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Original Article

Friction elements based on phenolic resin and slate powder

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ABSTRACT

Braking performance is dependent on friction materials of tribological components, generally metal and pad. The pad consists of a composite of thermosetting resin and reinforcement material that exhibits mechanical and wear resistant, heat conduction, lubricant, and others. Slate is a natural rock composed of extremely fine materials that brings interesting tribological properties, in addition, its industrial current activity generates a significant amount of mineral waste, which is a problem for the environment. The objective of this work was to propose a technological alternative as a new friction element based on slate particulate as tribological reinforcement in composite based on phenolic resin as a matrix, besides that, minimize the environmental impact due to the inadequate disposal of these slate tailings and add value to the mineral, classifying it as a residue to be used industrially and no longer as waste. In this sense, the effect of the amount of slate on the friction properties of the brake pads made was investigated. As the brake pad material, four different slate containing formulations were proposed, manufactured and analyzed, and as the brake wheel gray iron was chosen. The friction and wear characteristics were determined by Pin-on-Disk type tribological tests, the pair was composed by disk (pad formulations) and pin of gray iron, representing the brake wheel. The coefficients of friction of the composites were shown to be regular and stable, with an average of 0.44 between the samples. Among the results obtained, the formulation containing 40 % of slate and 35 % of phenolic resin, presented the most satisfactory parameters compared to commercial friction materials in current use.

1. Introduction

Friction materials generally consist of mineral particles and fibers embedded in a resin matrix and are used as scrubbing or braking mechanisms for certain devices. Braking performance is primarily controlled by the composition and microstructure of the incrusted material. These generally contain a

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large number of different raw materials, such as fibers, mineral, metallic and ceramic particles, elastomers and solid lubricants, all bound by a polymeric matrix which is often thermosetting [1]. These materials must meet several requirements, such as stable frictional strength, good wear resistance, and a reliable performance over a wide range of temperatures, speeds, pressures and environments [2]. The friction element must also be compatible with the rotor component so that there is little wear on the counterparts, low noise and low vibration during braking. All these requirements must be linked to a reasonable cost and to a minimum environmental impact [3].

For cars and many industrial applications, polymer matrix composites have long been used as brake pads. Phenolic resin is probably the most common binder used in these friction materials because of its relative low cost, with a good combination of mechanical properties, good adhesion with most components, and easy handling [4]. It has been tested and used in the production of friction elements for a long time, notably after the patent U.S. Pat. No. 3,873,490 was granted [5].

Due to the complex chemomechanical interaction on the friction materials surface, it is difficult to predict the brake performance [6]. The addition of different fibers positively affects the friction and wear behaviours of a composite friction material in diverse ways and at various extents. Thus the designing of formulations for such systems must be performed on a trial and error basis [7]. Therefore the purpose of this research is to study the friction and tribological behaviours of a new formulation containing slate powder as a ceramic filler, and other components involved in a novolak type phenolic resin matrix, with the purpose of evaluating the potential of this mining tailings rock in such formulations, and to reduce the environmental impacts resulting from its extractive activity.

Slate is a metamorphic rock that resembles clay and is composed of extremely fine material such as: muscovite, mica, quartz, titanium oxide, chlorite, among others [8]. It has a density of approximately 2.7 g/cm^3, which is half the density of barite, another material widely used in friction elements. Slate has interesting physical properties and its natural composition composed by solid lubricating (muscovite, mica, talc) and abrasive (quartz, titanium oxide) materials that render it a potential component for friction elements, such as average hardness (approximately 2.3 on the Mohs scale), low porosity, high mechanical strength, stable and weather resistant minerals [9].

This work proposes slate to apply as a new tribological reinforcement friction element, added, as a technological alternative to reuse of slate tailings, and so, to minimize the environmental problems of incorrect waste disposal.

