

Tribological Characteristics of Periwinkle Shell/Raffia Palm Seed Reinforced Epoxy Hybrid Composites

Oryina Mbaadega Injor¹, Moipone Linda Teffo¹, Emmanuel Rotimi Sadiku¹, Victor Ugbetan Agbogo¹

¹*Department of Chemical, Metallurgical and Materials Engineering, Tshwane University of Technology, Pretoria, 0001, South Africa.*

Abstract

Environmentally friendly and sustainable brake pad materials have attracted increasing attention due to the health and environmental concerns associated with asbestos-based friction materials. This study developed and characterized hybrid epoxy composites reinforced with periwinkle shell (PWS) and raffia palm seed (RPS) particles as potential eco-friendly brake pad materials. Unlike previous investigations that considered PWS or RPS individually, the present work examined their combined use in a hybrid system to evaluate the synergistic effects of agro-waste reinforcements on brake pad performance. Four composite formulations (Samples A–D) containing 4.41–17.71 wt.% PWS and 3.41–13.77 wt.% RPS were produced using epoxy resin as the matrix through molding and curing under a pressure of 18.25 kN/m². The composites were characterized by evaluating density, water absorption, hardness, wear depth, wear rate, and coefficient of friction. Results showed that increasing the reinforcement content significantly enhanced composite performance. Density increased from 2.60 to 3.28 g/cm³, while water absorption decreased from 0.1214% to 0.0388%. Hardness improved from 33.40 HV to 72 HV. Tribological evaluation revealed a reduction in wear depth from 257.859 µm to 140.297 µm and a decrease in wear rate from 0.00000893 to 0.00000819 mm³/(N·m). The coefficient of friction decreased from 0.70–0.75 to 0.57–0.60, indicating improved friction stability. Sample D exhibited the best overall performance. The findings demonstrate that hybrid PWS/RPS epoxy composites are promising low-cost, environmentally friendly brake pad materials that enhance performance while promoting agricultural waste valorization and sustainable manufacturing.

Keywords: Tribology, Periwinkle shell, Raffia palm seed, Hybrid composite, Asbestos-free brake pad, Natural reinforcement.

1. Introduction

The continuous growth in automobile usage has heightened the demand for efficient, eco-friendly, and cost-effective braking systems. Brake pads, as a critical part of vehicle safety mechanisms, require materials that possess high thermal stability, wear resistance, and mechanical strength [1]. Traditionally, asbestos-based materials have been widely used for brake pads because of their desirable properties. Conversely, the toxic

disposition of asbestos has demanded the development of alternative materials that are environmentally benign and sustainable [2], [3], [4], [5], [6].

In this context, natural fiber-reinforced composites have received significant attention as viable replacements. Agro-waste materials such as palm kernel, coconut, and periwinkle shells have emerged as promising reinforcements because of their availability, low cost, biodegradability, and relatively better mechanical

Corresponding author: Oryina Mbaadega Injor (InjorOM@tut.ac.za, injorman@yahoo.com)

Received: 21 March 2026; Revised: 15 June 2026; Accepted: 21 July 2026; Published: 13 October 2026

© 2026 The Author(s). This work is licensed under a Creative Commons Attribution 4.0 International License

properties [7], [8]. Periwinkle shells, which are predominantly composed of calcium carbonate, offer high hardness and thermal resistance, making them appropriate for friction [9]. Raffia palm seeds, rich in lignocellulosic content, enhance the mechanical integrity and reduce the density of composites, thus improving energy efficiency and sustainability [10].

The use of epoxy resin as a matrix in brake pad development is well-documented due to its excellent adhesion, thermal resistance, and dimensional stability [11]. When reinforced with hard agro-waste particulates, epoxy composites can be engineered to meet the demanding conditions encountered in braking systems. Combining two or more reinforcements yields improved properties by leveraging the strengths of individual constituents [12].

The growing need for environmentally sustainable and cost-effective materials has fueled studies on the role of natural fibers and agro-waste particulates in composite development, particularly for frictional applications such as brake pads. Conventional brake pads typically comprise asbestos or synthetic fibers combined with phenolic resins; however, the hazardous nature of asbestos and increasing material costs have prompted the search for alternatives [13], [14].

Natural fillers, such as periwinkle shell (PWS) and raffia palm seed (RPS), have gained attention because of their biodegradability, low density, and better mechanical properties. Periwinkle shells, being calcium-rich marine biowastes, have shown potential as reinforcements due to their inherent hardness, abrasive characteristics, and thermal stability [1]. Yawas et al. [15] examined the mechanical properties of asbestos-free brake pads with periwinkle shell dust as reinforcement and reported acceptable wear resistance and hardness suitable for frictional applications. Similarly, Aku et al. [1] confirmed that periwinkle shell-reinforced composites exhibit consistent frictional performance and dimensional stability under high operating temperatures.

Raffia palm seed particles, on the other hand, are less explored but have demonstrated promise in mechanical property enhancement when used in polymer composites. Their fibrous structure contributes to better interfacial bonding with matrices, such as epoxy resin, resulting in improved impact resistance and reduced wear [16].

According to Ipilakyaa et al. [17], raffia palm seed particles provide a reinforcing effect that enhances both the tensile strength and thermal properties of epoxy-based composites. This is similar to the findings of Oguocha et al. [18].

Epoxy resins are commonly used as matrices during composite production due to their excellent bonding characteristics, chemical resistance, and thermal stability [19]. The integration of natural fillers into epoxy matrices reduces overall composite weight while maintaining desirable tribological characteristics. Hybrid composites, in particular, are gaining attention for their ability to combine the benefits of multiple reinforcements, offering superior wear resistance, thermal stability, and mechanical performance compared to single-filler systems [20], [21].

