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RESEARCH

Investigation on the Tribological Properties of an Organic Matrix Composite Material Designed for Brake Applications

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A B S T R A C T

Metal and organic matrix composites are extensively used in the automotive industry and handling equipment like cranes, forklifts, overhead cranes, and elevators. One of the primary applications for these materials is in brake linings, where the ability to halt movement through braking is crucial for both safety and environmental protection. This experimental study involves tests conducted on a tribometer, using two different materials for the brake linings against a cast iron drum, the first sample (sample I) is a commercially available brake lining, and the second (sample II) is a locally prepared one. A comparative analysis was performed for both samples under identical experimental conditions, using the same normal load (5 and 10 N) and sliding speed (35 and 50 mm/s). Measurements taken during each test include mass loss, tangential force, and friction coefficient. Additionally, the wear tracks were examined microscopically to understand the observed phenomena and to identify a material with optimal performance. The obtained results from energy-dispersive X-ray analysis (EDAX) conducted via scanning electron microscopy (SEM) and shore hardness measurements are compared with values available in the technical literature and standard requirements. The measured friction coefficient (μ = 0.354 to 0.47) falls within the range specified by quality control standards for effective braking. Sample II demonstrated a wear rate of 4.57×10^{-8} cm^3/N.m, significantly outperforming Sample I, which had a wear rate of 11.53×10^{-8} cm^3/N.m. This highlights Sample II's superior wear resistance and its potential for brake lining applications.

1. INTRODUCTION

Brake pads are a crucial part of any vehicle's braking system, designed to grip the brake rotor and slow down or stop the vehicle. They come in different sizes and materials such as ceramic, metallic, and organic, with varying durability, noise levels, and cost. Routine brake pad checks and replacements are necessary for optimal performance and to ensure safe driving. Worn-out brake pads produce screeching or grinding noise during braking. It is of paramount importance to use high-quality brake pads for optimal safety and performance.

Technical standards for high-quality brake pads depend on the material used and regional regulations. Generally, high-quality brake pads need to offer consistent performance over a broad range of temperatures, resistance to wear and noise, as well as producing minimal dust and vibrations. The materials used must be able to withstand high heat and provide maximum friction with minimal rotor wear.

Traditional friction-based brake pads, pose environmental and health risks, especially from asbestos use [1]. They also contribute to heavy metal contamination via brake dust. Lower quality brake pads can cause vibrations, noise, reduced friction, and fading performance, compromising safety. There is a growing interest in safer, eco-friendly alternatives like carbon fiber-reinforced brake pads.

Hasan Öktema et al. [2] evaluated a new brake lining composition using Petro-coke powder as a friction adjuster, demonstrating improved stabilization of the friction coefficient and specific wear rate compared to commercial brake pads. Wear tests were conducted using a specially designed device simulating real-world conditions, highlighting the effectiveness of this brake lining composition for friction modification.

A. Daoud and M.T.Abou El-khair [3] investigated a study of wear and friction behavior of a sand cast brake rotor made of A359-20 vol % SiC particle composites sliding against automobile friction material. Their study aimed to investigate the effect of load range of 30–100N and speed range of 3–12 m/s on wear rates and friction coefficients, using a pin-on-disc type apparatus. Results of the study showed that wear rates decreased with increasing load from 30 to 50N, but then increased with further load increase up to 100N. However, wear rate decreased with increasing sliding speed at all load levels.

In 2014, M. Djafri et al. [4], Conducted a study examining the tribological performance of three different brake disc materials, cast iron (FG25), chromium steel (100Cr6) and an Al MMC-based aluminum composite (A359/SiCp), sliding against a commercial resin-based brake pad material. The author's main objective was to analyze the effects of applied normal load, sliding velocity, relative humidity and salt spray corrosion on friction behavior.

The parameters used to produce the friction material have an influence on the obtained characteristics:

Hentati et al. [5], investigated the impact of post curing duration on the mechanical, thermal, and tribological behavior of friction materials. Longer post curing durations were found to decrease thermal conductivity, homogenize surface mechanical properties, reduce friction levels, and increase wear resistance.

Brake pads and discs in automotive systems must meet requirements such as stable friction coefficient, minimal wear, and suitable thermal conductivity [6-11]. Asbestos, despite its mechanical and thermal properties, poses health hazards, prompting a shift to safer materials. Studies have explored eco-friendly alternatives, with Zhang S.Y et al., [12] studying the abrasive behavior of brake pads, Yun Rongping et al., [13] evaluating eco-friendly brake materials, and Cho Min Hyung et al., [14] investigating the influence of ingredients on brake lining tribological characteristics.

Different materials such as Kevlar 29, natural composites, and metal matrix composites have been studied for their suitability in terms of heat resistance, friction coefficient, and weight reduction (Trimble et al., Yavuz, Bayrakçeken et al.) [15,16]. The performance of brake pads has been evaluated through various tests including wear, hardness, tensile, impact, and microstructural analysis (Sundarrajan, Jeyachandran et al.) [17].

Hendry and Bachchhav conducted studies on non-asbestos brake pad materials where they investigated the tribological properties of CL-3003 [18] and AF-22 [19] using pin-on-disc tribometers. These studies revealed that sliding speed significantly affected friction, while temperature mainly affected the wear rate.

Our research aims to identify the factors affecting the frictional performance, including coefficient of friction (COF) and wear, of brake lining, the first sample is commercially available brake lining, and the second is locally manufactured one. Our goal is to analyze these factors and cooperate with national manufacturers to develop environmentally friendly materials that provide better cost-effectiveness and contribute to the growth of the national economy. In addition, this study emphasizes the importance of laboratory tribological testing by experimentally determining the actual values of density, chemical composition, coefficient of friction, and wear rate, which increases the reliability and quality of the results.

