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Tribological Performance of Polymer Composite Modified with Calcined Eggshell Particles Post High-Temperature Exposure

Sunardi Sunardi [1], Dody Ariawan [1*], Eko Surojo [1], Aditya Rio Prabowo [1*], Tohid Ghanbari-Ghazijahani [2, 3], Cahyo Hadi Wibowo [1], Hammar Ilham Akbar [4]

[1] *Department of Mechanical Engineering, Universitas Sebelas Maret, Surakarta 57126, Indonesia.*

[2] *Department of Civil Engineering, School of Engineering, Macquarie University, New South Wales 2113, Australia.*

[3] *Alexander von Humboldt Research Fellow (Humboldtian), Germany.*

[4] *Department of Mechanical Engineering, Vocational School, Universitas Sebelas Maret, Surakarta 57126, Indonesia.*

Abstract

During operation, brake lining material rubs against the disc to generate heat. This heat could decrease the brake lining performance, such as the friction coefficient, specific wear rate, and interface temperature of the rubbing surfaces. The resulting wear debris is environmentally harmful and poses risks to human health. Therefore, this study aimed to replace the harmful material using eggshell particles as a filler in brake lining composite and enhance tribological properties. The brake lining samples were manufactured through three stages: cold compaction, hot compaction, and post-curing. The next step is the samples were subjected to a one-hour high-temperature exposure at 200°C, 300°C, 400°C, and 500°C. The results showed that the high-temperature exposure significantly affected the specific wear rate, friction coefficient, and interface temperature between the brake lining and disc. An interesting finding was that adding calcined eggshell particles in composite could improve the tribological properties up to 400°C. However, the best material's performance resulted when the samples got an exposure temperature of 200°C.

Keywords:

Friction Material;

Calcined Eggshell;

High-Heat Exposure;

Tribological Properties.

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1- Introduction

Kinetic energy is often converted into heat due to the friction between the brake lining and the disc when the braking system is operational. This generated heat can impact the wear characteristics of brake lining material. The extent of frictional heat depends on factors such as contact load, friction speed, friction coefficient, and composite thermal conductivity [1]. In this context, sliding speed is the most significant factor [2], showing that the material's response to heat exposure is a crucial consideration in brake lining selection. Brake linings need to effectively absorb heat and withstand high temperatures to control the friction coefficient and wear rate.

Exposure to the temperature of 60 °C can reduce the hardness of UHMWPE, leading to an increase in both the coefficient of friction and wear rate [3]. Prolonged exposure at 80 °C can weaken the bond intensity, thermal stability, shear strength, epoxy adhesive strength, and connection strength between composite and aluminum [4], thereby breaking the polymer chains in UHMWPE [5]. With temperature increasing to 100 °C, there is a significant decrease in compressive strength, tensile strength, and stiffness [6, 7]. Phenolic resin composites reinforced with bamboo undergo degradation at temperatures ranging from 160 to 250 °C, resulting in a reduction in compressive strength [8].

* **CONTACT**: dodyariawan@staff.uns.ac.id; aditya@ft.uns.ac.id

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In a study conducted by Bellini et al. [9], it was found that the friction coefficient tends to increase with rising temperature, but then decreases. The friction coefficient was observed to increase from 0.4 to 0.6 within the temperature range of 100-180°C but then decreased to 0.2 at 350°C. Wear behavior is a crucial factor in selecting brake lining materials, as it determines the type and mechanism of wear on the brake lining. Environmental concerns have led to increased use of natural materials as brake lining. In one study, Gehlen et al. [10] utilized rice husk ash as an eco-friendly friction material. Yavuz et al. [11] showed that brake pad performance can be enhanced with the addition of Cortaderia Selloana fibers. These fibers are environmentally friendly and have excellent braking performance.

Studies are dedicated to enhancing the performance of composites under high-temperature conditions [8, 12–14]. The use of a hybrid matrix comprising polyetheretherketone (PEEK) and polybenzimidazole (PBI) has shown a positive impact on its tribological properties [15]. The incorporation of calcium silicate ($CaSiO_3$) and boron oxide (B_2O_3) influences the material properties and dimensional stability of silicone rubber composites when subjected to elevated temperatures. Linear shrinkage in composites increases with the rising content of boron oxide, reaching its peak at 800°C [16].

Solid lubricants are often added to composite brake linings to enhance their tribological performance. One widely used form of solid lubricant is MoS_2. The addition of MoS_2 leads to a gradual reduction in the friction coefficient and wear behavior. According to Zhen et al. [17], the use of MoS_2 results in improved tribology performance at temperatures ranging from 25°C to 250 °C. At temperatures of 200 °C and 250 °C, MoS_2 enhances the lubrication properties and wear resistance of the composite.

Recently, researchers have shown interest in using eggshells as fillers in polymer composites and metal composites. Eggshell, being a light and high-strength porous ceramic material, has the potential to be used for producing various economically valuable products. This can help to reduce the waste generated by eggshells, which otherwise contributes to pollution [18]. Vieira et al. [19] stated that the use of eggshell particles as reinforcement can enhance Young's modulus and impact resistance, making it an ideal material for manufacturing vehicle parts. The addition of 2 wt% eggshell nanoparticles significantly increases hardness, erosion resistance, as well as tensile and flexural strength [20]. The combination of eggshells and banana peels can be an effective alternative to commercial brake linings [21]. Incorporating carbonated eggshell particles into a composite improves the mechanical and tribological properties of the particulate composite [22, 23]. The carbonated eggshell contributes to improved hardness, compressive [24], tensile modulus, tensile strength, flexural modulus, and flexural strength [25].