2. Materials and methods

2.1. Materials

Industrial friction formulations contain 5–6 classes of components, classified according to their main function in the final product [10]. In this study 6 types of materials were used in 5 different categories. As a binder, the phenolic resin of the novolak type CCS 5159, in the form of a powder, containing hexamethylenetetramine as a curing agent, indicated for the manufacture of brake linings, was used as supplied by the company Comil Cover Sand Ind. Gray slate tailing, provided by Micapel Slate Company was used as filler in the formulations for the composites studied. One formulation was prepared with barite, supplied for the department of geology of the Federal University of Ouro Preto, to be used as the reference. Chopped glass fibers with an average length of 5 mm, supplied by Resinplast was used as a reinforcement agent and graphite from B Herzog Company as a lubricant. Alumina S3M supplied by Hindalco Brazil was used as abrasive.

Table 1 – Reference composite formulation (% mass).

Components	Formulation B
Barite	65
Fiberglass	10
Phenolic resin	10
Aluminum oxide	7.5
Graphite	7.5

Table 2 – Composites formulation (% mass).

Components	Formulation		
	A1	A2	A3
Slate	65	50	40
Fiberglass	10	10	10
Phenolic resin	10	25	35
Aluminum oxide	7.5	7.5	7.5
Graphite	7.5	7.5	7.5

2.2. Sample preparation

The formulation was based on prior work of Águila [11], and proposed to use slate particulates in substitution of other mineral components. in which achieved performance comparable to the commercial automotive brake pads with a relatively low raw materials diversity of components. It was also replaced the aramid fiber by glass fiber, which also ensures good mechanical properties, apart from lowering the cost of the composite, making it even more attractive. It should be noted that this material has not been optimized for best brake performance but contains the most important components that are currently used in conventional automotive brake systems.

The slate was comminuted by a jaw crusher. The resulting powder was wet sieved, in order to ensure that the particles were within a granulometric range of 105 μm - 12 μm, based on the work of Hamid, Stachowiak and Syahrullail [12], who investigated the effect of the size of grain particles embedded in friction elements.

In formulating the composite, we firstly chose to reproduce the best result found in Águila's work [11], as a parameter, which is shown Table 1, denoted as formulation B. Subsequently, formulations A1, A2 and A3 (Table 2) were established, in order to investigate the influence the slate content.

Thus, four test specimens were obtained: B, A1, A2 and A3.

For each formulation with the respective percentages of its components, 100 g of material was weighed. The mixing process lasted for about 1 min, in which all the material was

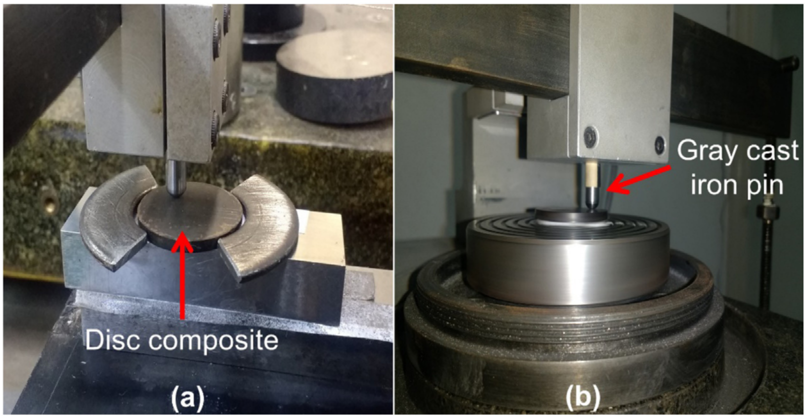

Fig. 1 – Pin-on-Disk tribometer. a) Adjusting the equipment, before the test. b) Test being performed.

already in the container in the form of powder or fiber. Each formulation was made in duplicate.

Then, parts of the mixture were packed in cylindrical metal mold and pressed at 160 °C for about 12 min with pressure values reaching 20 MPa; during this process there were pressure relief intervals for the degassing of the composite from the resin crosslinking reaction.

Finally, the molded pellets were maintained at 200 °C for 3 h in an oven in order to achieve a higher degree of cure.