Most previous studies on eco-friendly brake pad materials have focused on the use of single agro-waste reinforcements such as periwinkle shell and raffia palm seed individually [15], [17]. While these materials established varying degrees of improved wear resistance, hardness, and frictional performance, many suffered from limitations, such as excessive wear or poor mechanical strength, when used alone. The potential synergistic interaction between hard ceramic-rich PWS particles and the relatively lightweight, bio-based RPS particles within a single composite matrix has not been examined. Rather than relying on a single waste material, the hybrid approach seeks to exploit the complementary characteristics of both reinforcements to achieve a balanced combination of physical, mechanical, and tribological properties. This provides new knowledge on the synergistic performance of agro-waste reinforcements for sustainable brake pad applications. Other researchers have examined the synergistic effects of hybrid reinforcements in brake pad composites. For example, Popoola et al. [22] explored the development of hybrid composites using agro-waste materials, such as palm kernel shell, coconut shell, seashell, and cow bone, and observed improved tribological performance and reduced fade characteristics. Studies investigating the synergistic effect of combining PWS and RPS as reinforcements in brake pad production are limited in the existing literature. Therefore, the hybridization of PWS and RPS in an epoxy matrix is anticipated to leverage the hardness of PWS and the fibrous resilience of RPS, resulting in a composite

suitable for brake pad applications with enhanced performance under high-stress and high-temperature conditions.

In summary, while individual studies have demonstrated the utility of PWS and RPS in polymer composites, limited research has explored their combined effect in a hybrid system. Thus, this study aimed to investigate the physical, mechanical, and tribological properties of epoxy-based composites reinforced with periwinkle shell and raffia palm seed particles for brake pad applications. Specifically, the study evaluated the effects of reinforcement composition on density, hardness, and wear behavior. It also identified the optimum composite formulation and assessed the potential of these agro-waste materials as sustainable alternatives for high-performance brake pad materials.

2. Materials and Methodology

The major materials used in the study were periwinkle shell (Figure 1), raffia palm seed (Figure 2), epoxy resin (LY556), hardener (HY951), and aluminum chips. The epoxy matrix was prepared using LY556 epoxy resin and HY951 hardener in accordance with the manufacturer's recommended mixing ratio of 2:1 by weight (epoxy resin:hardener). The required quantity of HY951 hardener was steadily added to the LY556 resin and mechanically stirred to obtain a homogeneous mixture. No additional catalyst was used in the formulation.



Figure 1. Periwinkle shells.



Figure 2. Raffia palm seeds.

The material was produced through a process involving raw material selection, weighing, mixing, compaction, and bonding. Four formulations were prepared, each containing varying proportions of periwinkle shell powder, raffia palm seed powder, and aluminium chips as reinforcements. Aluminium chips were incorporated to modify the frictional and wear characteristics of the composites. The samples were grouped according to variations in the proportions of periwinkle shell powder, raffia palm seed powder, epoxy resin, hardener, and aluminium chips used in the formulation. Table 1 presents the detailed formulation of the four different material compositions.

Table 1. Composition of Brake Pad Material Formulations.

Materials		Samples (wt.%)			
		A	B	C	D
1	Epoxy Resin	60.12	53.61	47.00	40.28
2	Hardener	30.06	26.86	23.50	20.14
3	Periwinkle shells powder	4.41	8.79	13.30	17.81
4	Raffia palm seed powder	3.41	6.84	10.30	13.87
5	Aluminum chips	2.00	3.90	5.90	7.90

Based on the compositions presented in Table 1, the total reinforcement contents comprising PWS, RPS and aluminum chips were approximately 9.82, 19.53, 29.50, and 39.38 wt.% for Samples A, B, C, and D respectively. These values correspond approximately to the targeted reinforcement levels of 10, 20, 30, and 40 wt.%. The

combined contents of the natural reinforcements (PWS and RPS), excluding aluminium chips, were 7.82, 15.63, 23.60, and 31.48 wt.% for Samples A-D, respectively. The epoxy resin and hardener constituted the matrix system, with their proportions adjusted according to the formulation.

2.1 Preparation of the Samples

Periwinkle shells and raffia palm seeds were collected from discarded shells and cracked seeds, respectively. Contaminants in the collected raw materials were removed using ethanol. Following cleaning, the periwinkle shells and raffia palm seeds were sun-dried for 24 hours to reduce the moisture content. The dried PWS and RPS particles were subsequently ground using a crushing machine and sieved to obtain a particle size fraction between 100 and 200 μm . Particles passing through the 200 μm sieve and retained on the 100 μm sieve were collected for composite production. The particles were stored in airtight polyethylene desiccators at room temperature to reduce moisture reabsorption from the environment before composite fabrication. The periwinkle shell and raffia palm seed particles were used in their untreated form to preserve their natural characteristics, reduce processing costs, and evaluate their inherent reinforcing potential in the epoxy matrix. Four different combinations (samples A, B, C, and D) were prepared with variable proportions of periwinkle shell and raffia palm seed powders (Table 1). Periwinkle shell and raffia palm seed powders served as the reinforcements, epoxy resin served as the matrix, and aluminium chips served as abrasives.