Previous studies have not explored the use of calcined eggshells as green fillers in brake lining composite materials after exposure to high temperatures. This study aims to investigate the impact of calcined eggshell particles on tribological properties and temperature at the brake lining/disc interface. In this research, eggshell particles were subjected to calcination treatment at 900 °C for 2 hours, based on parameters from previous studies [23]. Subsequently, the brake lining samples were exposed to temperatures ranging from 200 °C to 500 °C for one hour. The use of eggshell particles as brake lining fillers also serves as an initiative to mitigate the negative environmental effects of eggshell waste.

2- Material and Methods

2-1- Preparation of Eggshell Particles

Chicken eggshells were gathered from household waste and selected as composite filler for their high strength and stiffness, easy degradability, environmental friendliness, and ability to withstand high temperatures. To prepare the eggshell, cleaning and boiling processes were carried out in water for 1 hour to remove attached membrane proteins. Subsequently, the shells were sun-dried for several days, ground, and filtered using a 200-mesh sieve.

2-2- Preparation of Bamboo

Bamboo fibers were obtained through water retting, and bamboo particles through grinding. These particles were then separated into short fibers and parenchyma based on density. Furthermore, bamboo was soaked in a 6% NaOH solution for 3 hours [26], with a ratio of 1:13 between bamboo fibers or particles and the NaOH solution [27]. After the alkali treatment, the bamboo was washed with distilled water till it reached a pH of 7.

The subsequent step comprised soaking the bamboo in a 1% vinyltrimethoxysilane solution for 1 hour [28]. This solution was created by mixing ethyl alcohol and distilled water in proportions of 80% and 20%, respectively, with a fiber weight and solution volume ratio of 0.04 grams/mL [28]. The bamboo material was cleaned with flowing distilled water till it reached a pH of 7, and was dried under the sun for two days and placed in an oven at 100 °C for 3 hours. To prevent water absorption from the atmosphere, the treated bamboo was stored in plastic bags.

2-3- Preparation of Composite Manufacturing

Brake lining composite consists of various materials with the following volume fractions: 35% phenolic resin, 10% bamboo fiber, 5% zinc, 10% alumina, 10% graphite, 5% bamboo particles, and 25% eggshell particles. All particle-form materials were stirred with a four-blade mixer for 2 minutes. Subsequently, the fiber-shaped material was added and stirred for an additional 2 minutes [29]. The first stage is cold compaction; the composite material was pressed at a pressure of 40 MPa for 5 minutes. The second stage is hot compaction; the green body formed was then subjected to hot compaction for 10 minutes at a pressure of 40 MPa and a temperature of 150 °C. The final step comprised post-curing and heating the sample in an electric furnace for 450 minutes [30]. The heating process includes the following details: from room temperature to 140 °C for 60 minutes, from 140 °C to 180 °C for 360 minutes, and from 180 °C to room temperature for 30 minutes. The detailed manufacturing information for composite preparation can be seen in Figure 1.

