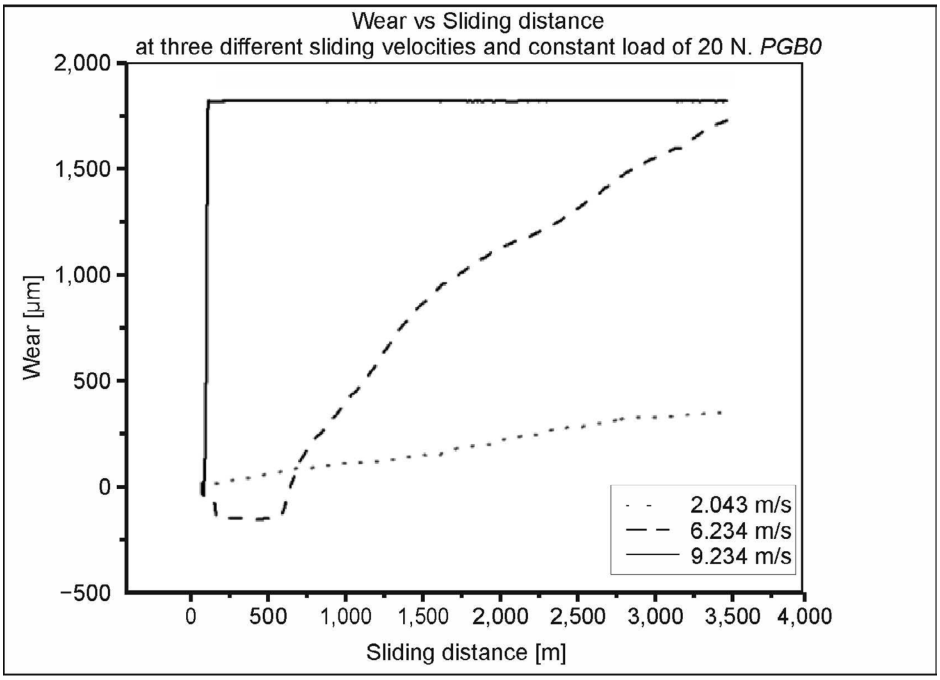


Fig. 3. Effect of Sliding velocity on Wear at constant Load of 20 N

speed, which corresponds to a three-stage wear pattern: an initial increase, reaching a maximum value, and a steady-state wear stage for each sliding speed. The minimum wear is observed at a speed of 2.0423 m/s, and the maximum at 6.1269 m/s, which emphasizes the significant influence of sliding speed on the wear process.

Influence of bentonite clay concentration on the wear resistance of the composite

The wear curve (Fig. 4) demonstrates the influence of the bentonite clay content on the wear of the composite. Composites without clay are characterized by the highest wear. Increasing the clay content to 3 % leads to a reduction in wear. Further increasing the clay content (to 5 and 7 %) causes an increase in wear, which indicates the existence of an optimal clay concentration.

Fig. 4 shows a decrease in wear for composites with 1 and 3 % bentonite clay and an increase in wear for composites with 5 and 7 % clay. The composite with 7 % clay demonstrates the highest wear. Therefore, to minimize dust formation when used in brake and clutch plates, the clay content should not exceed 3 %. An increase in wear with increasing load is also noted. The obtained results are supported by microscopy and energy-dispersive spectroscopy (EDS) data, presented in Figs. 5 and 6. At the initial stage of the tests, an increase in the wear rate is observed, which corresponds to the theory of adhesive wear and is due to the interlocking of asperities on the surface of the disk and the composite pin. As the tests continue, material transfer to the disk occurs, which temporarily increases the wear rate. At the final stage, the wear rate stabilizes. In the case of samples with bentonite clay, a decrease in wear is observed at the initial stages, which is associated with adhesion and the abrasive effect of agglomerated clay. Composites with 1 and 3 % clay show less wear, while composites with a clay content of 5 % or more demonstrate increased wear due to clay agglomeration (Table 5).

Influence of transfer film formation on wear

The formation of a transfer film on the counterface plays a crucial role in determining the tribological behavior of polymer composites. After the formation of a transfer film, the interaction occurs between the polymer and the material of the film, rather than with the polymer counterface. Studying the characteristics of transfer films is necessary for understanding wear mechanisms. The morphology of the worn surfaces of composites with varying clay content, investigated by scanning electron microscopy (*SEM*), is presented in Fig. 5.