2. MATERIALS AND EXPERIMENTAL PROCEDURES

2.1 Equipment used

The experiments were conducted using new brake linings (organic matrix composite materials), the composition of which is described later in Section 3.3) along with commercially available cast iron drum, and the specimen was then prepared through machining, finishing and polishing for characterization in the initial stage.

A quantitative and qualitative analysis using X-ray fluorescence spectrometry method allowed for the determination of the chemical composition and constituent elements. Hardness measurements were performed using a Mitutoyo HH-401 Harmatic durometer.

The density of each sample was experimentally calculated using the Archimedes' method. A CSM instruments DTR 70090 tribometer was used to conduct wear tests and determine friction coefficients under dry friction (without lubrication) by varying material parameters, normal load, and sliding velocity. Precision weighing before and after each test using a Kern balance was performed to evaluate wear rate.

The surfaces of the wear tracks were observed. Optical microscopy and scanning electron microscopy (SEM) with energy-dispersive X-ray analysis (EDAX) were employed to observe the wear tracks and analyze debris after the tests. Figure1 illustrates the experimental equipment used.

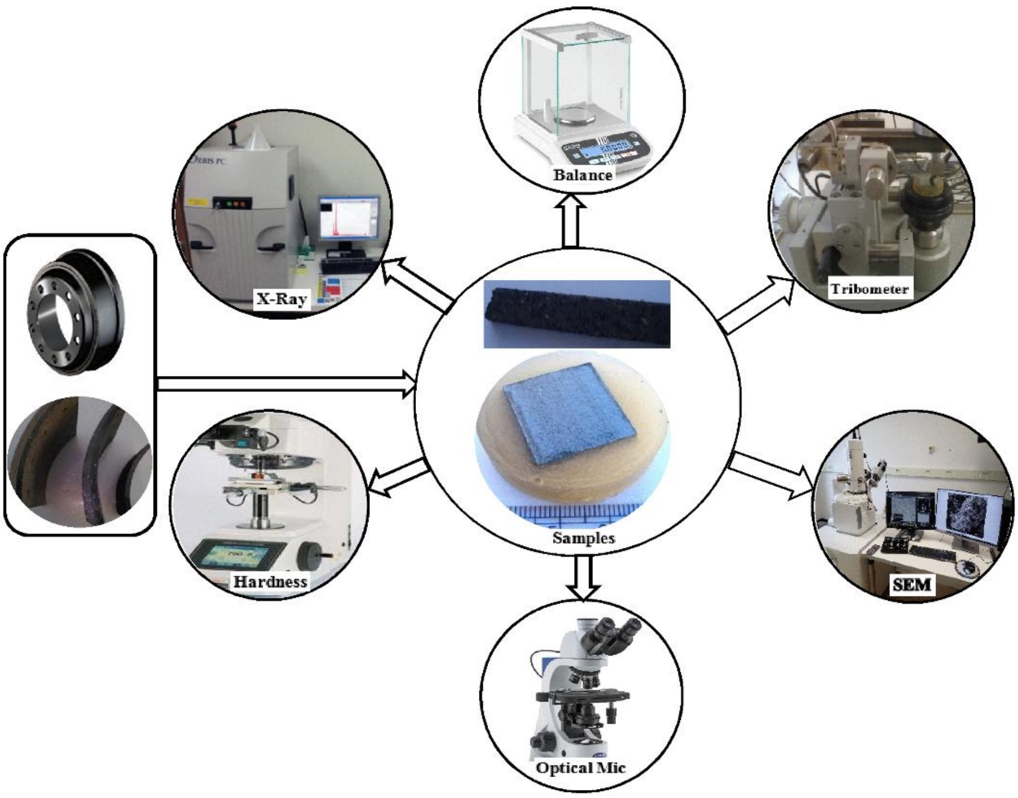


Fig. 1. The experimental equipment used.

2.2 Samples preparation

According to ASTM G99, standards for friction testing, samples were meticulously prepared to fit on a tribometer sample holder, forming a pin-disc couple [20]. To retain the original characteristics of the cast iron truck brake drum, a water-lubricated saw was used to cut the brake linings, mitigating overheating and unwanted variations. Square sections measuring 20x20 mm were prepared and embedded in resin to address flatness issues, their surfaces polished to a roughness of Ra = 0.8 µm. Two brake pads of differing materials were chosen for the friction test pins, one commercially available (sample I) and the other locally developed (sample II). Pins cut and polished to dimensions of 4x4x30 mm were analysed using X-ray Fluorescence (XRF) technology, enabling material loss assessment during testing.

3. CHARACTERIZATION OF SAMPLES

3.1 Hardness measurement

The hardness of the drum component was assessed using an INOVATEST brand durometer, recording a value of 57 HRc. Shore hardness (HS) for the brake linings was determined using a Mitutoyo Hardmatic durometer, yielding values of 32 HS for Sample I and 36 HS for Sample II.

3.2 Density measurement

The determination of the density was made using the gravimetric method (called Archimedes' one). Samples were weighted under normal laboratory temperature and humidity conditions using a digital scale. The sample volume is evaluated using precision graduated beakers ± 0.5ml filled with water. The obtained density values for the two samples II) and (I were *2.6 g/cm³* and *2.19 g/cm³*, respectively.

3.3 Determination of chemical brake lining composition

To determine the chemical composition of brake linings, a combination of XRF spectrometry methods using a BRUKER S8 TIGER analyzer with a reliability of *99%*, enabling precise determination of the composition by element and by oxide. Additionally, the carbon-sulphur percentage was determined through a loss on ignition test at 1000°C, using a LECO CCr analyzer.