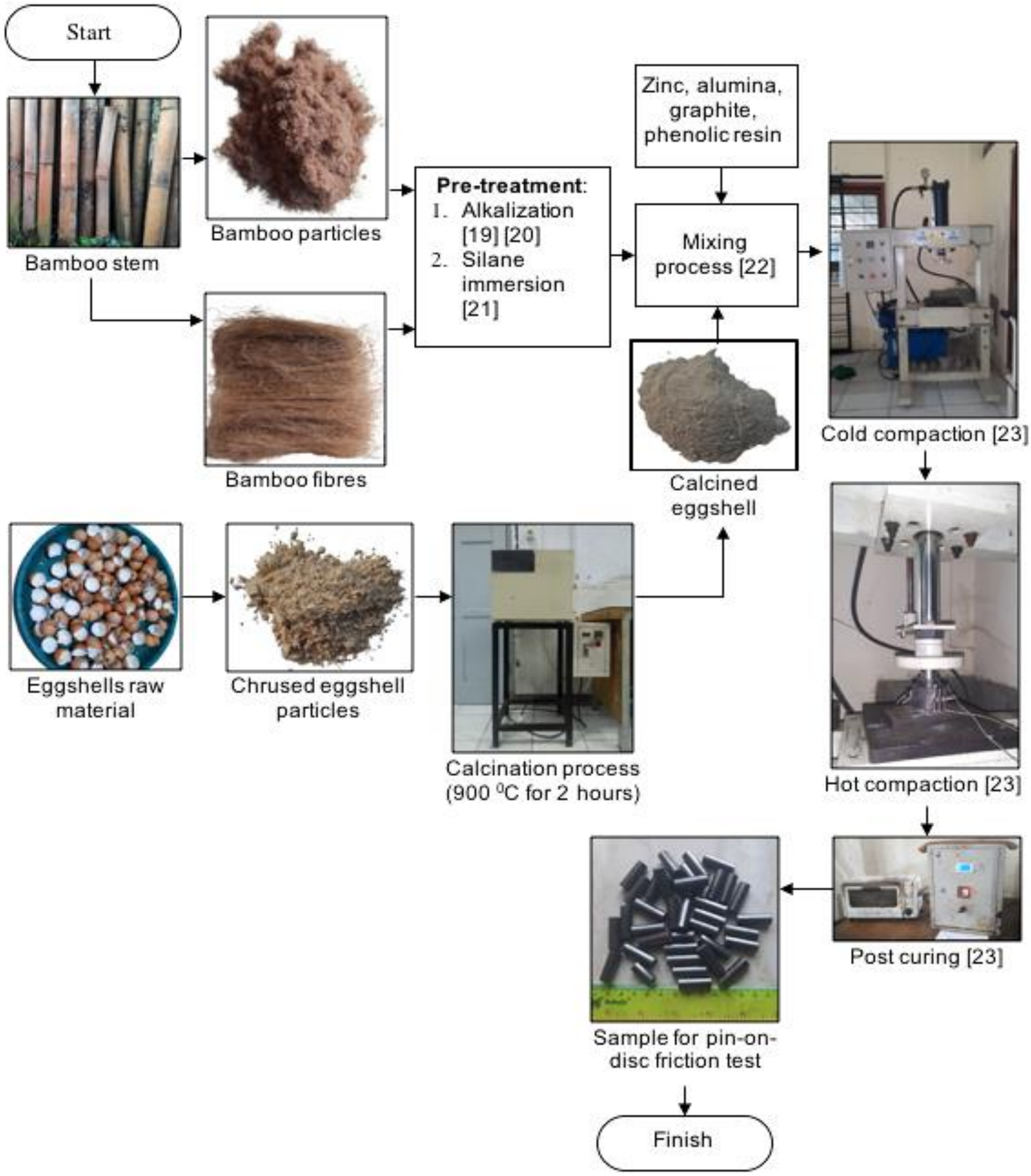


Figure 1. Manufacturing process of samples

2-4- High-Temperature Exposure

The specimens underwent heating at temperatures of 200 °C, 300 °C, 400 °C, and 500 °C for 1 hour in an electric furnace. After exposure to high temperatures, the samples were removed from the furnace and allowed to cool at room temperature. The subsequent step consisted of pin-on-disc testing to determine SWR, friction coefficient, and interface temperature.

2-5- Wear Rate and Coefficient of Friction Testing

The friction coefficient and wear rate were determined through pin-on-disc testing conducted in dry conditions. The disc material, made from DIN X153CrMoV12 steel, boasted a hardness of 54RC and a (surface) roughness ranging from 0.43 to 3.7 µm. The pin sample used in this study had a length of 15 mm and a diameter of 10 mm. The details of pin-on-disc testing can be seen in Figure 2.

Figure 2. Schematic pin-on-disc testing

The parameters for the pin-on-disc testing included a contact pressure of 0.75 MPa, a sliding speed of 12 m/s, and a sliding distance of 6020.76 m per test. After each test, the surface of the disc was wiped down using a gentle fabric to eliminate wear particles or impurities. The calculation of SWR involved dividing the wear volume by the multiplication of the contact load and sliding distance [31], as shown in Equation 1. Meanwhile, the friction coefficient was calculated by dividing the force of friction by the perpendicular force, as expressed in Equation 2:

$$\mu = \frac{f_s}{F_N} \tag{1}$$

$$SWR = \frac{\Delta V}{Ld} \tag{2}$$

where L is the contact load (N), d is the sliding distance (m), and ΔV is the volume of the material worn (mm^3).

2-6- Interface Temperature

Friction between the brake lining and disc surfaces generated heat, which could diminish the brake pad's tribological performance. Interface measurements were conducted after the sample had covered a distance of 5275 meters, using a digital laser infrared thermometer gun to measure the temperature interface between the lining and disc brake.

3- Result and Discussion

3-1- Characteristics of Eggshell

Eggshell particles were obtained from household waste. The appearance of eggshell raw material is shown in Figure 3-a. Eggshells were boiled at 100 °C for 1 hour to remove impurities from the eggshell. The next process is eggshells are finely crushed to a mesh size of 200, as shown in Figure 3-b. Figure 3-c shows the eggshell particles after calcination treatment. Figures 3-d and 3-e showed the phase comparison between eggshell without and with calcination treatment, indicating that the calcined eggshell phase includes a CaO phase. Particle size distribution was analyzed using a particle size analyzer (PSA), as seen in Figure 3-f with an average particle size of 74.74 µm.