The hand lay-up method was used for the fabrication process. For each sample, the periwinkle shell powder, raffia palm seed powder, and aluminium chips were thoroughly blended until a homogeneous mixture was obtained. Epoxy resin was added to the mixture and thoroughly blended for approximately 10 min to form a paste-like consistency. This was done to instigate the cross-linking reaction of the epoxy matrix while inhibiting extremely fast curing, unnecessary heat generation, shrinkage, and void formation within the composite. The blended mixture was poured into a pre-prepared steel mould measuring 150 mm $\times$ 150 mm $\times$ 5 mm and left to cure for 24 hours at an ambient temperature

of 25°C to ensure adequate cross-linking of the epoxy matrix, resulting in the achievement of adequate hardness, dimensional stability, and mechanical integrity. Post-curing was not performed on the mixture. At this stage, a pressure of 18.25 kN/m² was applied and maintained for 6 hours to ensure complete curing, after which excess material was stripped off from the samples by abrasive machining.

2.1.1. Density measurement

The volumes of the specimens were measured using the liquid displacement technique in accordance with ASTM D792-20. Each specimen was submerged in a 1000- mL graduated cylinder containing distilled water at an ambient temperature. The water volume was recorded before and after sample immersion, and the difference between these readings corresponded to the sample volume. The mass of the samples was determined using a Mettler E200 electronic balance with a precision of ± 0.01 g. The thicknesses of samples A, B, C, and D were measured using a micrometer. Measurements were taken at three locations (both ends and the center of each sample), and the mean value was determined.

2.1.2. Water absorption

The water absorption test was performed in accordance with ASTM D570-98 [23]. Samples with dimensions of 30 mm $\times$ 30 mm $\times$ 5 mm were initially weighed and then immersed in a water bath at room temperature for 24 hours. Weighing was conducted using a Mettler E200 electronic balance. After immersion, the samples were removed, and the surface moisture was wiped off before reweighing. The difference between the initial and final masses represents the quantity of absorbed water. Three samples were tested, and the mean values are reported. The percentage of water absorption (based on dry mass) was calculated using Eq. (1).

$$\% \text{ water absorption} = \frac{\text{Wet mass} - \text{Dry mass}}{\text{Dry mass}} \times 100 \quad (1)$$

2.1.3. Microstructural analysis

Microstructural images of the samples were obtained using a scanning electron microscope (SEM) (model:

Zeiss 540 Ultra) equipped with energy dispersive X-ray spectroscopy (EDS). The analysis was performed in high vacuum mode at an accelerating voltage of 20 kV at Ahmadu Bello University, Zaria, Nigeria. The EDS system was used to perform bulk elemental analysis of the composite. In addition, the mean grain size was estimated using the linear intercept method with the aid of ImageJ software.

2.1.4. X-ray diffraction analysis

A RAYONS X X-ray diffractometer (XRD) was utilized to investigate and identify the various phases present in the samples at the Institute for Nano-Engineering Research (INER), Tshwane University of Technology, Pretoria. The analysis was performed using a D/max-250 X-ray diffractometer with Cu-K α radiation of wavelength 1.54 Å, operating at a tube voltage of 30 kV and a current of 20 mA, at a scanning speed of 1°/min. The resulting crystal structures and corresponding peak intensities were identified using X'Pert HighScore Plus software integrated with the RAYONS X diffractometer.

2.1.5. Hardness test

The microhardness of the samples, each measuring 15 mm $\times$ 15 mm, was determined using the Vickers hardness method in accordance with ASTM E384-22 at INER, Tshwane University of Technology, Pretoria. The composite specimens were ground successively using silicon carbide (SiC) abrasive papers of increasing grit sizes of 240, 400, 600, 800, and 1200 grit to obtain a smooth and flat surface. The specimens were subsequently cleaned with ethanol and dried before testing. An FM-800 microhardness tester was used, applying a load of 50 g with a dwell time of 15 seconds on the samples. Three measurements were conducted for each condition, and the reported value for each sample represents the average of indentations spaced at 0.20 mm.

2.1.6. Wear test

A tribometer (Serial No: 10000392309, Version: 8.1.8) was employed to assess dry sliding wear using a ball-on-disc configuration in accordance with ASTM G-99. The test was conducted at the Surface Engineering

Laboratory, Tshwane University of Technology, Pretoria. Wear experiments were carried out at a temperature of 20 °C and a relative humidity of 50 $\pm$ 5%.

Samples for the wear test were machined to dimensions of 20 mm $\times$ 10 mm and mounted in resin. The contact surfaces of the specimens were ground and polished using successive grades of SiC abrasive paper to achieve a uniform surface finish. The specimens were then cleaned with ethanol and allowed to dry before testing. During testing, the specimens slid against a 6 mm zirconia ball with a hardness of 62 Rockwell C hardness (HRC) and a surface roughness of 0.3 μ m at a rotational speed of 130 rpm. The test was performed under a 5 N load and a sliding speed of 0.817 m/s, covering a sliding distance of 282.09 m along a 120 mm diameter track. The wear rate was determined using the weight-loss method with the aid of a digital microbalance with a precision of ± 0.0001 g, while the coefficient of friction was calculated using Eq. (2).

$$\mu = F_f / P \quad (2)$$

where F_f represents the frictional force measured by the computer, and P is the applied load.

The wear volume (V) was determined from the mass loss of the specimen during the wear test. The specimens were weighed before and after testing using a digital analytical balance, and the wear volume was calculated by dividing the mass loss by the density of the composite, as shown in Eq. (3).

$$V = \frac{\Delta m}{\rho} \quad (3)$$

where Δm is the mass loss, and ρ is the density of the composite. After each wear test, the counter-body disc was thoroughly cleaned with ethanol to remove adhered wear debris and contaminants. The disc surface was then allowed to dry before conducting the next test. To ensure reliability and repeatability, three samples were tested, and the mean values are reported.