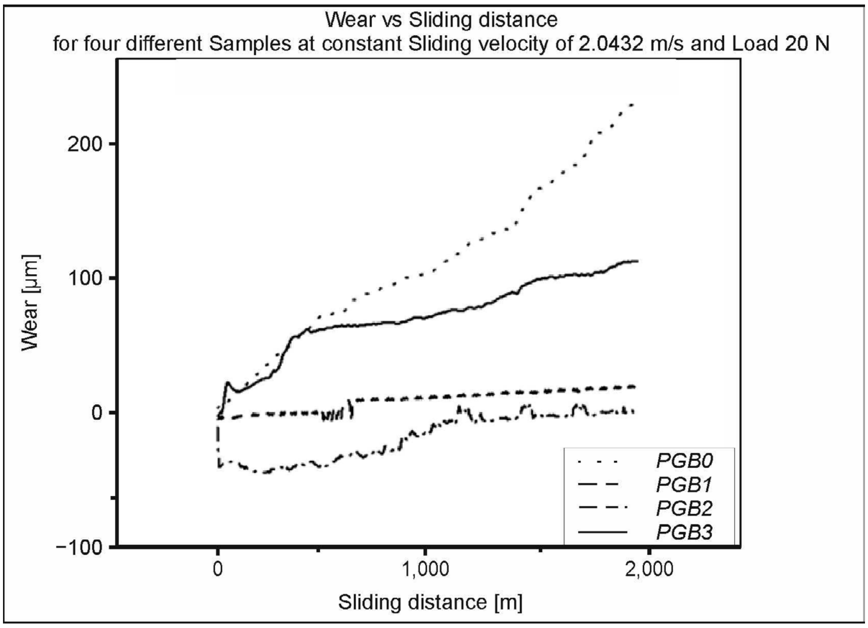


Fig. 4. Effect of bentonite clay on Wear value

Table 5

Investigation data Wear v/s Load

Sample	Average Wear Value [µm]		
	20 N	40 N	60 N
PGB0	5.58	166.14	422.1
PGB1	3.6	58.32	112.32
PGB2	96.3	128.52	131.58
PGB3	274.86	362.16	402.66
PGB4	289.98	521.46	604.26

Fig. 5, *a* shows that the composite pin with bentonite clay has fewer signs of adhesive wear and scuffing compared to the composite without bentonite clay. Figs. 5, *b* and *c* demonstrate the presence of fatigue cracks and agglomerated abrasive particles in the *PPS* / 40% *GF* / 5 % bentonite clay composite, indicating a reduction in wear with increasing clay content. On the steel disk, a thick incoherent transfer film is observed, which corresponds to the lower wear resistance of the *PPS* + *GF* composite without the addition of clay.

Fig. 5, *d* shows that in the composite with 5 % bentonite clay, agglomeration of clay particles occurs, which increases wear as a result of adhesive and abrasive processes. Thus, a small amount of bentonite clay prevents seizure and sticking to the matrix, promoting the formation of a high-quality transfer film on the steel surface and increasing wear resistance compared to the *PGB0* composite (without clay). However, at high clay concentrations, particle agglomeration occurs, leading to increased wear.

Energy-Dispersive Spectroscopy (EDS) Data

Energy-dispersive spectroscopy (*EDS*) was used for elemental analysis of the composites. Fig. 6 presents the results of *EDS* analysis of *PGB0*, *PGB1*, *PGB2*, and *PGB3* samples, demonstrating their qualitative composition. The *EDS* system, integrated with a scanning electron microscope (*SEM*), allowed for chemical analysis of the samples. The following elements were identified in the *EDS* spectra: silicon,