This approach provided valuable insights into the carbon and sulfide content within the brake linings. The results obtained were presented in detailed chart (Fig. 2), offering a comprehensive representation of the composition analysis. XRF analysis, a non-destructive technique, uses a spectrometer to measure the intensity of fluorescent radiation emitted by a sample after irradiation with X-rays.

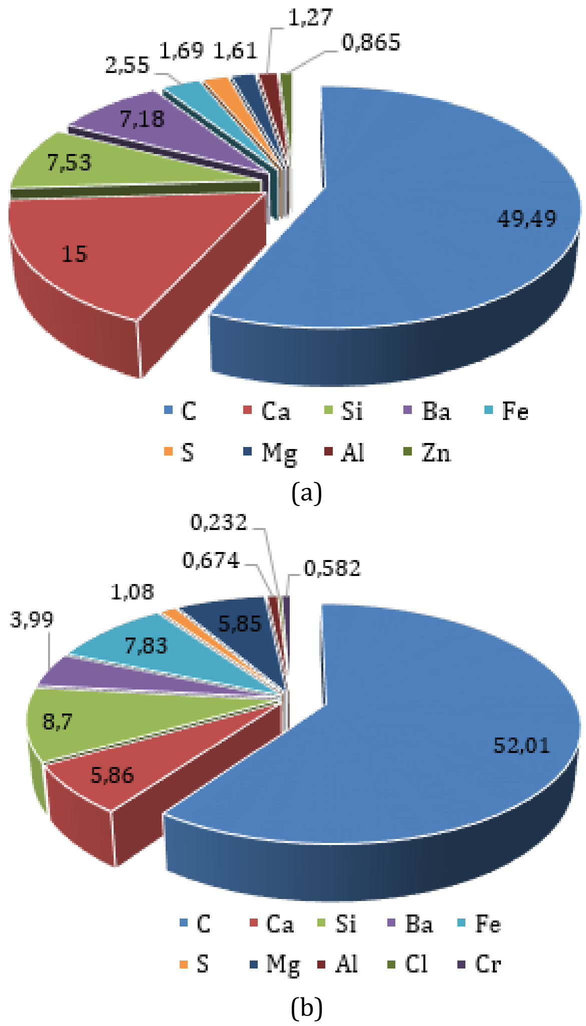


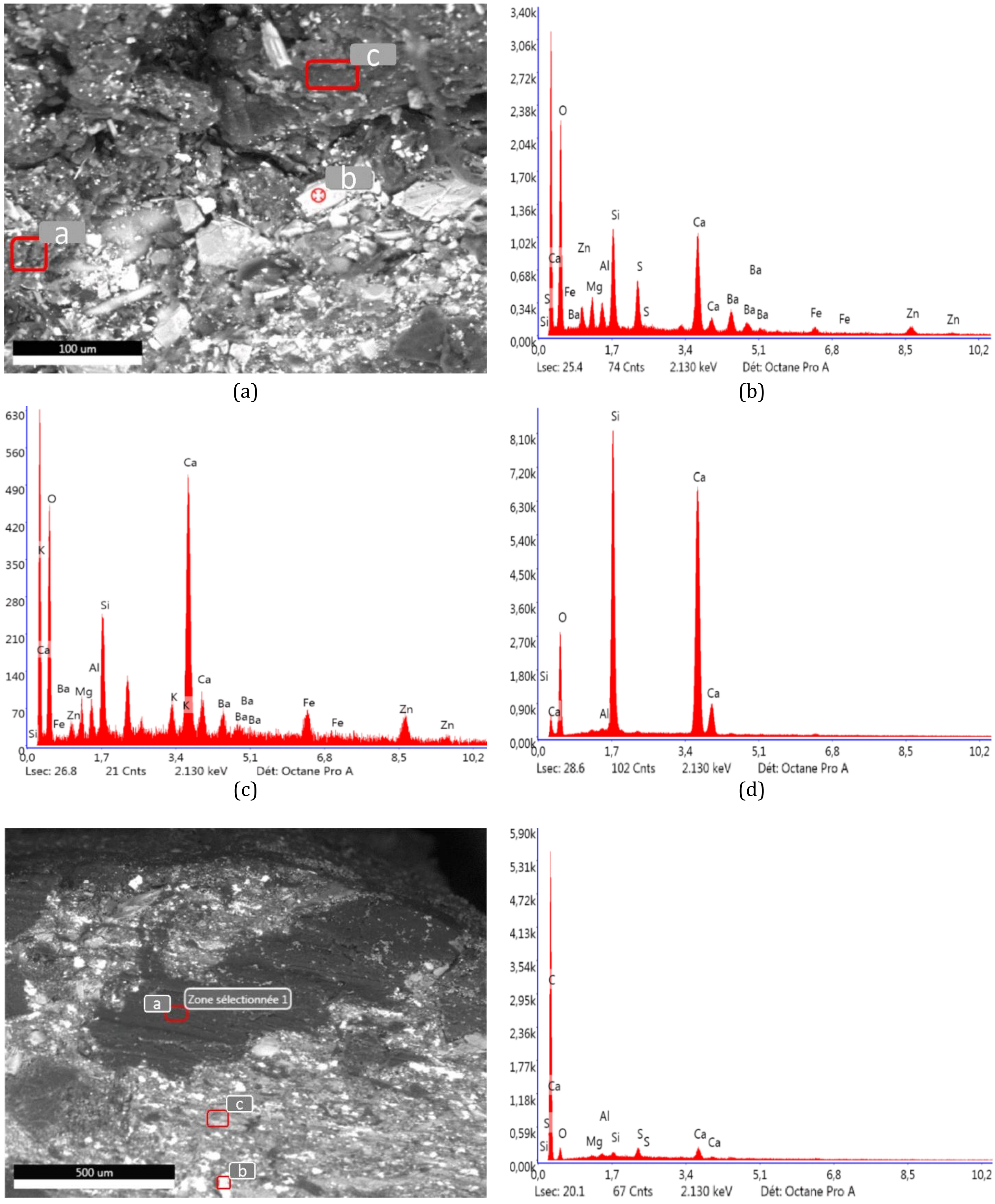
Fig. 2. X-Ray fluorescence spectrometry Samples analysis, (a) sample I and (b) sample II.

