

Effect of Phenolic Resin on the Friction Performance of Composites with Hemp Fiber for Automotive Brake Pads

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Abstract: *The paper presents the results of experimental research conducted in the laboratory with the aim of obtaining new composite materials used for automotive brake pads. The friction material consists of seven components; five of them were kept in a constant proportion (60%wt), and two (40%wt) were varied in fraction volume, namely phenolic resin and hemp fiber. For each recipe, the effect of phenolic resin on density, porosity, hardness, thermal stability, and friction-wear characteristics was analyzed. The study indicates that finding the optimal balance between phenolic resin and hemp fiber constitutes an efficient approach in improving friction performance. Additionally, the study demonstrates the viability of organic composite materials in braking applications, as they are capable of meeting the performance levels established by traditional materials.*

Keywords: *resin, composite, hemp, friction, brake pad*

1. Introduction

Road transport, despite its global expansion, faces significant challenges in environmental protection. An approach for more sustainable transportation involves reducing pollutant emissions and adopting environmentally friendly production technologies [1]. During operation, automotive engines go through various operating regimes determined by speed and torque. When these parameters remain constant, the engine operates in a stabilized regime. This type of operation is common in road traffic when the vehicle is moving at a constant speed, especially outside urban areas and/or on the freeway. In these situations, pollutant emissions are minimal compared to those in other operating regimes. If the engine parameters undergo changes over time, the engine operates in a transient regime. This regime is frequent encountered in urban areas due to the constraints and obstacles present in these environments, leading to an increase in atmospheric pollutant emissions. The way a vehicle's engine works affects the level of variation in pollutant emissions. These emissions arise from the exhaust of combustion gases and as a result of repeated braking actions [3]. A significant source of fine and ultrafine particles in urban environments comes from brake and tire wear. This issue will persist even in the context of widespread use of electric automotive [4]. Currently, environmental protection organizations have identified an exceeding of permitted limits regarding the quantity of these particles released into the air, a situation that generates very serious effects on human health and the surrounding environment [5]. The effects of particles generated by brake wear on the environment and human health, depending on their size, chemical composition, and concentration in the atmosphere [6]. The problems caused by brake wear arise due to the brake pads, which are influenced by operating conditions, such as vehicle speed, brake pedal pressure, temperature, environment, and their durability [7]. Studies on the behavior of the friction materials used for brake pads are essential to minimize wear, develop new high-performance friction materials, and enhance the reliability of the braking system. Therefore, in recent years, extensive research has been conducted in this direction.

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Friction materials used in brake pads include abrasives, friction modifiers, filler materials, and binders in their composition. Among these components, the binder has the most significant impact on braking performance because the wear process and friction characteristics influenced by the physical and chemical properties of the binder [7]. Currently, phenolic resin is one of the most important binders in composite materials used for manufacturing brake pads [8]. It is used to bind the reinforcement and is characterized by low thermal conductivity, corrosion resistance, low density, high chemical and fatigue resistance, and high acoustic performance [9]. However, phenolic resin has certain limitations, such as high hardness that can make the brake pad brittle, increased sensitivity to humidity, and low impact resistance. The choice of the relative amount of phenolic resin in the composition of brake pads, as a binder, is an important issue for maintaining their structural integrity under different temperature conditions. This ensures that the brake pad composite can withstand the mechanical and thermal demands during braking operations. Specialized literature has conducted studies on the effect of resin concentration in friction materials.

In the work [10], the effects of phenolic resin as a binder in organic brake pads were investigated. Thus, density, porosity, hardness, thermal stability and friction-wear performance were examined. The results showed that with the increase in the phenolic resin content, the density, porosity, thermal stability, friction coefficient and wear rate can decrease for the studied friction materials.

According to the study by Jang and colleagues [11], a high concentration of phenolic resin led to an instability of the friction coefficient, caused by a decrease in its thermal stability.

Öztürk et al. show that phenolic resin presents constraints in that increased hardness imparts high fragility, sensitivity to humidity, and extremely reduced impact resistance to brake pads [12].

Mutlu et al. studied the friction-wear characteristics of composites with different quantities of organic fibers and phenolic resin intended for the brake pads of vehicles. The results showed that the analyzed friction materials can only be used for low temperatures and pressures. Thermal treatments were applied to some samples and it was observed that hardness increased, density decreased, microstructure changed, but noise appeared during the operation of the brake pad [13].