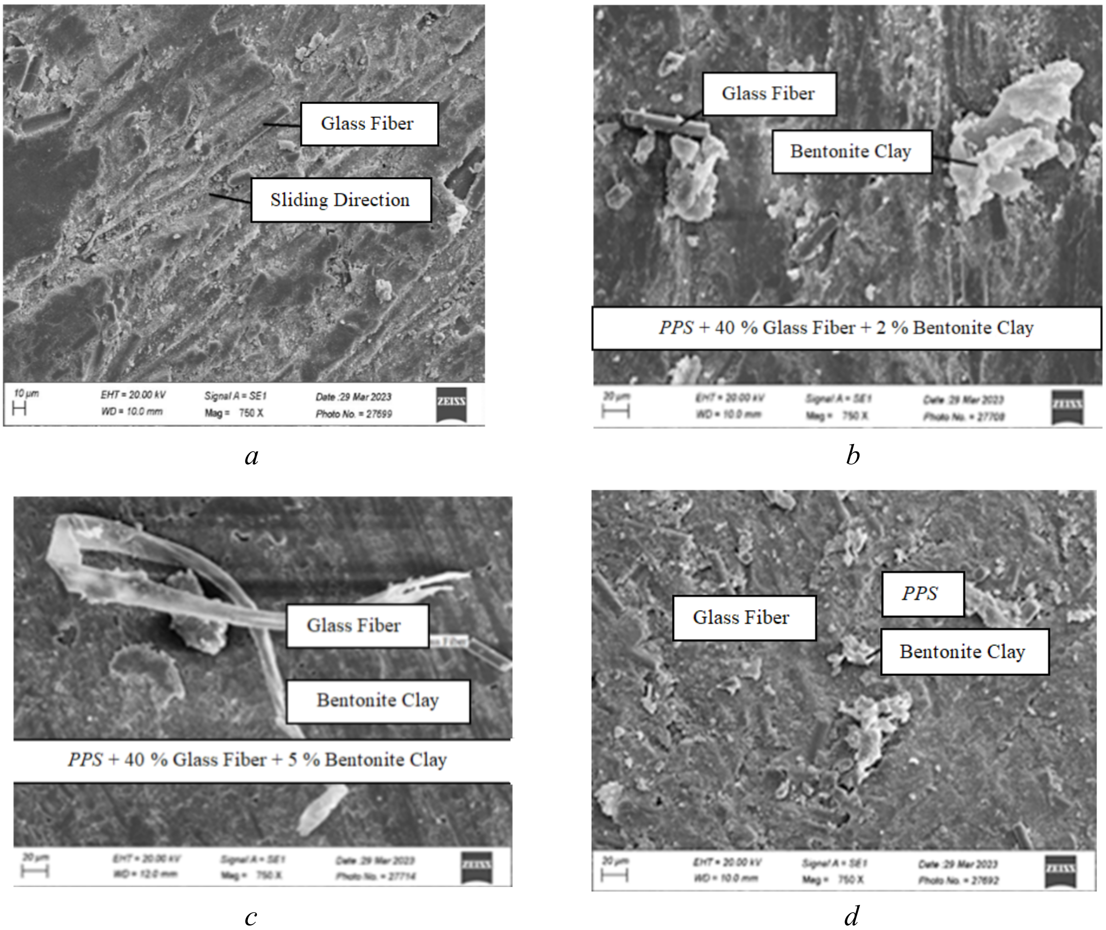


Fig. 5. SEM images of the composite pin surface:
a – PGB0; *b – PGB1*; *c – PGB2* and *d – PGB3*