3. Results and Discussion

3.1. Density

The density of the developed periwinkle shell and raffia palm seed-reinforced epoxy composites was evaluated to determine the impact of fillers on the

compactness and structural integrity of the material. As shown in Figure 3, with error bars representing the standard deviation of repeated measurements, the density of the composite samples progressively increased from 2.60 g/cm³ in Sample A to 3.28 g/cm³ in Sample D. This trend can be attributed to variations in the filler composition and packing efficiency within the epoxy matrix. The increase in density suggests improved dispersion and compaction of the periwinkle shell and raffia palm seed particles, which likely reduced void content and enhanced structural coherence.

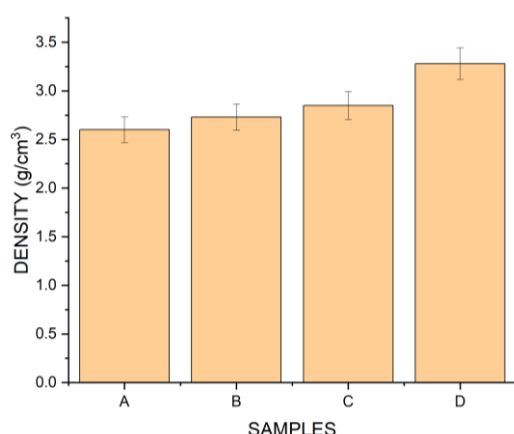


Figure 3. Variation of density with PWS/RPS composite samples.

The higher density observed in Sample D indicates a more compact microstructure, which is beneficial for products that demand high thermal stability and wear resistance, such as brake pads. Denser materials typically exhibit greater resistance to compressive forces and reduced porosity, which enhance the frictional performance and longevity of brake pads under operational stress [10], [24]. Moreover, the increase in density may also be associated with the increased proportion of periwinkle shell particles, which are known for their relatively high specific gravity due to their calcium carbonate content [1]. The hybridization with raffia palm seed particles potentially improves matrix-filler interaction, leading to better mechanical interlocking and reduced interfacial voids.

These results agree with previous studies, such as those of Yawas et al. [15], who reported enhanced density in periwinkle shell-based composites due to improved particle dispersion, and Ikpambese et al. [25], who observed similar trends in palm kernel fiber-reinforced materials. The density values obtained in this study fall

within the range required for commercial non-asbestos brake pads, further supporting the viability of these natural fillers in brake pad formulations.

3.2. Water absorption

Water absorption characteristics of the developed periwinkle shell/raffia palm seed-reinforced epoxy composites are shown in Figure 4, with error bars representing the standard deviation of repeated measurements. The results showed a progressive reduction in water absorption across samples A - D. Sample A exhibited the highest water uptake, corresponding to a water absorption percentage of 0.1214%, while sample D showed the lowest value of 0.0388%. This variation is primarily attributed to differences in filler composition and dispersion, interfacial adhesion between the fillers and epoxy matrix, and inherent porosity of each composite sample.

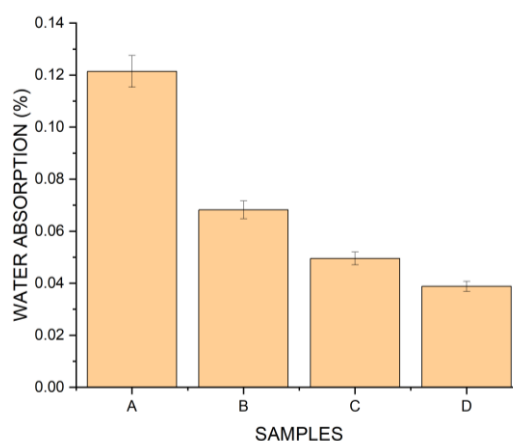


Figure 4. Variation in water absorption with the PWS/RPS composite samples.

The relatively high-water absorption in sample A suggests a higher concentration of hydrophilic constituents, which can include raffia palm seed particles with insufficient interfacial bonding, thereby facilitating water ingress. Conversely, sample D demonstrated superior resistance to moisture penetration, likely due to more uniform filler distribution and improved matrix-filler interaction, resulting in reduced void content and capillary action.

Water absorption in composite materials, particularly those intended for tribological applications, such as brake pads, is a critical factor influencing long-term

performance. Elevated moisture uptake can lead to the plasticization of the matrix, dimensional instability, and degradation of mechanical and thermal properties. This agrees with the observations of Athijayamani et al. [26], who stated that increased water absorption compromises the structural integrity of fiber-reinforced thermoset composites. Similarly, Koppaarthi and Netravali [27] emphasized that natural fiber composites with optimized filler treatment and matrix compatibility can significantly reduce moisture uptake, enhancing their suitability for humid and wet operational environments. The low water absorption observed in sample D reinforces its potential applicability in automotive brake systems, where moisture resistance is essential to ensure consistent braking efficiency and extended service life.

3.3. X-ray diffraction

The XRD pattern of the composite is presented in Figure 5, and the major diffraction peaks along with their corresponding interplanar distances are listed in Table 2.

For sample A, the major peaks corresponded to the phases of amorphous silica, mullite, quartz, calcium silicate, and hematite, while sample B exhibited the

phases of calcite, quartz, magnesia, calcium silicate, and alumina. Sample C had amorphous silica, quartz, kaolinite, hematite, and silicon carbide phases at major peaks, while sample D had calcite, aragonite/calcite, quartz, periclase, and calcium silicate as phases. Although aluminium chips were used as the abrasive additive during composite fabrication, XRD analysis revealed alumina (Al_2O_3) peaks in the spectrum. This phase is attributed to the naturally formed oxide layer on the surface of the aluminium chips. Aluminium readily reacts with atmospheric oxygen to form a thin, stable alumina film, which can be detected by XRD. Therefore, the identified alumina phase originated from the oxidation of the incorporated aluminium chips.