It provides critical information for process optimization, material selection, and product development, ensuring compliance with regulatory standards. The results demonstrate differences in the percentage of elements in sample I and sample II.

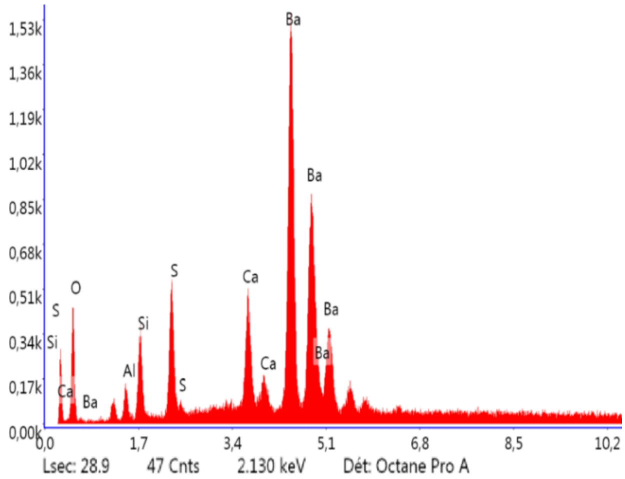
3.4 SEM and EDS analysis: Quantitative and qualitative examination

The QUANTA 250 scanning electron microscope from EDAX Ametek was utilized, equipped with precise EDS analysis capabilities to determine the percentage of constituent elements. Its exceptional high-resolution imaging capability enhanced our ability to observe and analyse intricate microscopic details of the samples under investigation (Fig. 3).

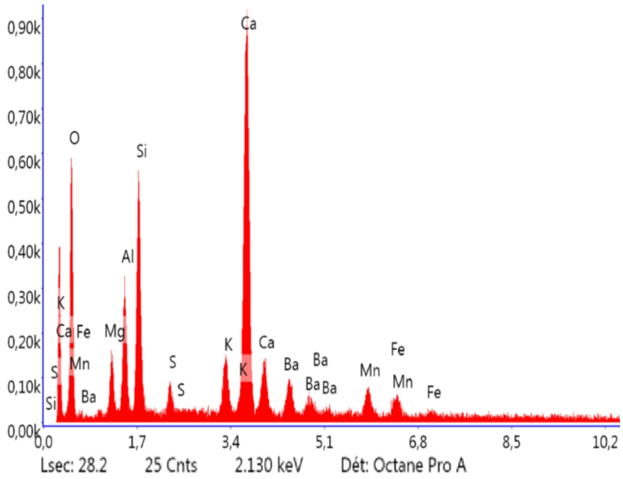
Figure 3 provides a view of the sample's microstructure, revealing an array of diverse particles held together by phenolic resin.



(a) (b) (c) (d) (e) (f)



(g)



(h)

Fig. 3. SEM observations of the surface of (a) sample I, (e) sample II, EDS analysis of samples I and II at locations (a, b, c), (b), (c), and (d) EDS analysis of sample I, (f), (g), and (h) EDS analysis of sample II.

Inferences drawn from the Energy Dispersive Spectroscopy (EDS) results suggest that these samples likely contain a range of compounds, including $CaCO_3$, CaO, Al_2O_3, MgO, graphite (C), SiO_2, BaO, and iron oxide. The detection of carbon (C) and oxygen (O) elements can be attributed to the phenolic resin within the composite brake block. The presence of SiO_2, Al_2O_3, and CaO particles hints at the utilization of glass fibre for reinforcement purposes, a hypothesis that gains credence from the XRF analysis depicted in Figure 2. Glass fibre, commonly used in industrial applications, is primarily composed of SiO_2, Al_2O_3, and CaO.

3.5 Tribometer wear test

Experimental tests were conducted using a CSM instrument DTRB70090 tribometer (Fig. 4) that operates on the pin-disc principle. The tests involved the manipulation of two key parameters: the normal load and sliding speed (*Fn_1= 5N, Fn_2= 10N, V_1= 35 mm/s, V_2= 50 mm/s)*.

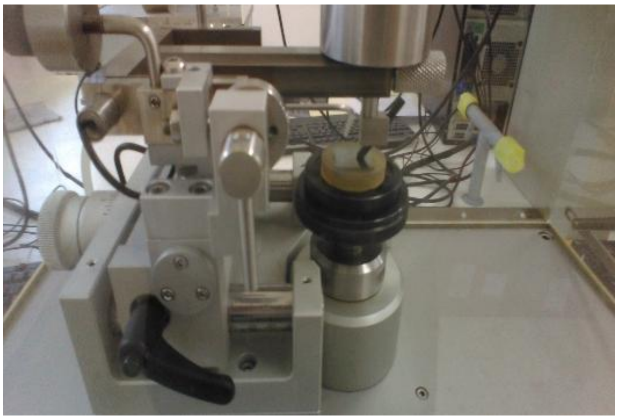


Fig. 4. Test on tribometer.