2.3. *Surface characterization*

In order to know the morphology of the composites, the adhesion of the particulate and the fiber to the matrix, and the level of dispersion of these, an electronic scanning electron microscope (SEM) was used, model Vega 3 LM of Tescan (HV: 21 kV). The samples were covered with vaporized gold and images taken at a magnitude of 500×.

2.4. *Hardness testing*

Hardness of the composites was measured by a Rockwell hardness apparatus (HR) using the S-scale, according to ASTM D785 of 2008 [13]. A universal hardness equipment of the manufacturer Wolpert, model Dia Testor 2 Rc was used.

2.5. *Friction and wear coefficients testing*

This test was performed by a pin-on-disk tribometer in which a pin is loaded against a rotating disk (Fig. 1).

In order to reproduce real operating conditions, a gray cast iron was used as pin and the different composite formulations as disks. The cylindrical pin was 6 mm in diameter and 32 mm high, the disk samples were 30 mm in diameter and 3 mm thick. The pin was assembled under the arm of the equipment applying a uniform load of 1 kgf against the surface of the sample disk, which rotated at a constant speed of 0.5 m/s in the air. The total travel distance and the test time were respectively 1000 m (2000s).

Samples were washed in isopropyl alcohol and stored in a desiccator with silica gel for a minimum of 48 h (quarantine) before the experiment started and at the end of the experiment.

The coefficients of dynamic friction (COF) of the samples were calculated by the tangential force (F_T) between the two bodies in contact, pin and disk, divided by the acting normal force (F_N) on the disk, which were measured during the test. The COF was expressed by the relationship:

$$COF = \frac{F_T}{F_N}$$

Subsequently, the average coefficient of friction (μ_m) of each composite was evaluated, taking the mean of the values found in the stabilization period of the test, from 1500 to 2000s.

In order to calculate the mass wear, the samples were weighed at a resolution of 0.00001 g after the quarantine, just before the experiment and again shortly after quarantining the end of the experiment. The environmental conditions for the test were: 41 % relative humidity and temperature ranging from 23 ± 1 °C considered well established for this proposal.

3. Results and discussion

3.1. *Surface characterization*

Fig. 2 shows the general appearance of the friction elements prepared. The elements have a silvery gray color due to the slate and macroscopically present relatively smooth surfaces, where the presence of other components such as the glass fiber can be noticed.

From the SEM images of the composites (Fig. 3) it is possible to identify the glass fiber embedded by the polymer matrix, as well as some particles that can be attributed to the slate and alumina used in the formulation. The darkest surface seen at the background corresponds to the phenolic resin matrix and the most elongated constituent, in grayscale, are glass fibers.

Although the morphology is not particularly homogeneous, a result already predicted by Mutlu, Eldogan and Findik [14], it can be inferred that there is an efficient adhesion between the matrix and the other components, pointing out the interaction with fiberglass which is important to promote good surface protection and distribution of the stresses.

Fig. 2 – General appearance of the friction elements prepared.

3.2. *Hardness testing*

The values obtained from the hardness tests (Table 3) are compatible with those found in the literature [15,16], ranging from 50 to 100, and indicated that the porosity of the material decreases as the hardness increases. On the other hand, hardness values increased with increasing amount of phenolic resin content, and this was attributed to a more efficient involvement by the matrix which decreased the voids, since it fills the composite pores during the hot press step. This explains the low values of hardness found for sample A1, which can be attributed to difficulties during the manufacturing process, due to the excess of slate powder, and not enough resin to involve all the material. Although 65 % of mineral filler was used in formulation B, barite presents higher density than slate, so that a lower volume of material was used and the embossing of the components by the phenolic matrix presented no difficulty.

Table 3 – Results of composite hardness tests.

Composite	Hardness HR_S
A1	51.00 ± 2.08
A2	92.00 ± 0.20
A3	93.00 ± 0.81
B	86.00 ± 1.41

Table 4 – Results of the average friction coefficients of the different types of composites.

Composite	μ_m
A1	0.430 ± 0.008
A2	0.500 ± 0.014
A3	0.450 ± 0.007
B	0.400 ± 0.008

3.3. *Friction and wear coefficients testing*

Fig. 4 shows the results of the dynamic friction coefficient tests for all the samples, which together with the results of the average coefficients, calculated in the interval between 1500 and 2000s (Table 4), were generally comparable to values found in literature [17,18].