CaO is highly reactive to water vapor and will quickly form $Ca(OH)_2$. The $Ca(OH)_2$ bond is ionic and creates a strong bond due to the transfer of electrons from one atom to another. Mohd Pu'ad et al. [32] demonstrated the chemical reaction between CaO and H_2O.

$$CaCO_{3(s)} \rightarrow CaO_{(s)} + CO_{2(g)} \tag{3}$$

$$CaO_{(s)} + H_2O_{(l)} \rightarrow Ca(OH)_{2(s)} \tag{4}$$

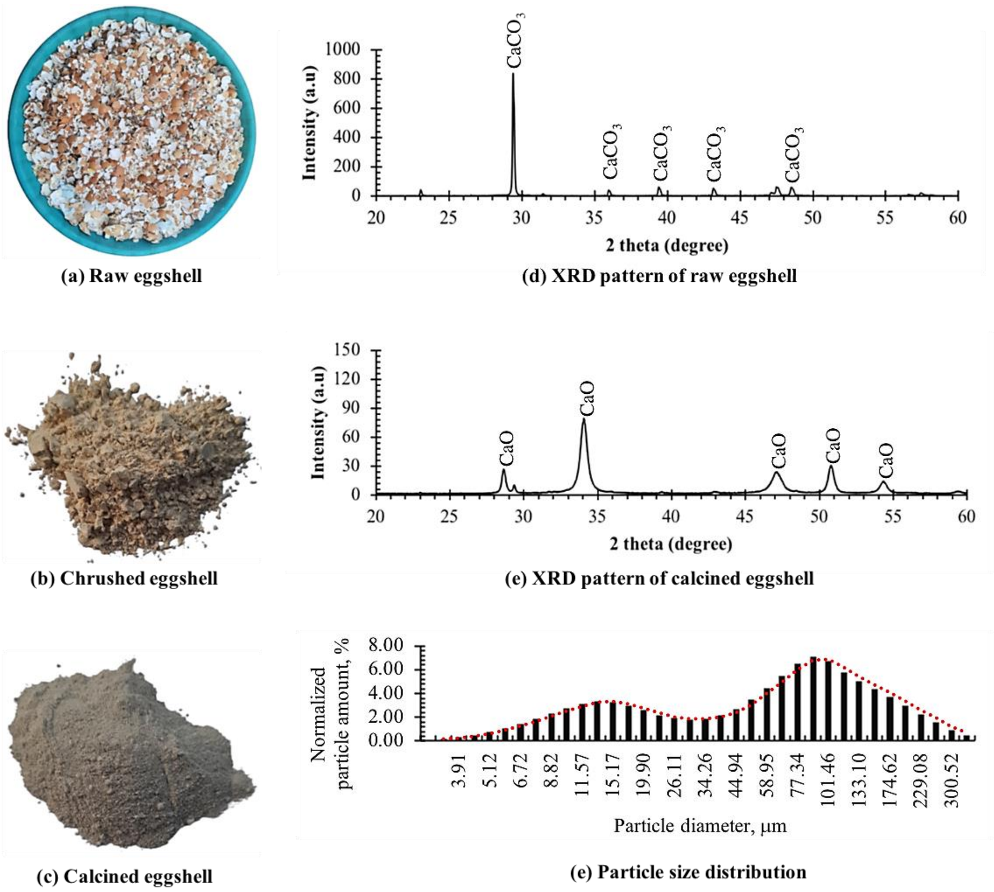


Figure 3. Characteristics of eggshell (a) raw eggshell, (b) crushed eggshell, (c) calcined eggshell, (d) XRD of raw eggshell, (e) XRD of calcined eggshell at temperature 900 °C for 2 hours, and (f) distribution of eggshell particles

3-2- FTIR Analysis

High-temperature exposure significantly impacted the tribological performance of organic brake linings. Heat exposure was assessed based on the interface contact between the disc and brake lining, which could generate heat. This interface heat might reduce brake lining performance and degrade tribological properties. Figure 4 shows that exposure to high temperatures could break atomic bonding in the matrix. Weakening this atomic bonding strength that binds other particles led to a reduction in the composite's performance. Exposure temperature of 200-400 °C resulted in stronger bonds between the O-H atoms, at 3641 cm^{-1} in phenolic resin. However, this bond was broken after exposure to a temperature of 500 °C. The peak at 2941 cm^{-1} originated from the stretching vibration of the C-H bonds in the aliphatic arrangement [33]. This condition resulted in a higher coefficient of friction and SWR of the composite.