These parameters were varied to simulate different operating conditions. The duration of each test was set at 30 minutes, corresponding to a sliding distance of 100 meters. The tests were carried out under controlled room temperature conditions of 25°C.

To address the challenge of maintaining straightness during the tests, the cast iron samples, measuring 20mm x 20mm, were carefully embedded in a resin. Subsequently, the samples were meticulously polished to achieve a consistent roughness across all specimens. The slider used in the tribometer consisted of composite material pins with dimensions of 4x4x30mm.The arrangement of the pins facilitated a plane/plane contact configuration, allowing for precise and controlled interaction between the pin and disc surfaces.

After each test on the tribometer, detailed observations of the wear tracks were conducted using an optical microscope. This microscopic analysis provided valuable insights into the wear patterns and mechanisms occurring at the interface. Additionally, the mass loss of the samples was quantitatively evaluated by weighing them using a high-precision balance with an accuracy of 10^{-4} g, as shown in table1. The tribological behavior is evaluated by determining the friction coefficient (μ) and mass loss.

- Friction coefficient :

$$\mu = \frac{F_t}{F_n} \quad (1)$$

With: F_t the tangential force and F_n the normal force.

- The wear rate :

$$W_s = \frac{m_1 - m_2}{\rho \times L \times F_n} \quad (2)$$

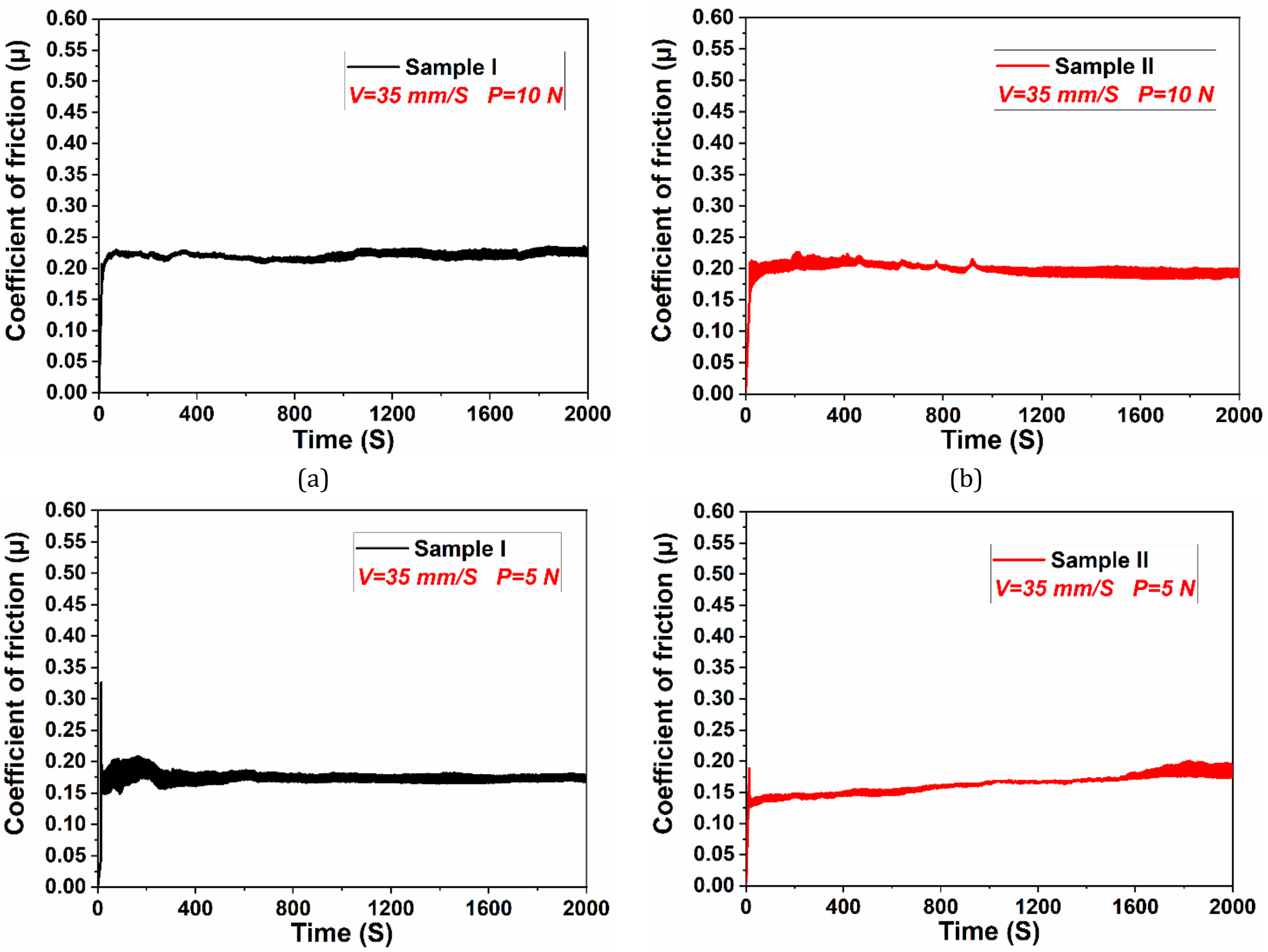
Ws: wear rate [*cm³/N.m*], m_1: weight before test [g], m_2: weight after test[g], *ρ*: density [*g/cm³*], *L*: distance covered [*m*], F_n: normal force [*N*].