In the work [14], comparative studies were carried out on the use of modified and unmodified resins in the production of friction materials. The obtained results indicated that an increase in the quantity of modified resin decreased the friction coefficient, and mechanical properties improved.

According to the statements of Cheol and colleagues [15], adding a higher amount of phenolic resin in combination with potassium titanate led to an increase in hardness and friction coefficient at moderate temperatures.

In recent times, numerous studies have been published investigating the effects of phenolic resins on improving the mechanical characteristics and friction performance of brake pads for vehicles [16-19].

In this study, four laboratory recipes of composite materials were developed for the manufacturing of automotive brake pads. Each recipe contains seven components, including various fractions of phenolic resin as a binder and hemp fibers as reinforcement. The concentration of the other five components in the recipes has been kept constant. The experimental evaluation included the analysis of the impact of phenolic resin on density, porosity, hardness, and thermal stability characteristics for each recipe, as well as the analysis of the variation of the friction coefficient and mass wear as a function of temperature.

2. Materials and methods

In this study the composition of the friction material consisted of seven components, five were kept constant (60% wt), and two ingredients (40% wt) were varied in volume fraction, namely phenolic resin and hemp fiber.

Hemp fibers serve as reinforcement, and its selection was based on the fact that its tensile strength and Young's modulus have the highest values compared to other natural fibers [10]. On the other hand, hemp fiber contains 68 % wt cellulose, 15% wt hemicellulose, and 10% wt lignin, these being the highest percentages compared to other natural fibers [10]. Cellulose, along with hemicellulose and lignin present in any natural fiber, provides strength, rigidity, thermal stability, and imparts superior mechanical

properties to the friction material [8]. In the paper, hemp fibers have a gray color, average length of 2 mm, specific mass of 1.45 g/m^3 . Phenolic resin is produced through the reaction of formaldehyde and excess phenol under the action of acidic catalysts, forming a three-dimensional network structure [20]. Unmodified phenolic resin is a Novolac phenolic resin in paste form. In this work, the phenolic resin was treated with hexamethylenetetramine for reinforcement purposes.

The additive materials used in the recipes are: graphite, zirconium oxide and silicon carbide. The graphite added in the composites with silicon carbide improves the anti-friction properties, acting as a lubricant. With a density of 2.2 g/cm^3 , it is resistant to high temperatures and pressures. Silicon carbide added to aluminum alloys contributes to increasing wear resistance, especially abrasive wear. The density of the material is 3.21 g/cm^3 and the high hardness gives to the composite a suitable behavior in conditions of wear by friction. Zirconium oxide is a material used to make components resistant to thermomechanical stress, it has very good resistance to abrasion and impact. It has high compressive strength and can be used at high process temperatures, low density of 4.8 g/cm^3 and low porosity. The additive materials used are present in Figure 1.



Figure 1. Additive materials used in composites

The formulation of the recipes was based on thorough research from the specialized literature and a multitude of trials until establishing the proportion of the components. Recipes that did not bring an evolution in the development of new composite materials were excluded from the work. In this sense, the experiments were carried out successively, removing the great majority of the problems that arose by making decisions in the direction of obtaining appropriate samples from the point of view of the geometric shape, integrity, structure, compactness, hardness of the sample, as well as porosity. Table 1 presents the composition of the friction materials studied in this work.

Table 1. The composition of the friction materials

Sample	Aluminium (% wt)	Graphite (% wt)	Zirconium oxide (% wt)	Silicon carbide (% wt)	Aluminum oxide (% wt)	Phenolic resin (% wt)	Hemp fibre (% wt)	Total (% wt)
N1	30	10	5	5	10	30	10	100
N2	30	10	5	5	10	25	15	100
N3	30	10	5	5	10	15	25	100
N4	30	10	5	5	10	10	30	100

In order to improve the adhesion of hemp fibers to the phenolic matrix, they were treated with 5% NaOH for 6 h, followed by washing with water and air-drying at room temperature for 72 h.