During the run-in stage the dynamic COF increases with time, except for formulation A3 where a slight increase was

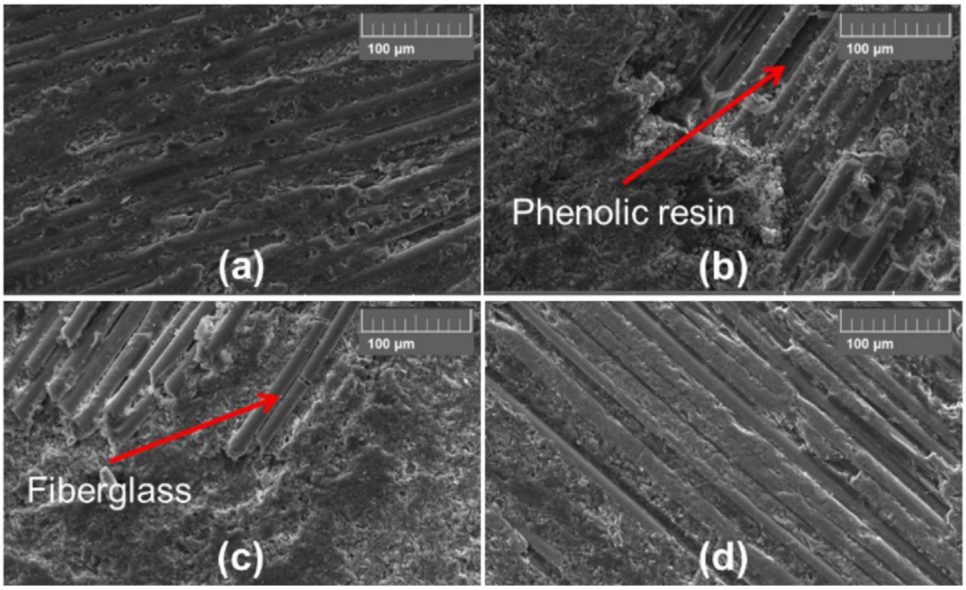

Fig. 3 – SEM images of the composites: (a) B, (b) A1, (c) A2 and (d) A3.

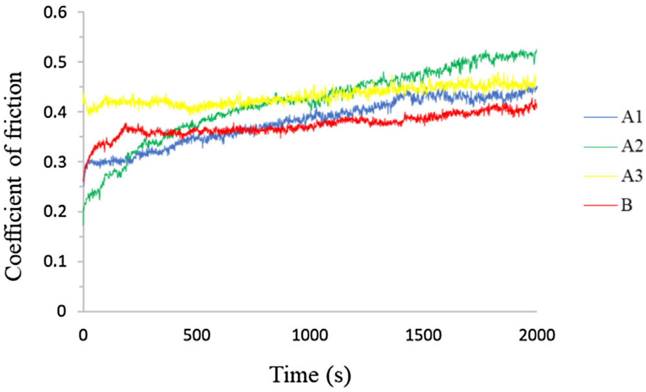

Fig. 4 – Dynamic friction coefficient sampling A1, A2, A3 and B.

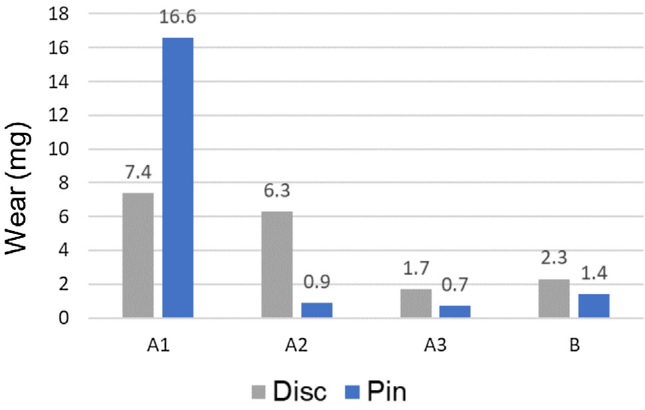

Fig. 5 – Wear Results of the different tribological pairs.