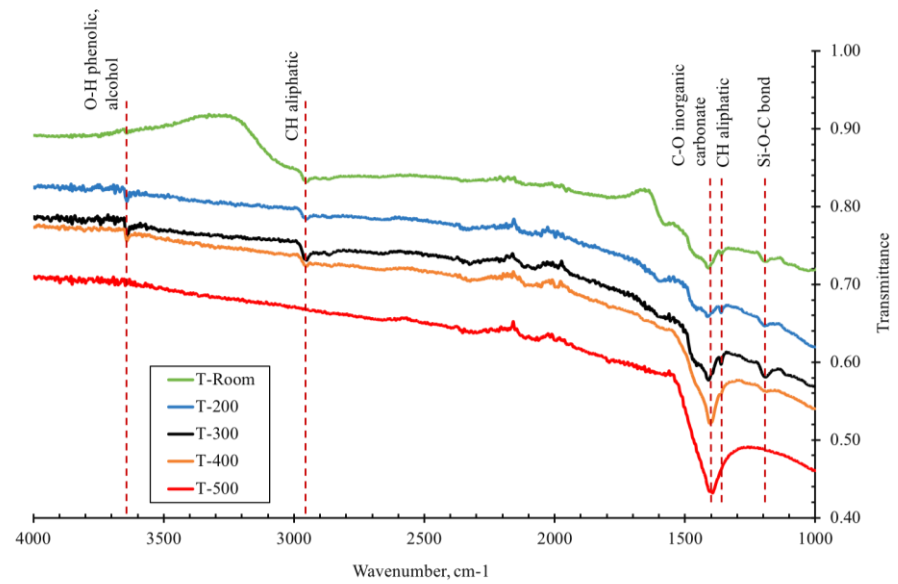


Figure 4. FTIR data of friction material composite at high-temperature exposure

FTIR data showed that brake lining composites maintained relatively good tribological performance when exposed to temperatures up to 400°C. At the same temperature, the disappearance of peaks at 1181cm^{-1} and 1351cm^{-1}, representing Si-O and O-H bonds, respectively, showed the occurrence of heat-induced changes. However, when exposed to 500 °C, significant degradation of tribological properties was observed, marked by a drastic increase in the wear rate.

The 1100-1200 cm^{-1} band indicates the Si-O-C bond, which is the product of the alkali-silane treatment of bamboo fibers and particles [34]. On exposure to temperatures of 400 °C and 500 °C, this band disappeared, indicating the breaking of the Si-O-C bond. This condition affects the decline in the mechanical and tribological performance of the composite. The 2940 cm^{-1} band corresponds to the C-H bond vibration in methyl, including the characteristic peak of cellulose [35]. The FTIR spectrum shows a peak at 3800-3300 cm^{-1} related to O-H stretching [27]. The stretching at the peak of 3600 cm^{-1} is caused by aliphatic OH groups of phenolic resin compounds [36]. The peak at band 3639 cm^{-1} as a result of OH in $Ca(OH)_2$ formed during the water absorption by CaO [37]. On exposure to a temperature of 500°C, this band disappeared, indicating the breakdown of the hydroxyl (OH) group originating from phenolics and alcohol. The impact is the degradation of the tribological performance of the composite.

3-3- Temperature of Interface Contact Between Friction Material and Disc

In Figure 5, it was evident that the interface temperature of the friction surface rose with the increasing exposure temperature of the composite. The magnitude of the friction coefficient significantly influenced changes in the temperature of the friction surface interface. The surface interface temperature experienced a significant increase at an exposure temperature of 400 °C, attributed to the loss of phenolic resin, bamboo fibers, and bamboo particles during high-temperature exposure. The presence of phenolic resin and bamboo in the composite contributed to retaining the frictional heat of brake linings and discs. However, at a temperature of 500 °C, metal elements such as zinc, alumina, and graphite dominated the composite friction surface. Under this condition, the composite showed high thermal conductivity, triggering a rapid increase in frictional heat. From room temperature up to 400 °C, friction heat was retained by phenolic resin, bamboo, and eggshell.

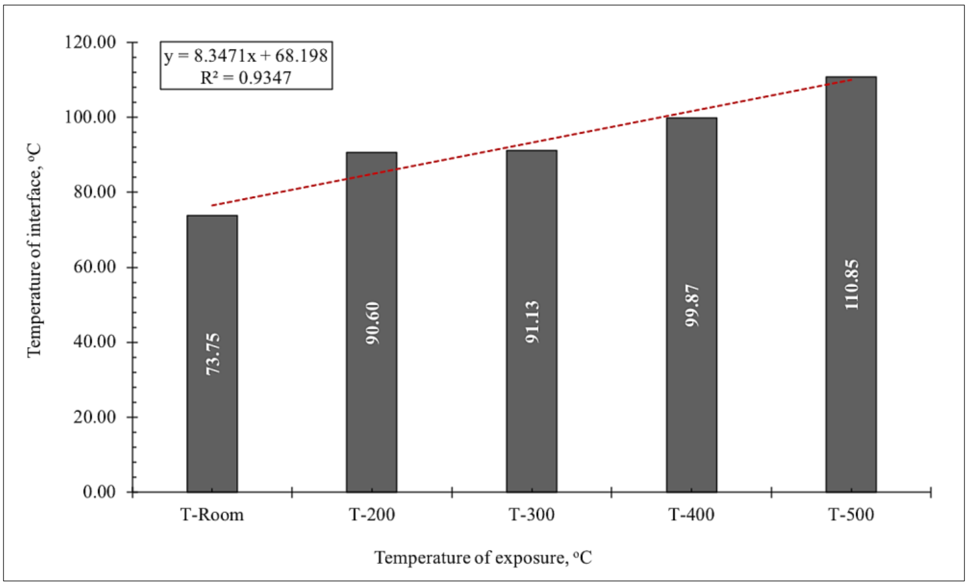


Figure 5. Interface temperature of brake linings at various high temperatures

The temperature at the interface of the brake lining and disc depends on the materials used. Research indicates that materials with high tensile strength and hardness result in higher temperatures during braking [38]. This is due to the thermal conductivity of the brake disc and lining. Heat accumulation at the friction surface raises the disc temperature rapidly. In composite samples exposed to a temperature of 500 °C, organic materials decompose earlier, leaving the brake lining surface dominated by alumina, zinc, and graphite. This leads to an increase in the interface temperature of the brake lining and disc surfaces.