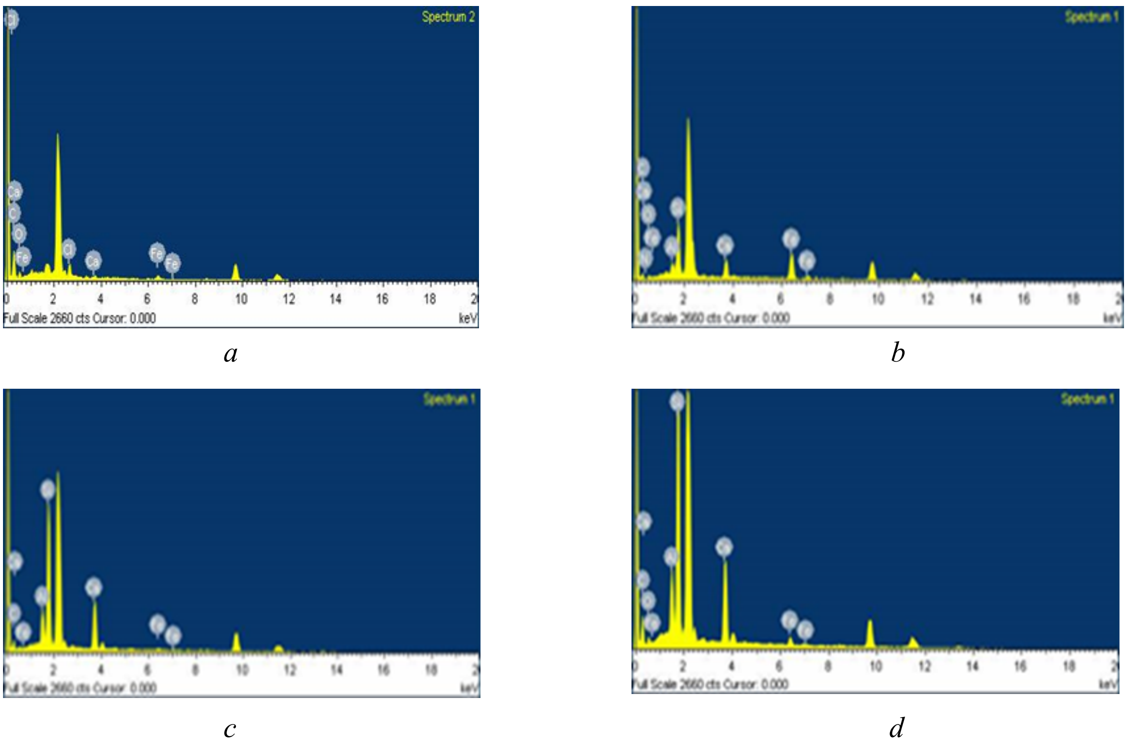


Рис. 6. EDS Spectrum of:
a – PGB0; *b – PGB1*; *c – PGB2*; *d – PGB3*

carbon, chlorine, iron, and calcium. The presence of iron and calcium is likely related to the wear of the steel disks and the transfer of wear debris to the sample surfaces.

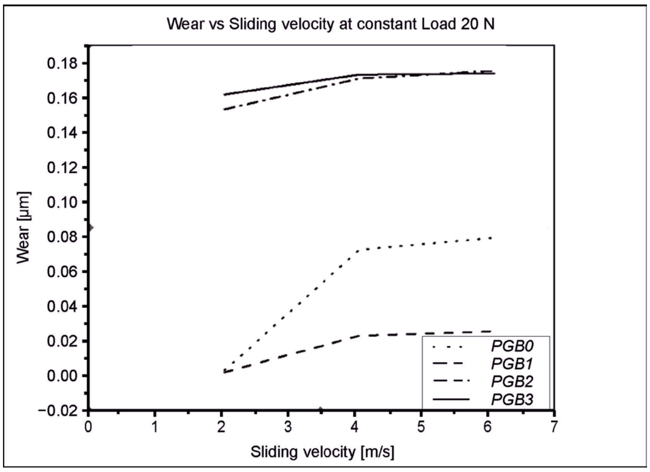
Figs. 6, *a–d* show the presence of bentonite, carbon, and chlorine, which confirms the inclusion of polyphenylene sulfide (*PPS*) and glass fiber in the composite. The *EDS* analysis also shows an increase in the concentration of aluminum, iron, and calcium.

Influence of sliding speed on wear at different loads

Further research is planned to compare the tribological characteristics of samples with different clay contents at constant load and sliding speed values. Fig. 7, *a-c* shows the dependence of wear on sliding speed for each sample. Increasing the sliding speed leads to an increase in wear. This effect may be due to the dominance of the adhesive wear mechanism over the abrasive one. The present study investigated the influence of bentonite clay on the wear of *PPS* + *GF* composites.

Influence of load on wear at constant and minimum sliding speed

Analyzing the influence of load on wear at a minimum sliding speed is important for planning experiments using the *Taguchi* method. Fig. 8 shows that increasing the load leads to a sharp increase in wear for the *PGB0* sample (without clay). Samples containing bentonite clay exhibit a smaller increase in wear with increasing load compared to *PGB0*. Increasing the load leads to an increase in temperature in the contact zone, which softens the polymer matrix and promotes delamination of the polymer film, exposing the glass



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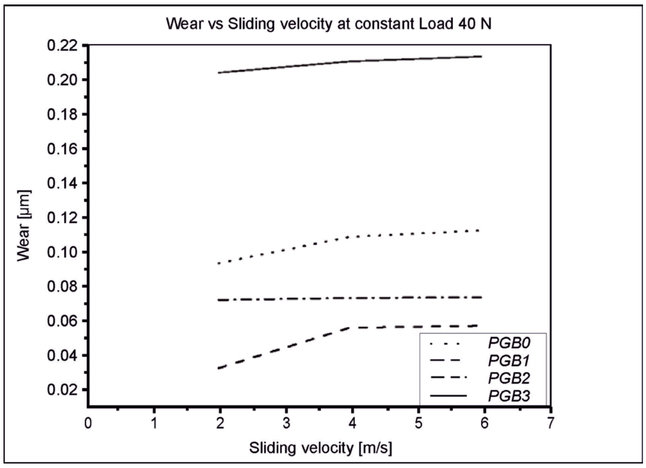