4. RESULTS AND DISCUSSION

4.1 Friction behavior

The comparative analysis between the commercial brake pad sample and the prepared one was conducted under identical experimental condition by choosing the same speed and force, where the force and speed were chosen as follows, respectively *(5 and 10 N), (35 and 50 mm/s).*

The examination of Figure 5 (a, b, c, d, e, f, g, and h) reveals a noticeable resemblance in the temporal evolution of the coefficient of friction when considering the aforementioned experimental variables. This coefficient of friction holds significant significance in braking tests as it signifies the proportion between the frictional force exerted between the brake pad and brake disc surfaces and the compressive force applied to merge them. It serves as a critical determinant of the braking system's efficacy. Additionally, Figure 5 (e, f, g, h) corroborates the proximity of the coefficient of friction to established standard values. Generally, original equipment manufacturer (OEM) brake pads exhibit a higher coefficient of friction ranging from 0.3 to 0.4. Conversely, high-performance brake pads, engineered to endure the most demanding driving conditions, often exhibit a coefficient of friction in the range of 0.4 to 0.5. Nonetheless, it is essential to acknowledge that these values can be subject to variation based on various factors inclusive of the specific composition of the brake pads, the surface roughness of the brake disc, and the prevailing operating conditions.



(a) (b)

(c) (d)

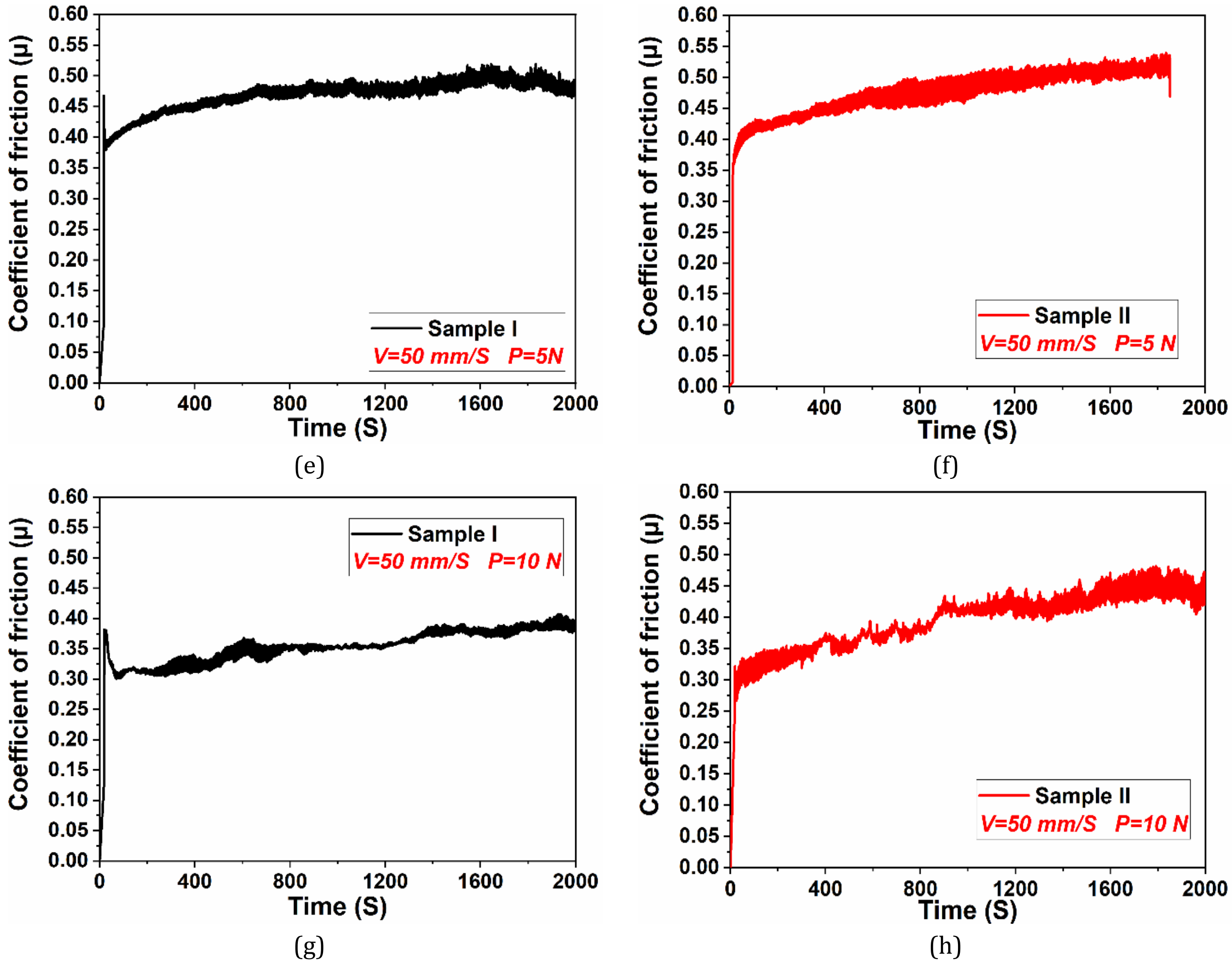


Fig. 5. Variation of the coefficient of friction between the disc-pad pair over time for samples I, II under different sliding speeds and normal loads.

4.2 Wear behavior

Mass loss tests on brake pad samples are crucial for determining wear performance and their durability and effectiveness. These tests provide insights into the materials wear resistance, which is fundamental to reliable braking performance. The resulting data contributes to material improvements in brake lining production, extending their lifespan and reducing replacement frequency. Thus, mass loss tests are vital for brake lining material development and maintaining braking system safety standards. In this research, a balance with an accuracy of 10^{-4} g was used to measure the masses of the samples.

Table 1. Experimental mass loss values and calculated wear rate.