3-4- Coefficient of Friction

The friction coefficient of the sample without heat exposure was 0.32. After exposure to a temperature of 400 °C, the organic brake lining gradually increased in friction coefficient, as shown in Figure 6. These results were in line with studies conducted by other studies, asserting that higher friction coefficient corresponded to higher temperature [1, 12, 14]. Heat exposure at a temperature of 500 °C led to a drastic 87.5% increase in the friction coefficient. In the case of polyimide and thermoplastic polyimide composites reinforced with mesoporous silica, the friction coefficient continued to rise until the temperature of 300 °C [39]. These results were contrary to those reported by Meresse et al. [40] and Pan et al. [41], where the friction coefficient gradually decreased with increasing exposure temperature.

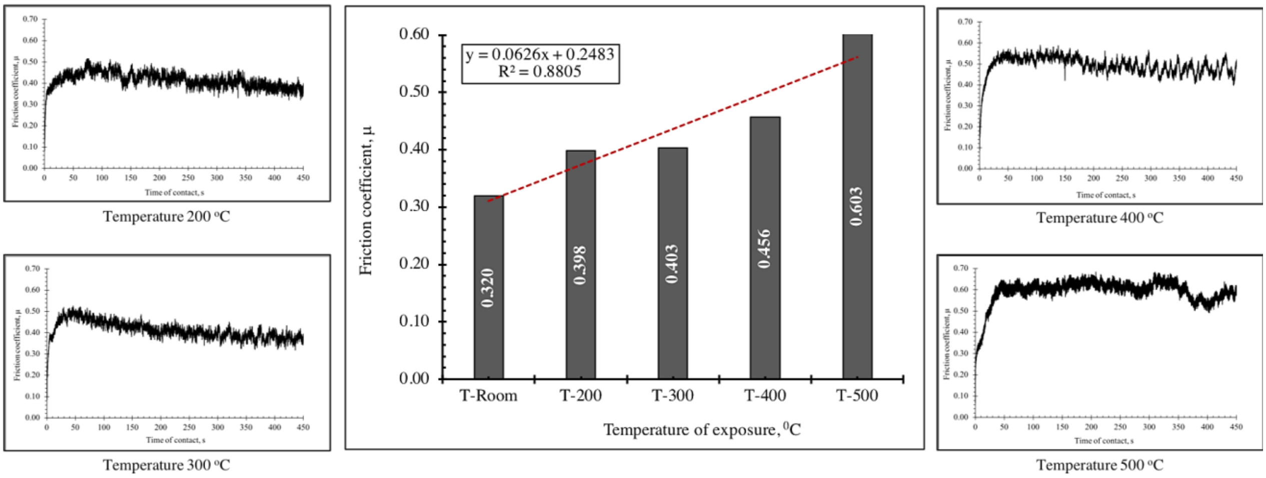


Figure 6. Brake lining friction coefficient at high-temperature exposure

The incorporation of graphite in polyimide composites served as a solid lubricant, enhancing their resistance to high temperatures. Although graphite began to degrade at a temperature of 350 °C [42], its addition resulted in a higher percentage of water absorption [43]. Beyond a 20% graphite content, the friction coefficient remained relatively stable, even though it decreased [44]. In this study, the graphite content was 10%. The friction coefficient rose with an increase in the sample's exposure temperature. A critical phenomenon occurred with heat exposure at 500 °C, where the friction coefficient significantly increased. This increase in the coefficient of friction is caused by the degradation of graphite at a temperature of 500 °C, which causes it to lose its function as a solid lubricant.

Generally, the friction coefficient is directly correlated with the SWR of the brake lining sample. A higher coefficient of friction corresponded to an increase in SWR of the composite. This rise in the friction coefficient was associated with a decline in the tribological performance of brake lining.

3-5- Specific Wear Rate

SWR of the organic brake lining gradually increased up to the temperature of 400 °C as indicated in Figure 7. However, at 500 °C, the composite showed a significantly higher SWR, reaching 65.23% from 4.43×10^{-6} mm³/Nm. Both SWR and friction coefficient increased with the rising exposure temperature [11, 32]. At high temperatures, brake linings degraded tribological properties through substantial wear erosion. The carbon element became susceptible to oxidation at temperatures exceeding 400 °C, resulting in carbon decay due to air exposure [45]. The interfacial adhesion properties of the composite weaken due to an increase in temperature, causing a significant increase in SWR when exposed to heat at 500 °C [46].