In the production of organic materials, powder metallurgy was used in order to obtain an increased homogeneity of the components. The raw materials used in the recipes were ground, weighed, compacted, and then subjected to the sintering process. The grinding was carried out in a ball mill, type PM100. The weighing process was performed using a Sartorius CP2202S-OCE with a precision of 0.01g. The powder components were dry-blended to ensure macroscopic homogeneity in a Rotation Drive STR4 mixer for 40 min, at 20 rpm at room temperature. The obtained mixture was combined with the resin, and mixing continued for 20 min at a speed of 1500 rpm. Subsequently, it was introduced into the mold. The components of the mold are illustrated in Figure 2. The mixture was cold-pressed using a hydraulic press, type PH 40, at 0.5 MPa, and then subjected to drying at room temperature. The mold was placed in a resistance furnace, Nabertherm L15/12/B180, where the post-curing of the sample was

conducted at a temperature of 150°C for 30 min. At the end of the sintering process, the samples were extracted from the mold and cooled and dried in air at room temperature, as shown in Figure 3.



Figure 2. Component elements of the matrix



Figure 3. Air cooling of the sample at room temperature

The factors that influenced the achievement of a compact sample that could be extracted from the mold without disintegration and defects were: the heating-cooling regime, the homogeneity of the components, and the pressing force. Figure 4 shows the samples and SEM images based of the studied recipes. In samples N2 and N3, a relatively homogeneous distribution of the constituents in the aluminum matrix can be observed. Also, in samples N1, N2 and N3, areas with resin (black areas) unevenly distributed in the structure are observed and in all samples the presence of aluminum is observed (white areas), unevenly distributed.

The dimensions of the disc samples comply with the technical conditions presented in the diagram in Figure 5.

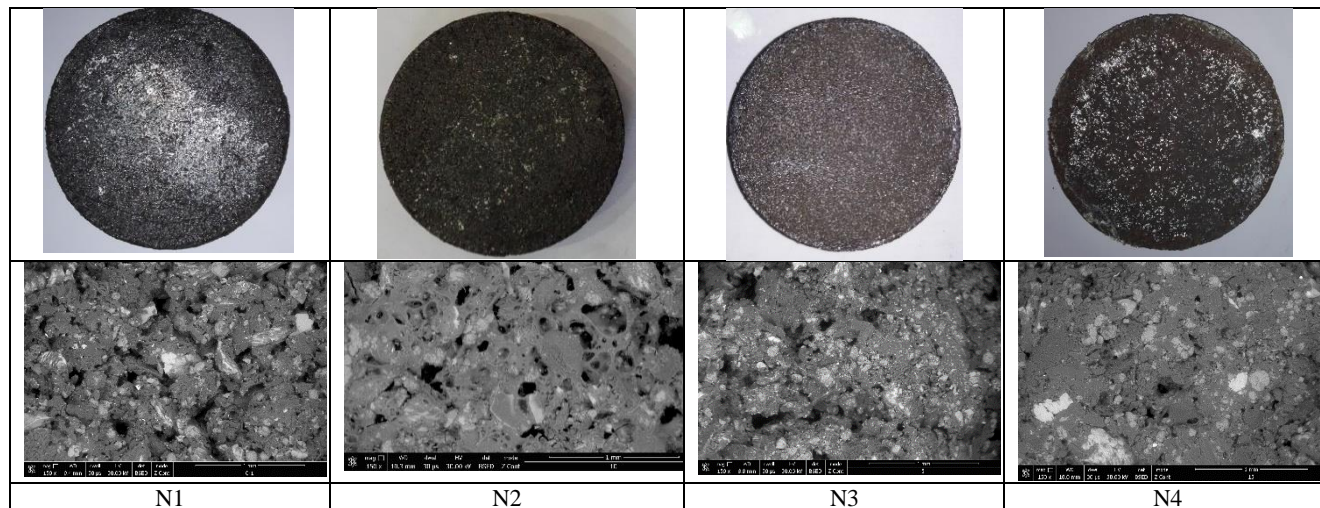


Figure 4. Samples fabricated based on the four recipes

The disc samples with the dimensions shown in Figure 4 were used for hardness determination and conducting friction-wear test. For the characterization of materials in terms of density, porosity, and thermos-gravimetric analysis, the samples were prepared at 30 mm x 30 mm x 8 mm, as shown in Figure 6. In the present study, five tests were conducted for each recipe and under each testing condition, with the average values being presented in the paper.