observed. This desirable behavior for a brake system, by which the surface conditions are preserved during endurance and suggests the maintenance of behavior over life, resembles type I wear curve according to Kato and Adachi [19] who investigated different friction models and proposed a classification for the wear mechanisms. The COF increase can be attributed to the progressive removal of the surface contaminants which causes an increase in the surface energies at the contacting asperities, leading to the formation of the contacting plateaus [20]. The run-in stage is quite prolonged for A1, A2 and B, because of the relatively high content of abrasive particles, influencing the dynamics of formation of secondary plateaus rich of Fe-oxide debris from cast iron pin [21].

The average friction coefficients (μ_m) obtained indicated a good performance of the formulated composites, suggesting a good regularity between the samples, with values within a range for use in automotive vehicles, as well as the reference model B, the values of which are predictably in accordance with those in the literature. Among the formulations prepared, the sample A3 presented the most stable dynamic friction coefficient, which was reflected in its lower standard deviation.

In evaluating automotive brake pads, Santos [22] found that in the commercial pads researched, the friction coefficients ranged from 0.30 to 0.40. Liew and Nirmal [23] in their studies, found coefficients of friction in the order of 0.40 for commercial tablets and values of up to 0.50 for new pads developed based on the substitution of asbestos.

Wear tests results (Fig. 5) showed that with increasing amount of slate and a concomitantly decrease in the amount of resin, there was an increase in disk and pin wear. In this present case, the predominant mechanism of wear is abrasive, where the pin made with gray iron has ductile mode, whereas the pad is a hybrid system between ductile (resin) and brittle (slate, fiberglass) and fracture toughness becomes of significant additional importance [19].

This was attributed to the fact that the fillers are strongly held by the matrix on the friction surface. As the braking action is triggered, a shear stress is generated on the surface causing the formation of loose debris from these fillers which either result in loss of structural integrity [18] or act as lubricant. In fact, Silveira [24] explains that the large amount of mineral filler used in the confection of the pellets renders them more abrasive. Therefore, the A3 formulation sample presented the best performance, with 40 % of slate and 35 % of resin, and the minimum wear of the mixture for both components of the tribological pair (disk and pin) can be understood as a performance commitment.

4. Conclusions

A series of composites of phenolic resins with residues of slate powder, in addition to other components, was prepared for the use of this material as friction elements. In the micrographs of the composites it is observed that they have heterogeneous structures, without significant differences, and it is possible to infer a good compatibility between the glass fiber and the polymer matrix.

Sample A1 presented a low hardness value and this was attributed to the higher porosity of the composite, when compared to the others, which presented values similar to those found in the literature.

The formulations did not show large variations in the coefficient of friction, with values slightly higher than the commercial pads. In relation to the mass wear, however, the composites showed a significant variation.

Formulation A3 resulted in a composite with good performance in friction tests and even better as far as wear is concerned. Therefore, a formulation containing 40 % by mass of slate was the best option among those investigated from a low cost to benefit ratio point of view. This is true mainly in relation to the low wear rate of the brake rotor which, in general, is the most costly part of a set that uses friction elements.

The increased slate content implied higher wear values in the material. Thus, the formulation containing 65 % of slate was not considered a good manufacturing option due to the increase in wear of the parts and consequently a shorter life. In addition, its preparation presented a number of difficulties, such as the presence of slate particles that were not adequately agglomerated due to insufficient phenolic resin.

Conflict of interest: none

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REFERENCES

[1] Österle W, Griepentrog M, Gross Th, Urban I. Chemical and microstructural changes induced by friction and wear of brakes. Wear 2001;251:1469–76.

[2] Öztürk B, Öztürk S. Effects of resin type and fiber length on the mechanical and tribological properties of brake friction materials. Tribol Lett 2011;42:339–50.