Sample (S)	**S(I)**	**S(II)**	**S(I)**	**S(II)**	**S(I)**	**S(II)**	**S(I)**	**S(II)**
V/F_n	35/5	35/5	35/10	35/10	50/5	50/5	50/10	50/10
m_1 [g]	1,0256	1,0731	0,7382	0,5848	0,7413	0,7171	0,7871	1,0721
m_2 [g]	1,0255	1,0727	0,7379	0,5847	0,7410	0,7167	0,7868	1,0720
$Ml \times 10^{-3}$ [g]	0,1	0,4	0,3	0,1	0,3	0,4	0,3	0,1
$W_s \times 10^{-8}$ [Cm³/N.m]	7,6923	36,5296	11.5384	4.5662	23,0769	36,5296	11,5384	4.5662

m_1: Weight before test, m_2: Weight after test, Ml: Mass loss, V: Speed [mm/s], F_n: Load [N], W_s: wear rate [cm³/N.m].

The analysis of mass loss and wear rates for the two brake lining samples, Sample I (commercially available) and Sample II (locally developed), reveals significant differences in their wear resistance under varying testing conditions. At a low load and velocity (35 mm/s, 5 N), Sample I exhibited a minimal mass loss of 0.1 mg with a wear rate of 7.6923×10^{-8} Cm^3/N.m. However, under the same conditions, Sample II displayed a higher mass loss of 0.4 mg, resulting in a considerably higher wear rate of 36.5296×10^{-8} Cm^3/N.m. This suggests that Sample I initially performs better under low-stress conditions, with less material degradation.

However, the performance dynamics shift under increased load and velocity. When subjected to 35 mm/s and 10 N, Sample I's wear rate escalated to 11.5384×10^{-8} Cm^3/N.m, accompanied by a mass loss of 0.3 mg, indicating a notable increase in material wear. On the other hand, Sample II's wear rate decreased significantly to 4.5662×10^{-8} Cm^3/N.m with a much lower mass loss of 0.1 mg under the same conditions, suggesting superior wear resistance as the operational stress increases. The trend continues as the load and velocity are further heightened to 50 mm/s and 10 N, where Sample I maintains a wear rate of 11.5384×10^{-8} Cm^3/N.m, while Sample II demonstrates a much lower wear rate of 4.5662 $\times 10^{-8}$ Cm^3/N.m with a minimal mass loss of 0.1 mg. These results indicate that Sample II's material composition provides enhanced durability and consistent performance, particularly in high-stress environments, making it a more suitable candidate for demanding brake applications.

We explain the decrease in the wear rate despite the increase in speed due to the chemical nature of the samples and the proportion of components in each sample, taking into account the heterogeneity of the sample, as the geometric shape of the surface contact, which directly affects the results.

A comparison of the XRF analysis results for Sample I and Sample II reveals notable differences in their chemical compositions:

- The elemental percentages of Ca, Ba, Fe, and Mg exhibit significant variations between the two samples. Specifically, Sample I contains 15% Ca, 7.18% Ba, 2.55% Fe, and 1.61% Mg, whereas Sample II comprises 5.86% Ca, 3.99% Ba, 7.83% Fe, and 5.85% Mg.
- Calcium (Ca), a constituent of lime (CaO) and calcium carbonate ($CaCO3$), serves as a filler that has been shown to have a non-inert effect, enhancing the density, wear resistance, and mechanical resistance of materials. This difference in calcium content may contribute to the distinct properties observed between the two samples.
- Furthermore, the presence of magnesium (Mg), a component of magnesium oxide (MgO), which_acts as a reinforcing fiber, is known to improve tribological and mechanical properties [21, 22]. The varying Mg content in the two samples may also influence their performance.

To deepen the discussion on the influence of chemical composition on brake pad wear resistance, the study by Maleque et al. [21] provides valuable insights. Their research on natural fiber reinforced aluminium composites for automotive brake pads supports the idea that material engineering significantly impacts performance, particularly in terms of durability and wear resistance. Similarly, E. Surojo et al. [22] and T.R. Prabhu [23] have explored the effects of composite material design on wear characteristics, further emphasizing the critical role of chemical composition in enhancing the functionality and longevity of brake pads.

This aligns with the observation that the higher iron content and reduced silicon in Sample II enhance its durability by reducing abrasive wear, thus corroborating the connection between composition and wear resistance.

Furthermore, Wahlström et al. [24] conducted a field test study on airborne wear particles from disc brakes, emphasizing the importance of materials that minimize particle emissions under high load conditions. This finding complements the existing text's mention of Sample II's lower mass loss and wear rate, particularly under such conditions, suggesting that the engineered composition not only improves durability but also reduces environmental impact, making it a more sustainable option in automotive applications.

4.3 Observation of wear tracks

After conducting tests, observations were made using a scanning electron microscope (SEM) on the wear facets of the pins. The results are presented in Figure 6, where the specific wear mechanisms observed include abrasion (Figure 6b) with presence of debris (Figure 6a). These show the presence of abrasion striations on the friction surface of the pin. Indeed, the severities of the disc cause a material removal from the pin. Some of which escapes as debris, while the remaining part adheres to the disc's sliding track. The change in color of the wear tracks on the disc, which has turned black, suggests an adhesion wear.