These phases influence the mechanical and tribological behavior of the composite material produced. The presence of phases such as quartz, mullite, alumina, and silica contributes to improved hardness and wear resistance by increasing the resistance of the composite surface to plastic deformation and abrasive wear. Similarly, calcium silicate and calcite phases obtained from the periwinkle shell reinforcement may enhance the load-bearing capacity of the composite and improve frictional stability during sliding contact [28].

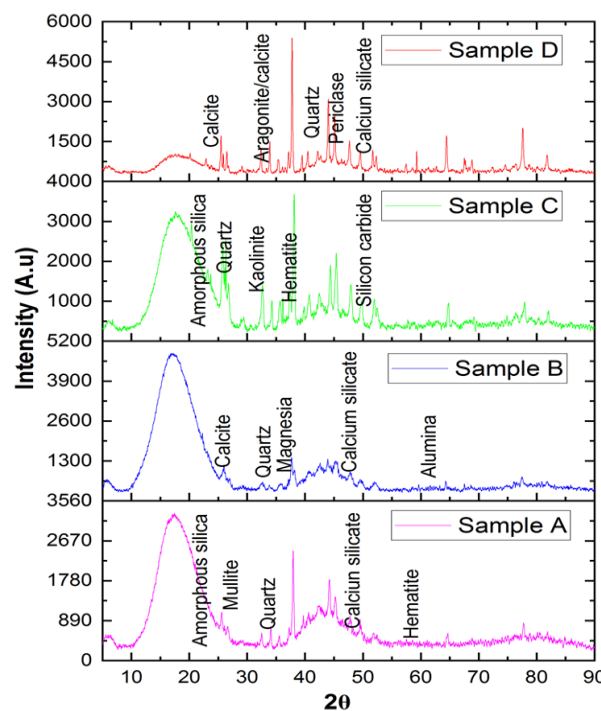


Figure 5. X-ray diffraction of the PWS/RPS composite.

Table 2. Diffraction peaks and their interplanar distances in the PWS/RPS composite.

Sample A		Sample B		Sample C		Sample D	
Major diffraction peaks (°)	Interplanar distance (Å)	Major diffraction peaks (°)	Interplanar distance (Å)	Major diffraction peaks (°)	Interplanar distance (Å)	Major diffraction peaks (°)	Interplanar distance (Å)
21.3	4.17	28.9	3.09	22.0	4.04	29.4	3.04
27.7	3.21	33.1	2.70	26.5	3.36	36.1	2.48
32.9	2.72	36.2	2.48	31.8	2.81	39.4	2.28
50.1	1.82	47.9	1.89	36.4	2.47	43.2	2.09
59.4	1.55	56.6	1.62	50.2	1.81	47.5	1.91

3.4. Hardness

The effect of hardness on the developed composite samples is as indicated in Figure 6, with error bars representing the standard deviation of repeated measurements.

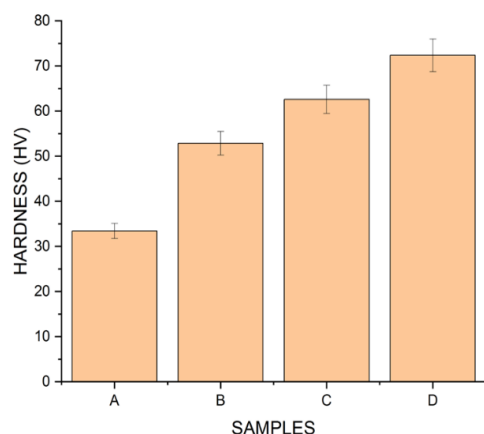


Figure 6. Variation in Hardness with the PWS/RPS composite samples.

It was found that the hardness increased with increasing composition of both periwinkle shell and raffia palm seed. Sample D has the highest hardness of 72 HV, and contains, 19.13g periwinkle shell powder and 14.87g raffia palm seed powder. This superior hardness makes it more suitable for high-performance brake pad materials. Higher hardness values like this are typically associated with enhanced thermal resistance, improved load-bearing capability, and lower wear rate, which is essential for

automotive and industrial brake systems [29], [30]. Periwinkle shells are also known for their high mineral content, predominantly calcium carbonate, which significantly contributes to enhanced hardness when integrated into a polymer matrix [31], [32].

Sample C has a hardness of 62.58 HV, whereas sample B has a hardness of 52.86 HV. The lowest hardness was found in sample A with 33.40 HV, having 4.78g periwinkle shell powder and 3.73g raffia palm seed powder. The progressive enhancement in hardness may be attributed to an increasing content or better dispersion of hard reinforcements (periwinkle shell and raffia palm seed) within the epoxy matrix, as hard phases enhance the material's resistance to plastic deformation under mechanical stress [5]. The synergistic effect of periwinkle shell (hard, brittle) and raffia palm seed (fibrous, moderately tough) may also contribute to a balanced hardness-toughness profile, with the increasing filler loading leading to better particle-particle interaction and reduced matrix plasticity [10].

3.5. Wear behavior

3.5.1. Wear depth

The wear behavior of the composites is presented in Figure 7. Results revealed significant differences in wear resistance across the samples, with error bars representing the standard deviation of repeated measurements. Sample

D exhibited the lowest wear depth (140.297 μm), signifying the highest wear resistance, while Sample A recorded the highest wear depth (257.859 μm), suggesting the poorest wear resistance.