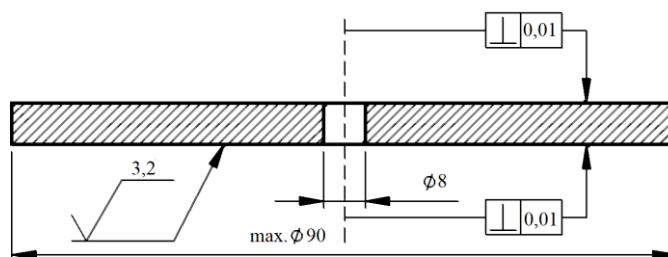


Figure 5. The dimensions of the disc samples

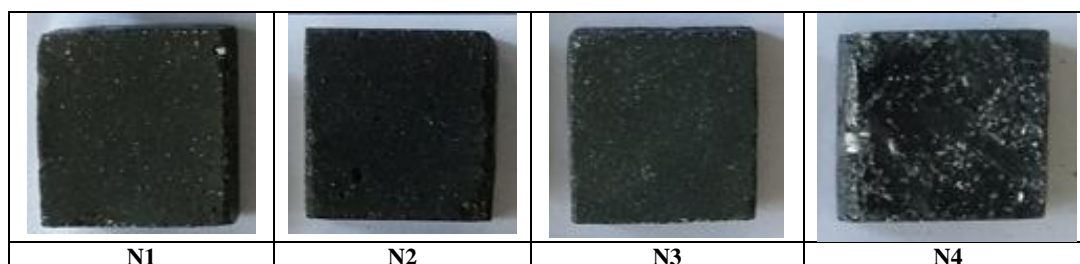


Figure 6. Samples measuring 30 mm x 30 mm x 8 mm used for determining density, Porosity and thermogravimetric analysis

The determination of density was carried out based on Archimedes' principle [2, 10, 16]. Porosity was determined using a mercury porosimeter, type PASCAL 240/140, with the operating pressure of 100 MPa. The hardness of the composite sample was measured using a Shore D hardness tester. Thermogravimetric analysis was conducted to investigate the weight change and thermal degradation of friction materials. For this purpose, a Netzsch STA 409 CD analyzer was used with a heating rate of 15°C/min up to 800°C. The friction coefficient was determined on a TR-20 tribometer, at a constant ambient temperature, which operates based on the "pin-on-disc" method in dry friction conditions. The equipment allows the mounting of disc samples made of the test materials with dimensions presented in Figure 4. The pin of the equipment is a cast iron ball with a diameter of 6 mm. Each test was conducted at a radius of 15 mm from the symmetry axis of the equipment. The testing parameters are: rotation speed of 150 rot/min, average pressure 0.53 MPa and testing time of 18000 s. The experimental data were obtained in the form of a text file from the software of the tribometer's computer, imported and processed in the Excel program. The results are presented in graphical form.

The effect of temperature on the friction coefficient and the wear mass was studied using a friction apparatus based on the operating principle of the Krauss test [7, 21]. The installation has a power of 1 kW and a maximum speed of 1500 rotations per minute, with the testing method being "pin-on-disc" under dry friction conditions. To perform the experiments were used discs made of each recipe of composite material, with the dimensions shown in Figure 4, and the pins were made of cast iron brand G 2500, intended for the production of automotive brake discs. The pin of the equipment and its dimensions are presented in Figure 7.

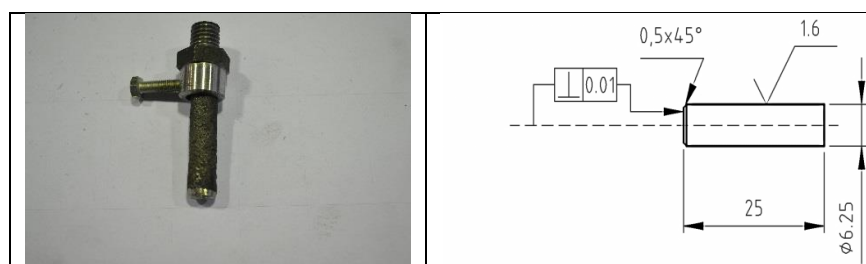


Figure 7. Dimensions of the pin in the tribometer assembly

The sliding speed of the disc was 2.35 m/s, the radius of action of the pin was 15 mm from the center of the disc, and the distance used in the experiments was 2115 m. The testing method involves heating the rotating disc sample through a resistor located at its lower part at four test temperatures: 100°C, 150°C, 200°C, and 250°C, and maintaining them at these temperatures for 20 s. The total test time for each temperature is 900 s. The evaluation of the behavior of the tested materials is performed through the gravimetric method (weight loss).

