



Effect of the addition of carbonaceous fibers on the tribological behavior of a phenolic resin sliding against cast iron

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ABSTRACT

The tribological behavior of novolac phenolic resin matrix composites reinforced with three kinds of carbonaceous fibers was studied in sliding contact against cast iron. Slow pyrolysis was used to obtain carbonaceous fibers from Colombian plantain fiber bundles (crops residues from Urabá region). After the carbonization process the samples were heated up to either 1200 or 1400 °C ensuring that many morphological aspects of the natural fibers were retained. Then, novolac phenolic resin with HMTA as curing agent and the carbonaceous fibers were used to obtain a composite material by compression molding process. Samples with different type and volume fraction of carbonaceous fibers were prepared and tested in sliding contact against cast iron in a pin-on-disc wear testing machine. At the end of the tests, the worn surfaces and the debris were analyzed by SEM.

A decrease in both friction coefficient and wear of composites was observed with the increase in fiber volume fraction, which was associated to a beneficial effect of the detachment of carbonaceous material from the worn surface. Under the tested conditions, this material remains at the interface between the composite and the cast iron and helps reduce the shear resistance of the interface. On the other hand, surface fatigue and adhesion wear was identified as the dominant wear mechanism of the phenolic resin matrix.

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1. Introduction

Pyrolysis process transforms lignocellulosic materials into charcoal and slow heating rate pyrolysis gives rise to a porous carbon frame with the morphology derived from its precursor [1–3], which can be used to ceramic synthesis such as silicon carbide, alumina and titania [3]. Typical lignocellulosic precursors of charcoal materials that retain morphological aspects of precursor are wood resources. Although non-wood plants are used to get carbon powders and activated carbon, fibrous wastes from agricultural residues of edibles fruits from *Musa* species such as commercial plantain (*Musa AAB*, cv “Dominico Harton”) produced in Colombia have been used to get carbonaceous fibers [4–7].

The sliding friction coefficients of some carbon-based materials, either amorphous or crystalline, are among the lowest for any solids. They are important ingredients of brake composite materials including different types such as graphite, coke, carbon black, and carbon fiber [8]. Charcoal and carbonaceous substances, for instance, have good frictional characteristics even though they do not exhibit the basal slip properties of graphitic structures.

On the other hand, novolac phenolic resin is a common binder for resin-based friction materials [9]. Tribological applications of phenolic resins are usually limited due to their relatively poor stability and wear resistance. Therefore, it is imperative to incorporate various reinforcing and filling constituents such as reinforcing fibers, abrasives, binders, fillers, and friction modifiers (solid lubricants) into phenolic resin-based friction composites with the purpose of increasing their stability and wear resistance [10–12].

The type and relative amount of solid lubricants and abrasives in brake friction materials significantly affect the brake performance [13]. Solid lubricants are added in relatively small amounts but they strongly affect wear resistance, stopping distance, friction stability and torque variation. Graphite and MoS₂ are frequently used in commercial brake linings and other chalcogenide compounds such as Sb₂S₃, ZnS, PbS, and Cu₂S are often added for better brake performance. However, a few reports focused on the effect of solid lubricants on tribological properties of solid films coated on the metal substrates are available [14,15].

Charcoal in particular has been reported as a potential replacement for graphite into composites for antifriction and antiwear applications as aluminum alloys applications [16,17], wood ceramics and in brake friction materials using carbonized coconut char powders [18]. Nevertheless, it is rarely discussed in the literature the effect of charcoal materials on wear mechanisms acting on the

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surfaces in contact. In this work, novolac phenolic resin matrix composites reinforced with three kinds of carbonaceous fibers and different volume fraction of reinforcement were prepared. Chemical composition and structural characteristics of carbonaceous fibers were studied. Tribological behavior of composites was tested in sliding contact against cast iron in a pin-on-disc wear testing machine. Worn surfaces were studied by SEM and wear mechanisms were identified.

2. Materials and experimental methods

2.1. Materials

Carbonaceous fibers were obtained by pyrolysis of plantain fibers from leaf sheaths from Urabá, Colombia region. A mechanical extraction process for plantain fibers was used according to the method described in [19]. The samples were further air-dried for at least 24 h and milled with a RETSCH SM 100 (Haan, Germany) to obtain a particle size smaller than 5 mm.