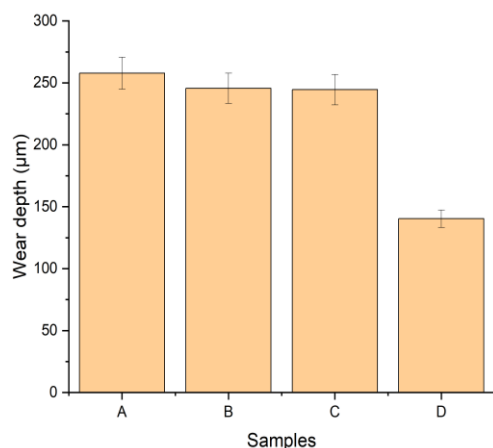


Figure 7. Variation in wear depth with the PWS/RPS composite samples.

The trend observed ($A > B > C > D$) implies that the composition and distribution of the reinforcing materials in Sample D provided a more effective barrier against abrasive forces, making it the most suitable formulation for brake pad application. This result corroborates the findings of Onuoha et al. [33], who observed that improved filler dispersion and interfacial bonding in composites can significantly enhance wear resistance.

Furthermore, the reduced wear depth in Sample D may be attributed to an optimal balance between the hard and tough components of the periwinkle shell and raffia palm seed particles. Studies have shown that incorporating bio-fillers like periwinkle shells, which possess good hardness characteristics, and fibrous materials such as raffia palm seed, can enhance load distribution and surface integrity under sliding wear conditions [34]. Adhesion and delamination were identified as wear mechanisms [10]. The superior performance of Sample D suggests that it could provide a longer service life in brake applications, reduce material loss, and maintain braking efficiency. This aligns with the work of Ibhade and Dagwa [35], who emphasized the role of natural hybrid fillers in improving the tribological behavior of composite brake materials.

3.5.2. Wear Rate

The wear rate values obtained revealed a clear trend of decreasing wear rate from Sample A (0.00000893 $\text{mm}^3/(\text{N}\cdot\text{m})$) to Sample D (0.00000819 $\text{mm}^3/(\text{N}\cdot\text{m})$), as shown in Figure 8, with error bars representing the standard deviation of repeated measurements. This reduction suggests improved wear resistance with changes in composition, likely due to optimized filler loading or improved interfacial bonding between the matrix and natural reinforcements. Lower wear rate is a critical indicator of a composite's suitability for brake pad applications.

The result for Sample D, which records the lowest wear rate, implies that this composition has the most efficient combination of periwinkle shell and raffia palm seed fillers, enhancing the composite's resistance to abrasion and surface degradation during braking.

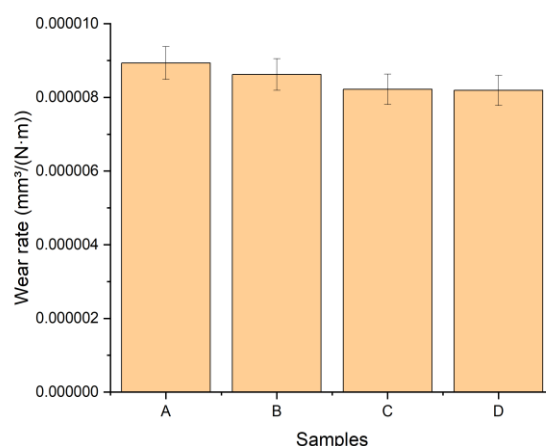


Figure 8. Variation in wear rate with the PWS/RPS composite samples.

This trend aligns with existing literature, which reports that proper reinforcement of epoxy composites with hard, particulate natural fillers, such as periwinkle shell, significantly enhances their tribological properties [10], [33]. The presence of high-silica and calcium-rich constituents in the periwinkle shell and lignocellulosic components in raffia palm seed likely contributes to improved load-bearing capacity and reduced micro-cutting during sliding. Furthermore, as the reinforcement percentage becomes more optimized across samples, the distribution of fillers likely becomes more uniform, reducing localized stress concentrations and contributing to lower wear.

3.5.3. Coefficient of friction

Figure 9 illustrates how the coefficient of friction (COF) values varied across the developed composites. All the four formulations exhibited an initial running-in period, where there was a quick rise in COF, followed by a more stable frictional state. The steady state COF values were approximately 0.70-0.75 for Sample A, 0.65-0.68 for Sample B, 0.60-0.63 for Sample C, and 0.57-0.60 for Sample D. These findings showed a clear trend of decreasing friction from Sample A to Sample D, with Sample D having the lowest COF. Notably, Sample A displayed more significant fluctuations during sliding, indicating a less stable frictional behavior under the tested conditions.

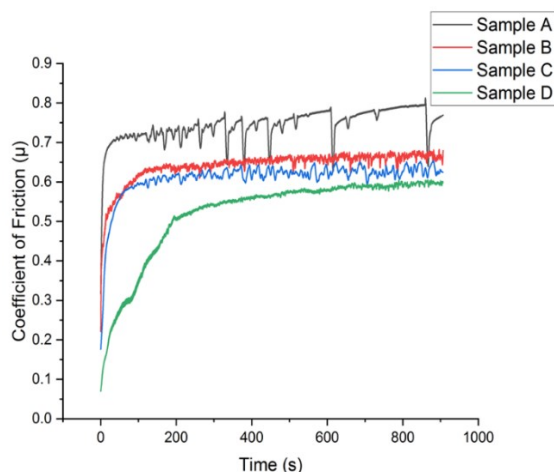


Figure 9. Variation in coefficient of friction among PWS/RPS samples over time.