3. Results and discussions

Table 2 presents the results of the density, porosity, hardness test, and thermo-gravimetric analysis.

Table 2. The results of the density, porosity, hardness test, and thermos-gravimetric analysis

Sample	Density (g/cm ³)	Porosity (%)	Hardness Sh D	Thermogravimetric Analysis		
				Initial mass M _i (g)	Final mass M _f (g)	Mass difference ΔM (g)
N1	1.87	25	74	25.778	25.128	0.65
N2	1.95	15	62	24.345	23.885	0.46
N3	2.01	14	59	23.964	23.374	0.59
N4	2.20	28	45	22.857	22.057	0.80

The density of composites is relatively low compared to the density of metals due to the low density of phenolic resin, $\rho = 1.2 \text{ g/cm}^3$. The test result indicates that the density of the samples decreases with the increase in the volume fraction of phenolic resin. Similar results were obtained in reference [10].

The porosity of the friction material plays a significant role in improving braking efficiency because it absorbs heat during sliding and dampens operational noise. The specialized literature indicates that commercial brake pads have a porosity of approximately 10% to 14% to achieve good performance and low wear during operation [10, 22]. In this context, composites obtained by recipes N2 and N3 exhibit a porosity in accordance with the specific ations found in the literature. On the other hand, the low porosity obtained in these samples can be attributed to the increased bond between the binder and hemp fiber, resulting from a balance that exists between the quantities of resin and organic fiber.

The hardness of the composite samples increases with the increase in the volume fraction of phenolic resin. This is explained by the fact that with the increase in the quantity of phenolic resin, the amount of cross-linking will also increase during the curing process. Similar results were obtained in reference [10], indicating that with the increase in the quantity of resin, the density decreased, while the hardness increased.

The thermogravimetric analysis of the four tested composites indicates a tendency for weight loss due to the evaporation of water resulting from the chemical degradation of phenolic resin. A lower weight loss implies better thermal stability [10]. In this context, composite N2 exhibits the best thermal stability compared to the others. The results of the thermogravimetric analysis indicate that the thermal stability of the composite samples decreases with the increase in the volume fraction of phenolic resin.

The friction coefficient of the composite material is an important parameter that characterizes the braking performance. The evolution of the friction coefficient at constant temperature was analyzed using the TR-20 tribometer, and the principle of experimental determinations is presented in subsection 3 of the paper. Figure 8 illustrates the evolution of the friction coefficient at constant temperature for the analyzed samples.

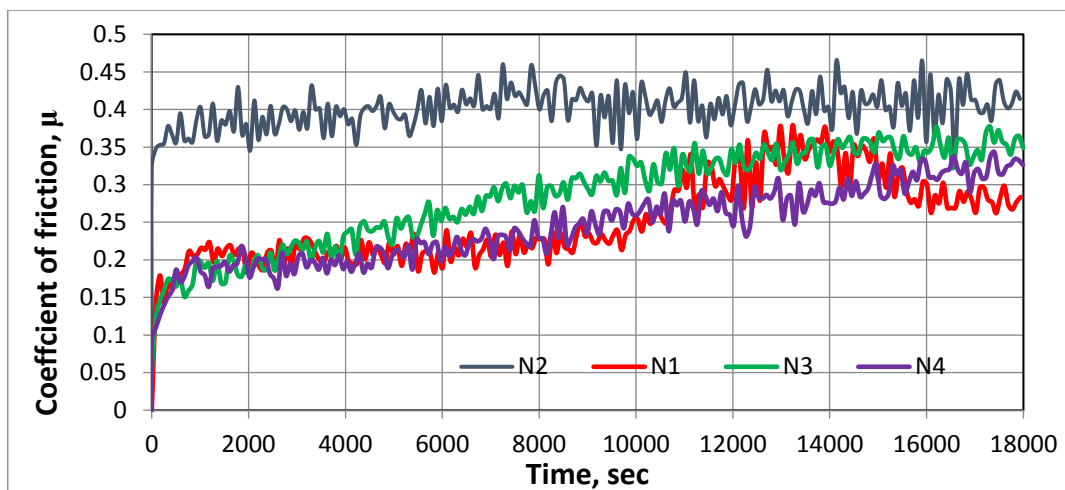


Figure 8. The evolution of the friction coefficient at constant temperature

From the beginning of the test, the friction coefficient for sample N2 reached the value of 0.35, and after 6000 s, it exceeded of 0.4. For samples N1 and N4, the friction coefficient is relatively low, reaching the value of 0.2 after 6000 s and for sample N3, it is 0.25.