[3] Filip P, Weiss Z, Rafaja D. Friction layer formation in polymer matrix composite materials for brake applications. Wear 2002;252:189–98.

[4] Chan D, Stachowiak GW. Review of automotive brake friction materials. Arch Proc Inst Mech Eng Part D J Automob Eng 1989-1996 2005;218:953–66.

[5] Grazen FS, Buike M, Bryzinsky. Friction particles for brake lining. U.S Pat 3,873,490; 1975.

[6] Filip P. Friction and wear of polymer matrix composite materials for automotive braking industry. In: Barton D, Shilton B, editors. Braking 2002, professional engineering publishing ltd., Bury St. Edmunds. 2002. p. 341–54.

[7] Ghosh P, Ghosh D, Khastgir D, Chaki TK. Effect of Aramid Pulp and Lapinas Fiber on the Friction and Wear Behavior of NBR Powder-Modified Phenolic Resin Composite. Tribology Transactions 2016;59(3):391–8, http://dx.doi.org/10.1080/10402004.2015.1081999.

[8] Palhares LB. Addition of tailings from slate extraction to concrete. Pós em revista 2013;8:100–3.

[9] Silva M. Characterization of the products generated in the thermal expansion process of slate tailings [phd. Thesis]. In: Belo Horizonte: school of engineering. Federal University of Minas Gerais; 2005.

[10] Baklouti M, Cristol A, Desplanques Y, Elleuch R. Impact of the glass fibers addition on tribological behavior and braking performances of organic matrix composites for brake lining. Wear 2014;330(331):507–14.

[11] Águila Z. Formulation of composites reinforced with PANOX fibers and aramid pulp used in friction materials [M. Sc. Dissertation]. Campinas: school of Chemical Engineering, State University of Campinas; 1999.

[12] Hamid M, Stachowiak G, Syahrullail S. The effect of external grit particle size on friction coefficients and grit embedment of brake friction material. Procedia Eng 2013;68:7–11.

[13] ASTM. D785-03- standard test method for rockwell hardness of plastics and electrical insulating. Annu Book ASTM Stand Sect 11 Water Environ Technol 2008;14:1–6.

[14] Mutlu I, Eldogan O, Findik F. Tribological properties of some phenolic composites suggested for automotive brakes. Tribol Int 2016;39:317–25.

[15] Cho M, Kim S, Kim D, Jang H. Effects of ingredients on tribological characteristics of a brake lining: an experimental case study. Wear 2005;258:1682–7.

[16] Kim S, Cho M, Lim D, Jang H. Synergistic effects of aramid pulp and potassium titanate whiskers in the automotive friction material. Wear 2001;251:1484–91.

[17] Aranganathan N, Mahale V, Bijwe J. Effects of aramid fiber concentration on the friction and wear characteristics of non-asbestos organic friction composites using standardized braking tests. Wear 2016;354(355):69–77.

[18] Cai P, Wang Y, Wang T, Wang Q. Effect of resins on thermal, mechanical and tribological properties of friction materials. Tribiology International 2015;87:1–10.

[19] Kato K, Adachi K. Wear mechanisms. In: BHUSHAN B, editor. Modern tribology handbook: principles of tribology. [S.L.]. CRC Press; 2001, v. 1 Cap. 7.

[20] Blau PJ. On the nature of running-in. Tribol Int 2005;38:1007–12.

[21] Federici M, Gialanella S, Leonardi M, Perricone G, Straffelini G. A preliminary investigation on the use of the pin-on-disc test to simulate off-brake friction and wear characteristics of friction materials. Wear 2018;410(411):202–9.

[22] Santos AHC. Evaluation of the performance of national automotive brake pads [M. Sc. Dissertation]. Campinas: school of Mechanical Engineering, State University of Campinas; 2005.

[23] Liew K, Nirmal U. Frictional performance evaluation of newly designed brake pad materials. Mater Des 2013;48:25–33.

[24] Silveira JVW. Development of a new brake pad material based on substitution of phenol-formaldehyde resin for lignosulfonate [M. Sc. dissertation]. Campinas: school of chemical engineering, state university of campinas; 2010.