However, a COF range of approximately 0.30-0.60 has been reported as a broad literature benchmark for some commercial non-asbestos organic (NAO) brake friction materials. In comparison to this benchmark, Samples A and B showed values that exceeded the upper limit of this range, while Sample C was at or above the upper boundary. High COF values may lead to increased heat generation, noise, vibration, and braking instability. However, excessively low values may reduce braking efficiency while a stable friction profile over time is critical for consistent braking action [36]. Sample D on the other hand, fell within the upper end of the specified range. This suggests that Sample D had the most similar

frictional response to the stated benchmark. This is likely due to a higher reinforcement content or better interfacial bonding between the filler and matrix, as also reported by Onuoha et al. [33].

3.6. Microstructural analysis of the PWS/RPS composites

The SEM/EDS micrographs of the composites demonstrate the influence of increasing reinforcement on the microstructure. The samples exhibited a homogeneous distribution of PWS/RPS particles within the resin matrix influenced by a good interfacial bonding, as shown in Figures 10–13.

The dispersion of PWS/RPS particles is evident in the white areas, while the resin is visible in the black areas. Sample A shows a smooth matrix surface with limited reinforcement distribution, indicating weak filler-matrix interfacial bonding and the presence of resin-rich regions. In Sample B, reinforcement particles become more visible and better dispersed, suggesting improved particle-matrix interaction.

Sample C presents a more uniform distribution of periwinkle shell, raffia palm seed, and aluminum particles within the epoxy matrix, with fewer voids and good adhesion. This is expected to enhance mechanical integrity and wear resistance. In contrast, Sample D exhibits a dense concentration of reinforcements with signs of clustering and agglomeration. While higher particle loading increases hardness and frictional stability, localized agglomerates may act as stress concentration sites, potentially initiating microcracks under service conditions.

Furthermore, the Al_2O_3 region identified corresponds to an aluminium-rich area containing both aluminium and oxygen signals in the EDS spectrum, confirming the presence of an oxide layer on the aluminium chip surface. These observations align with previous studies which reported that homogeneous dispersion of agro-waste reinforcements improves composite performance, whereas excessive filler content can lead to particle pullout and reduced toughness [10], [35], [37].

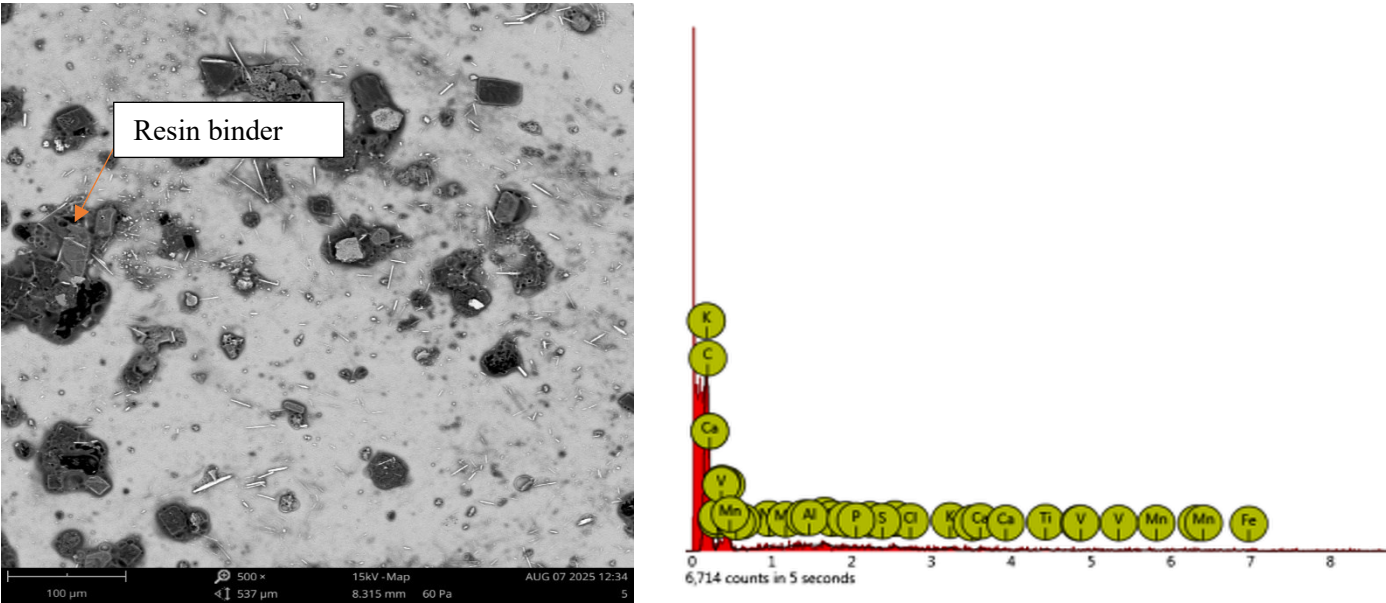


Figure 10. SEM/EDS images for the composition of sample A.