After 10000 seconds, the friction coefficient of sample N2 stabilized around the value of 0.4, and for sample N3, the friction coefficient stabilized around 0.35 after 14000 s. For sample N1, the friction coefficient began to decrease after 16000 s, while for sample N4, the friction coefficient did not stabilize until the end of the experiments. The friction coefficient values for samples N2 and N3 fall within the range specified in the specialized literature, 0.3-0.6 [8]. Fluctuations in the friction coefficients around a value are due to the surface roughness present on the friction material surfaces.

The results of the friction-wear test were reported only for samples N2 and N3, as samples N1 and N4 were damaged during the experiments. For sample N1, the damage occurred at around a temperature of 150°C, while for sample N4, it occurred at a temperature of 100°C. Figures 9 and 10 shows the damaged sample N1, respectively N4. The damage of sample N1 is attributed to an excessive amount of phenolic resin, which resulted in a high hardness of the sample that did not withstand the mechanical stresses under the friction-wear test. The high hardness of the sample caused the friction material to become brittle, reducing its impact resistance. Similar conclusions were obtained in [12]. The damage of sample N4 is caused by the fact that there is too little resin, which does not bind the components in the recipe. This is supported by the lowest hardness value and a high porosity level.



Figure 9. Sample N1 damaged during friction tests



Figure 10. Sample N4 damaged during friction tests

To highlight the influence of the amount of phenolic resin on the behavior of friction materials, experiments were conducted at temperatures ranging from 100°C to 250°C, with an increment of 50°C. The friction coefficient was determined as a function of temperature to highlight the influence of

phenolic resin on the friction-wear characteristics at different temperatures. The evolution of the friction coefficient with temperature is shown in Figure 11.

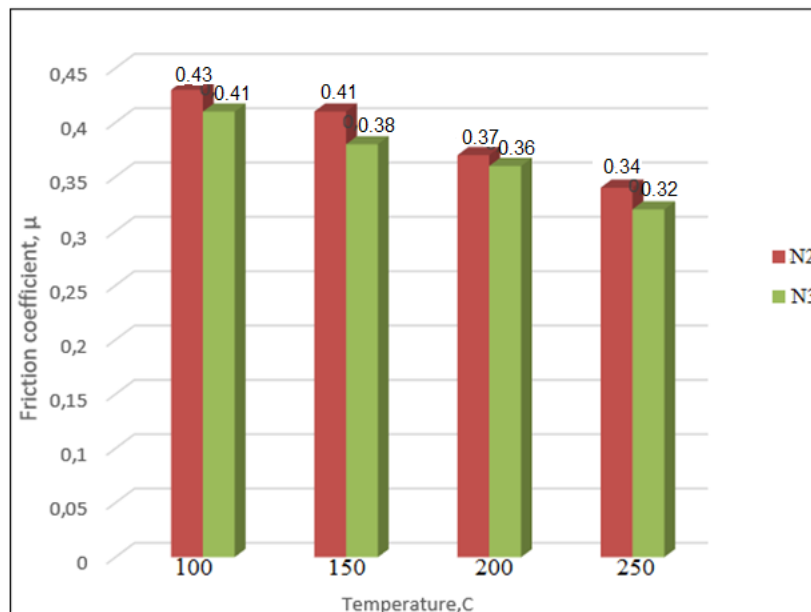


Figure 11. The variation of the friction coefficient with temperature

The graph presented in Figure 11 shows that for both samples the friction coefficient decreased with increasing temperature, this is explained by the softening of the phenolic resin. For this reason, the friction materials cannot be used for heavy vehicles and intensive braking regimes. For these vehicles, the power required for braking is high as a result of the high own masses, which generates high thermal strains that lead to overheating of the brakes and loss of braking efficiency.