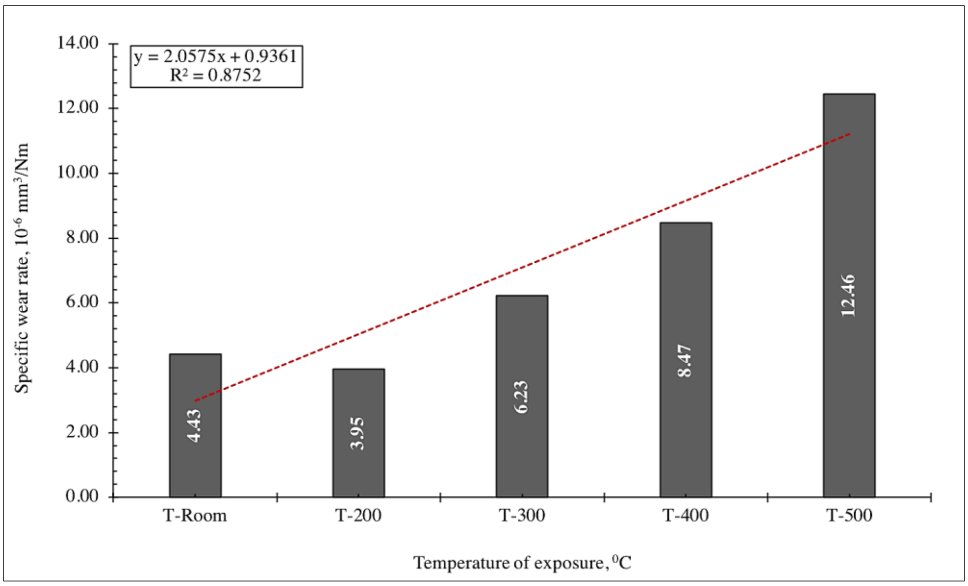


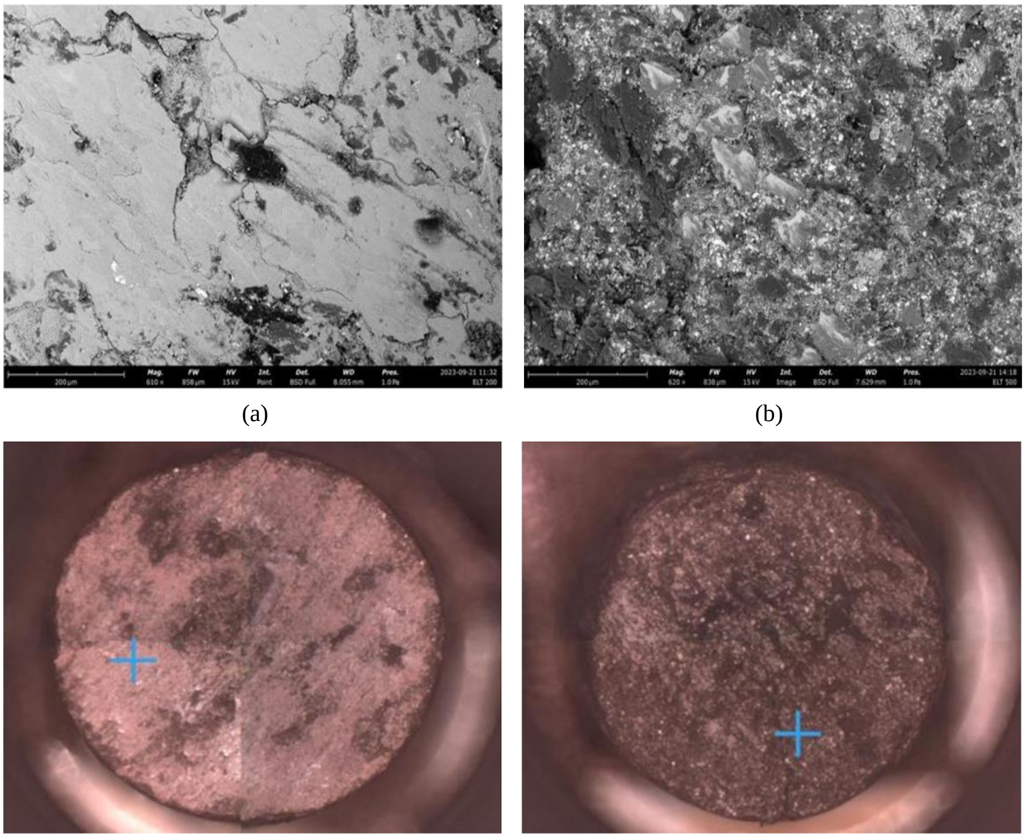
Figure 7. The SWR of brake lining at high-temperature exposure

At a temperature 200 °C, organic brake linings achieved the lowest SWR at 3.95 x 10^{-6} mm³/Nm. This represented a 10.84% reduction compared to composites without heat exposure. According to Dai et al. [47], an increase in surface energy and contact angle, due to the post-curing of the composite from 180 °C to 200 °C, caused a decrease in SWR value. This increase in surface energy also affects the adhesion of the matrix and fiber interface, resulting in higher wear resistance [48]. These research findings were verified by Dwiwedi et al. [18], who demonstrated that incorporating eggshell particles could enhance the wear resistance of the matrix material. Tang et al. [49] also concluded that the addition of expanded graphite in the composite resulted in a reduction in the wear rate of brake lining composites. Karacor & Ozcanli [50] demonstrated that elevating the post-curing temperature from 50 °C to 90 °C led to a 1.06 times increase in the tensile strength of jute fiber composites, while the hardness rose by up to 1.36 times. Campana et al. [51] demonstrated that post-curing at a temperature of 80°C for 24 hours, followed by heating at a temperature of 120 °C for 2 hours, resulted in optimal mechanical properties in flax fiber-reinforced epoxy composites. However, post-curing at a temperature of 150 °C led to a reduction in the mechanical properties of the composite. In this study, it was found that the inclusion of calcined eggshell particles affected the mechanical and tribological properties achieved by heating at an exposure temperature of 200 °C. This process is equivalent to slow heating from room temperature to 180 °C, followed by rapid heating from 180 °C to 200 °C. Calcined eggshell particles have higher hardness and act as hard filler material, thereby increasing wear resistance. Eggshell particles can fill gaps in the matrix, resulting in a dense and compact structure.