Three types of carbonaceous fibers (T 1000, T 1200, T 1400) were synthesized by slow pyrolysis performed in an electrical tube furnace MTI GSL1600-80X. T 1000 samples were heated from room temperature up to 1000 °C, held at this temperature for 90 min and then cooled down to room temperature in N₂ atmosphere with 99.9999% purity. T 1200 and T 1400 samples were initially treated under the same conditions used for T 1000 samples, and then submitted to a second thermal process at 1200 °C and 1400 °C, respectively. The heating (5 °C min⁻¹) and cooling (-5 °C min⁻¹) rates, as well as the nitrogen flow rate (200 ml min⁻¹), were the same for all the samples.

Phenolic resin powder containing hexamethylenetetramine as curing agent was used as the matrix. Resin and carbonaceous fibers were molded by compression molding process. Previously, both components were mixed for 5 min by stirring them in a blender so as to improve the dispersion of the mixture. Contents of carbonaceous fibers were 2.5, 5.0, 7.5, 10 and 12.5% (v/v) and phenolic resin was the balance. Fully mixed raw material was loaded into a 140 mm diameter and 3.5 mm high steel mold and hot pressed at 200 bar, 165 °C for 15 min in a thermo hydraulic pressing machine.

2.2. Experimental

The carbon, hydrogen, nitrogen and sulphur content of the carbonaceous fiber bundles were determined using a LECO-CHNS-932 microanalyzer. Oxygen content was determined by means of a LECO-VTF-900 furnace coupled to the microanalyzer. Ash content and moisture were determined for each carbon content in accordance with UNE 32001 and 32002 standards, respectively. For XRD analysis, carbonaceous fibers were ground to a fine powder and the measurements were carried out in X'Pert Pro MPD, Panalytical, diffractometer operating at 25 mA and 40 kV, using Cu-K α radiation.

Table 1
Metallurgical and chemical characteristics of cast iron disc.

Nominal chemical composition (wt.%)	C	3.65–3.85	Sn	Max. 0.10
	Si	2.15–2.795	Cu	Max. 0.60
	S	Max. 0.15	Mo	Max. 0.10
	P	Max. 0.10	Ni	Max. 0.20
	Mn	0.5–0.9	Fe	Balance
	Cr	Max. 0.25		
	Microstructure	Graphite type: 1–1A (sheets)		
Grain size: ASTM 3–4				
Pearlite: 90% min.				
Ferrite: 5% max.				
Hardness	Cementite: 5% max.			
	170–217 HB			

Table 2
Pin-on-disc testing conditions.

Tribological test conditions		
Environmental temperature	25 ± 2	°C
Atmosphere	Air	–
Relative speed	1	m s ⁻¹
Load	15.11	N
Test distance	1000	m

2.3. Tribological behavior

Friction and wear behavior of the phenolic resin and composites were evaluated by using a pin-on-disc tribometer. A cast iron brake disc (220 mm in diameter and 8 mm in thickness) was adapted for the tests and the surface finishing was fixed to circa Ra = 0.5 μm in all the samples. Table 1 presents some of the most relevant metallurgical and chemical characteristics of cast iron disc.

Phenolic and composite pins were glued to a metallic pin holder with conventional instant glue and machined by lathe turning to get the final dimensions shown in Fig. 1a. The pin was fixed to a rigid arm (Fig. 1b) and put in contact with the surface of the disk in movement, while a normal load was applied by dead weights (Fig. 1c). A general view of the equipment is shown in Fig. 1d. All samples were tested under the same test conditions summarized in Table 2 and three replicas were obtained for each experimental condition. Friction force was registered 10 times per second with the aid of a load cell connected to a data acquisition card and software Labview 5.1 provided by National Instruments under an educational contract. After each friction test, the pins were ultrasonically cleaned in alcohol for 5 min, dried in air and weighed in an analytical balance Sartorius CPA225D with resolving power of 0.01 mg.

2.4. Surface examination

The worn surfaces of disks and pins were examined by stereomicroscopy (Nikon SMZ1500) and scanning electron microscopy (JEOL 5910LV) in order to identify the main wear mechanisms acting during the tests.

3. Results and discussion

Elemental composition of fibers after pyrolysis process and second thermal treatment are shown in Table 3. All samples have high amount of carbon as a consequence of carbonization of lignocellulosic compounds. A second thermal treatment increases the carbon content and reduces the amount of oxygen and hydrogen by volatilization. These changes are considered typical in carbon materials due to exposure to high temperature [20].

Carbonaceous fibers produced from plantain fibers bundles were crystallographically characterized by means of X-ray diffrac-

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