For all test temperatures, the friction coefficient of sample N3 is lower than sample N2, which can be explained by the lower thermal stability of this sample. For samples N2 and N3, a decrease in the friction coefficient is observed with increasing temperature. The obtained results have been confirmed by the study [10]. The change in the friction coefficient during the experiments depends on the modification of the contact area of the friction couples, the resistance of the resin, and the friction properties of the components in the recipes at high temperatures, [22].

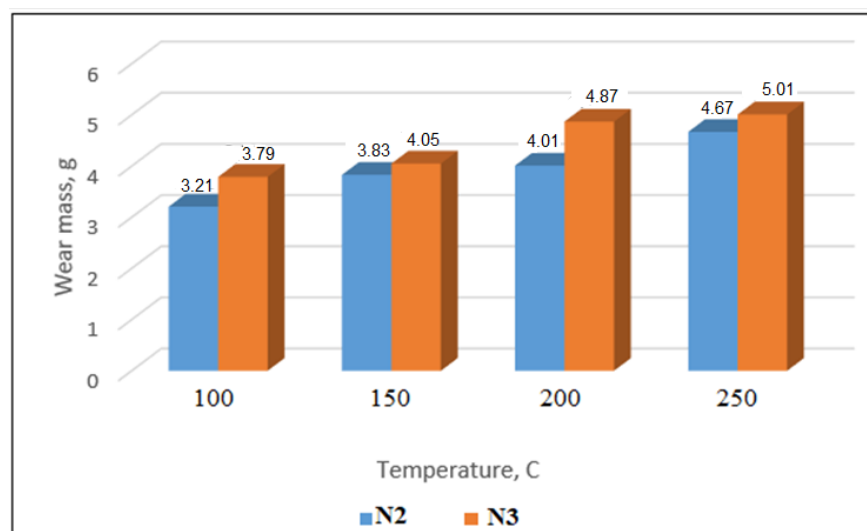


Figure 12. The variation of mass wear with temperature

Figure 12 shows the variation in mass wear of samples N2 and N3 with temperature, indicating that an increase in temperature leads to an increase in mass wear. This is explained by the fact that the increase in temperature allows the phenolic resin to decompose, and it no longer adequately binds the ingredients in the recipes. This leads to an increase in mass wear, [10]. Sample N2 has a lower mass wear than sample N3. Additionally, the thermal stability of sample N2 is better than thermal stability of sample N3. The obtained results indicate that sample N2 exhibits the best friction-wear behavior.

4. Conclusions

The conclusions obtained at the end of this study are:

- the constituent materials of the recipes proved to be compatible with each other because the obtained samples are well defined in shape and structure;
- the heating-cooling regime, homogeneity of components, and pressing force are determining factors for obtaining compact samples that can be extracted from the mold without disintegration and without defects;
- the density of the composite samples decreases with the increase in the volume fraction of phenolic resin;
- samples N2 and N3 have low porosity, consistent with values found in the specialized literature;
- the hardness of the composite samples increases with the increase in the volume fraction of phenolic resin;
- a low weight loss indicates improved thermal stability, and in this regard, composite N2 exhibits the best thermal stability, but also a good friction-wear behavior;
- the quantity of phenolic resin in friction materials is subject to restrictions, in the sense that an excessive amount leads to increased hardness, which makes the friction material brittle, as observed in sample N1;
- in the case of sample N2, there is an efficient interaction between hemp fiber and phenolic resin regarding friction-wear characteristics, an essential aspect for ensuring optimal braking system performance;
- samples N2 and N3 withstand moderate thermal, while maintaining a stable friction coefficient;
- finding the optimal concentration between phenolic resin and hemp fiber represents an efficient strategy for improving braking performance;
- as a result of the decrease in the coefficient of friction with temperature, the N2 sample with the best tribological behavior cannot be used in heavy vehicles with intensive braking regimes, it being suitable only for small and medium performance vehicles where the thermal regime is moderate;
- sample N2 exhibits the best behavior because the tribological characteristics are better than for the other tested materials;
- validation of the recipe with the best behavior requires some tests under the conditions of real regime, which is the subject of another work;
- integrating organic composite materials into the manufacturing process of brake pads plays a significant role in reducing the environmental impact in the automotive industry

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