The increase in SWR commenced at a temperature of 300 °C. It should be found that the wear mode observed in this study was abrasive wear. Meresse et al. [40] showed a similar phenomenon, indicating severe wear occurrences at temperatures surpassing 300 °C. The critical temperature (TC) for pure phenolic resin was 120 °C, while steel fiber-reinforced matrices approached 200 °C [40]. In previous studies, it was found that adding eggshell particles, whether calcined or not, can reduce specific wear rates [23]. Therefore, in this study, the increase in the specific wear rate is mainly caused by the exposure temperature of the composite samples.

3-6- Worn Surface Morphology

Figure 8 shows that the wear mode consisted of adhesive and abrasive wear. At 200 °C, the flat surface of the sample showed the completion of the curing process [2]. The height of the contact plateau increased with the elevation of exposure temperature. This condition caused the wear rate to escalate at temperatures of 300 °C, 400 °C, and 500 °C. The increase in the wear rate resulted from the weakening of atomic bonding in phenolic resin. According to FTIR data in Figure 4, at a temperature of 500 °C, the composite's surface went through abrasive wear mode, characterized by groove formation. This condition induced cracks and triggered the formation of catastrophic ruptures [52].



(a) (b)

(c) (d)

Figure 8. Surface wear track at temperature of exposure (a, c) 200 °C, and (b, d) 500 °C

Solid lubricants and exposure temperature are significant factors in determining wear mechanisms. Composites that are not exposed to heat typically exhibit a relatively smooth wear surface. At low temperatures, abrasive wear dominates the wear mechanism. According to Zhen et al. [17], as the temperature increases from 150 to 250 °C, a transformation in the wear mechanism occurs, resulting in delamination and the formation of grooves, ultimately leading to fatigue wear and severe abrasion.

Based on the performed characterization, the results indicated that eggshell-based particles have a promising future application in technical engineering. It is recommended to conduct a head-to-head comparison with other potential materials, such as nature-based [53-59], even semi-metallic [60-64], and advanced materials [65-69], and a regulation standard for specific engineering products in the next study to obtain comprehensive milestone research and the required industrial specification.

4- Conclusions

The use of eggshell particles with or without calcination treatment has been shown to enhance the mechanical and tribological properties of brake lining composites, as demonstrated in previous research. High temperatures significantly impact the tribological performance and interface temperature during the braking process. This research yields several important conclusions:

- The specific wear rate, friction coefficient, and brake lining/disc interface temperature increase with higher exposure temperatures of the composite.
- The increase in the coefficient of friction, specific wear rate, and composite interface temperature at temperatures of 400 °C and above is caused by the degradation of organic materials. Graphite particles, which act as a solid lubricant, are also degraded due to high temperatures, causing the loss of their lubrication function. As a result, this condition leads to an increase in the coefficient of friction and very high wear rates.
- The addition of calcined eggshell particles has the potential to improve the tribological characteristics of brake linings after being exposed to high temperatures.
- The composite exhibited the least specific wear rate when subjected to a temperature of 200 °C.
- Specifically, at the temperature of 400 °C, the O-H, C-H, and C-N bonds started to weaken, and when exposed to 500 °C, these bonds broke. SWR decreased at an exposure temperature of 200 °C, attributed to improvements in the post-curing process.
- The morphology of the worn surface observed via SEM-EDS also shows that the friction surface is dominated by metal materials, especially alumina and zinc. This condition causes an increase in temperature between the brake lining and the disc.

Calcined eggshell particles can be developed as an environmentally friendly material and are able to absorb carbon dioxide around the brake lining, so in the future it is necessary to study how much the brake lining is capable of absorbing carbon dioxide.

5- Declarations

5-1- Author Contributions

Conceptualization, S.S., D.A., E.S., and A.R.P.; methodology, S.S., D.A., E.S., and H.I.A.; software, A.R.P. and T.G.G.; validation, S.S., C.H.W., and H.I.A.; formal analysis, S.S., C.H.W., and H.I.A.; investigation, S.S., C.H.W., and H.I.A.; resources, D.A., E.S., and A.R.P.; data curation, S.S., A.R.P., and T.G.G.; writing—original draft preparation, S.S., D.A., E.S., and A.R.P.; writing—review and editing, S.S., A.R.P., and T.G.G.; visualization, S.S., A.R.P., and T.G.G.; supervision, D.A., E.S., and A.R.P.; project administration, S.S. and D.A.; funding acquisition, S.S., D.A., E.S., and A.R.P. All authors have read and agreed to the published version of the manuscript.

5-2- Data Availability Statement

The authors confirm that the data supporting the findings of this study are available within the article.

5-3- Funding

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5-4- Institutional Review Board Statement

Not applicable.

5-5- Informed Consent Statement

Not applicable.

5-6- Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/or falsification, double publication and/or submission, and redundancies have been completely observed by the authors.

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