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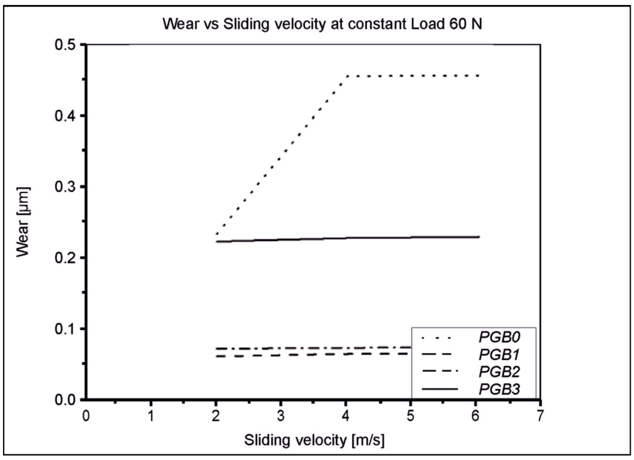

c

Fig . 7. Wear vs Sliding velocity

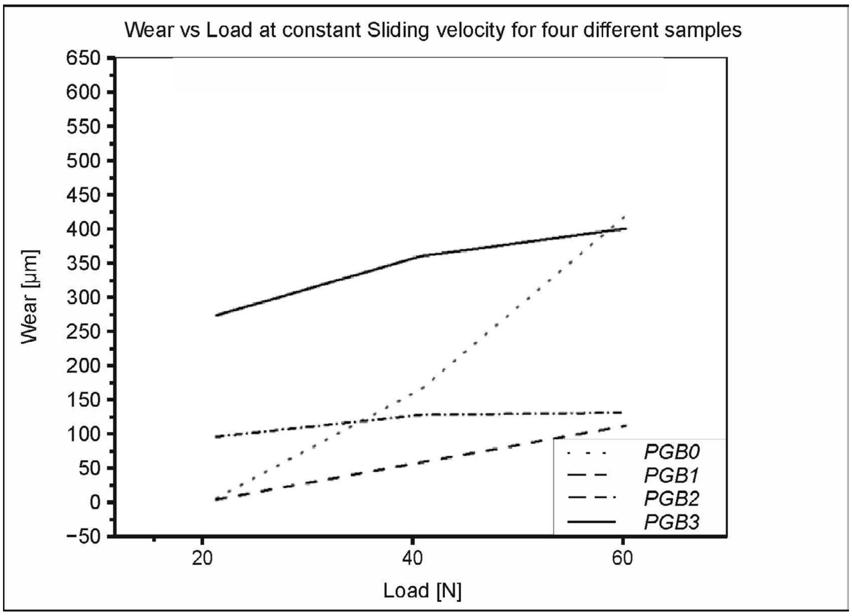

Fig. 8. Wear vs Load

fiber. The introduction of bentonite clay slows down the delamination process and reduces the rate of wear increase compared to *PGB0*. In the *PGB0* sample, friction is caused by the interaction between the severed and broken glass fibers and the asperities on the disk surface, which leads to wear. Bentonite clay particles, which act as a solid lubricant, reduce wear by delaying the transfer of composite material from the sample surface to the disk.

Conclusion

As a result of the study of the influence of bentonite clay on the wear of *PPS* + *GF* composites, the following conclusions were drawn.

• **Optimizing clay content reduces wear**: adding bentonite clay up to a certain concentration (up to 3 wt %) leads to a reduction in the wear rate. This suggests that moderate addition of clay increases the wear resistance of the composite. The lowest wear (3.6 µm) was observed at a clay content of 3 %, while the wear value for the composite without clay at the same load (20 N) was 5.58 µm. With an increase in load to 40 and 60 N, the wear of the composite without clay (166 and 422 µm, respectively) significantly exceeded the wear of the composite with 3 % clay (58.32 and 112.32 µm, respectively).

• **Exceeding the optimal clay content increases wear**: at a clay concentration above the optimum, an increase in the wear rate is observed. This is likely due to a change in the mechanical properties of the composite. At a clay content of more than 2 %, a significant increase in wear is observed: at 5 and 9 % clay, the wear values at a load of 20 N are 96.3 and 274.86 µm, respectively. These values significantly exceed the wear of the composite with 2 % clay. A similar pattern is observed at higher loads (40 and 60 N).

• **Clay agglomeration impairs wear resistance**: a high concentration of bentonite clay can lead to agglomeration of particles in the *PPS* + *GF* composite. Agglomerates, acting as an abrasive, increase the wear rate.

• **Clay affects the coefficient of friction**: the addition of bentonite clay can reduce the coefficient of friction due to its lubricating properties.

• **A small amount of clay improves tribological characteristics**: a small amount of bentonite clay (up to 3 %) allows for simultaneously reducing wear and maintaining an optimal coefficient of friction, providing a balance between wear resistance and frictional properties.

These findings emphasize the importance of carefully optimizing bentonite clay content to achieve the desired balance between wear rate and friction coefficient in *PPS* + *GF* composites.

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Conflicts of Interest

The authors declare no conflict of interest.