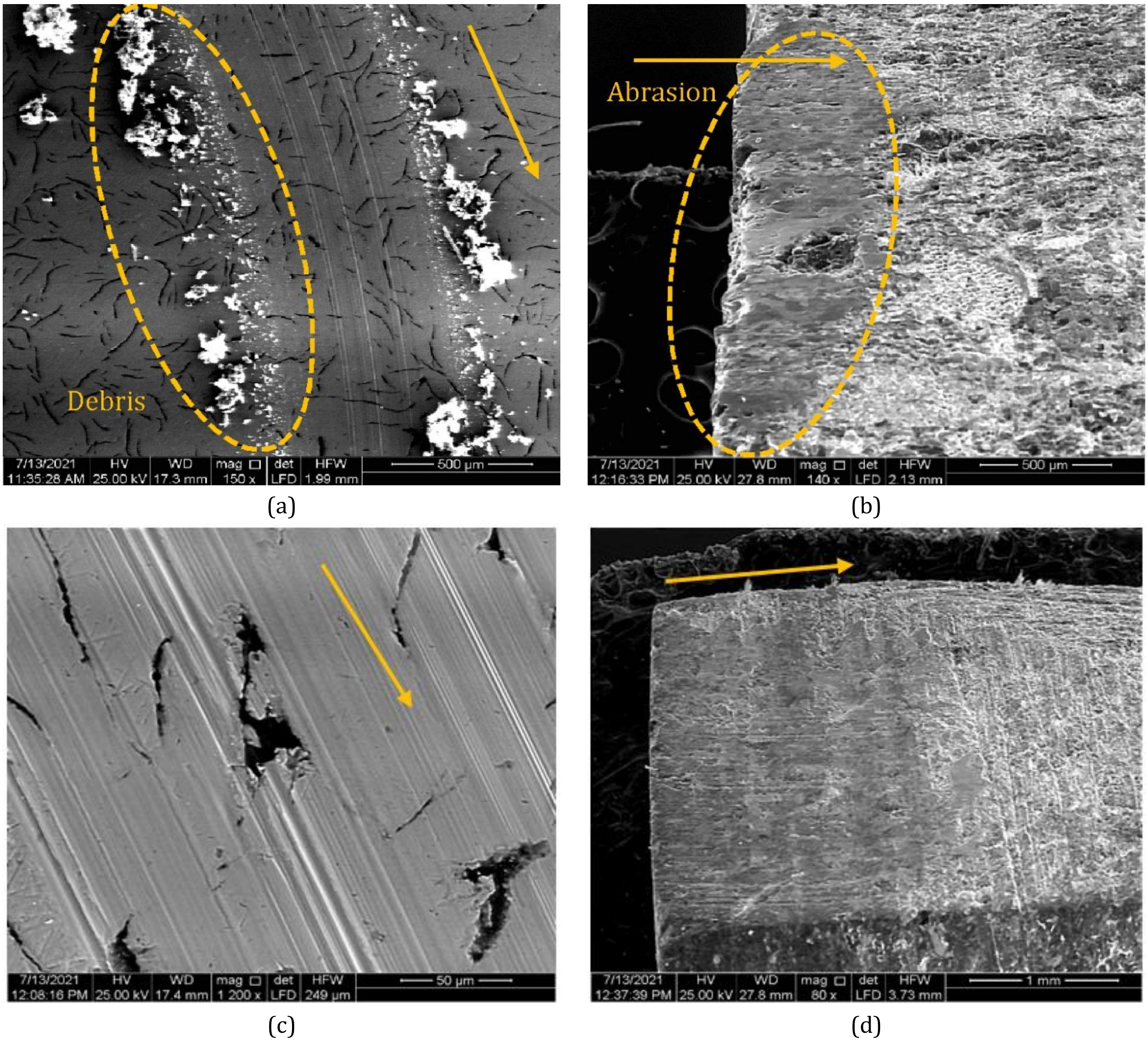


Fig. 6. SEM images, X-ray analysis of the worn face of the pin, disc when sliding at 50 mm/s, normal load of 10 N, (a) wear track on the disc (sample I), (b) wear track on the pin (sample I), (c) observed track on the disc (sample II), (d) SEM pin image (sample II).

Scanning electron microscopy and quantitative analysis were conducted on samples subjected to applied loads of 5 N and 10 N at constant speeds of 35 mm/s and 50 mm/s for 30 minutes. The findings include:

- Micrographs depicted in figure 6 (a) and (b) reveal the removal of surface material and the formation of variously deep furrows aligned with the direction of movement on the rubbing surfaces. The presence of wear debris on the surface contributes to this phenomenon.
- X-ray microanalysis of some debris identified multiple elements, notably a significant peak of iron, alongside smaller peaks of chromium and oxygen, with trace amounts of other elements.

These findings suggest the formation of iron oxides and chromium oxides, likely resulting from material transfer between the interacting surfaces.

5. CONCLUSIONS

The investigation of friction and wear characteristics in brake linings leads to the following main conclusions:

- Sample I and Sample II exhibit Shore hardness values of 32 HS and 36 HS, respectively, along with 2.6 g/cm^3 densities and 2.19 g/cm^3. These lightweight properties indicate that both samples are well suited for automotive applications.
- Tribometer tests show that the coefficient of friction of sample I is approximately 0.35, which is in line with international standards.The coefficient of friction of sample II varies between 0.2 and 0.4, indicating a low wear rate. Laboratory testing at lower loads provides reliable friction and wear data, reducing the need for more costly field testing.
- The wear rates for Sample II were determined to be 4.57 × 10^{-8} cm^3/N·m, indicating minimal thickness loss and superior wear performance compared to Sample I which has a wear rate of 11.53×10^{-8}cm^3/N·m. The optimized composition of Sample II represents a promising alternative for brake linings applications.
- X-ray fluorescence (XRF) and scanning electron microscopy (SEM) analysis confirmed that both brake linings contain at least 49% carbon. The formulation of Sample II, enhanced with $CaCO_3$, Al_2O_3, and SiO_2, offers superior wear resistance and durability compared to conventional options.
- Future studies should focus on optimizing the formulation of Sample II to improve stability and wear resistance, especially concerning high-temperature resistance and incorporation of environmentally friendly filler.

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