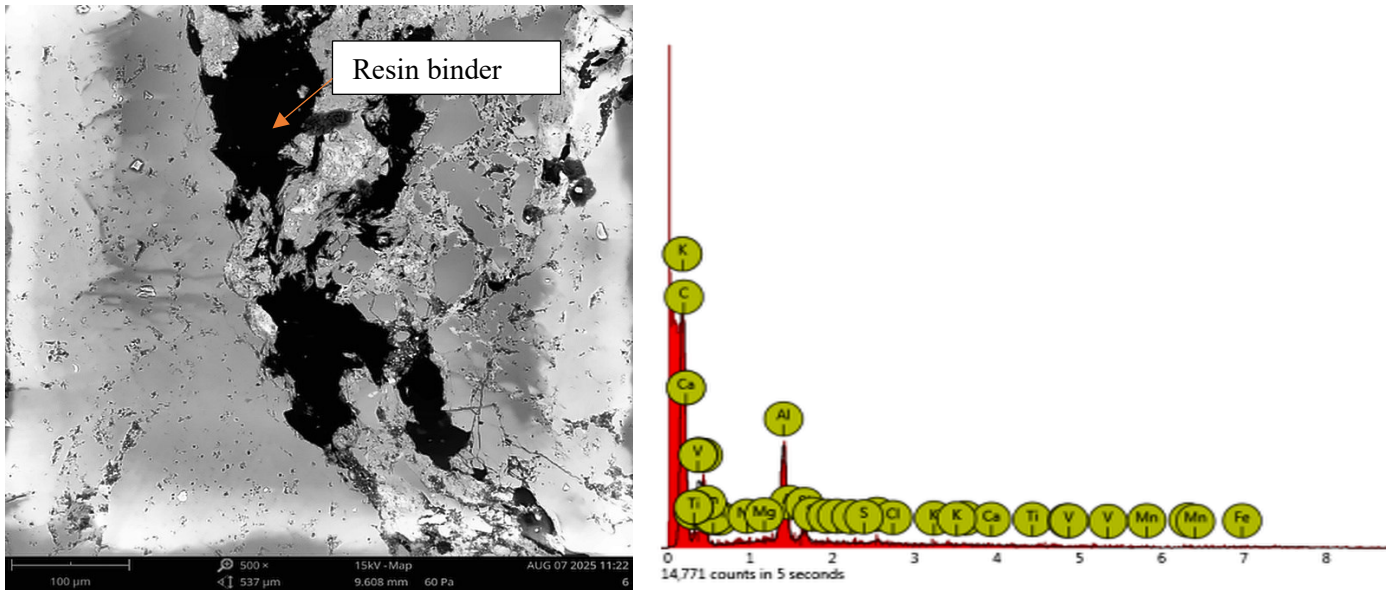


Figure 11. SEM/EDS images for the composition of sample B.

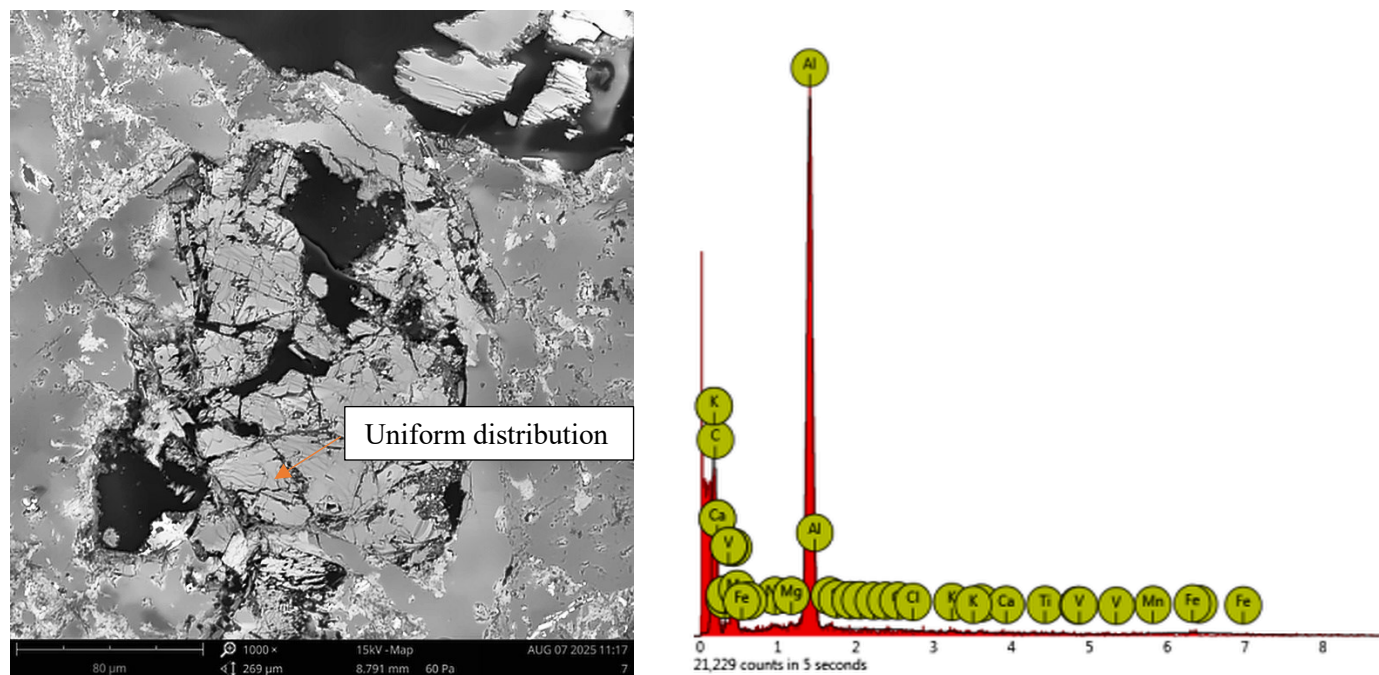


Figure 12. SEM/EDS images for the composition of sample C.

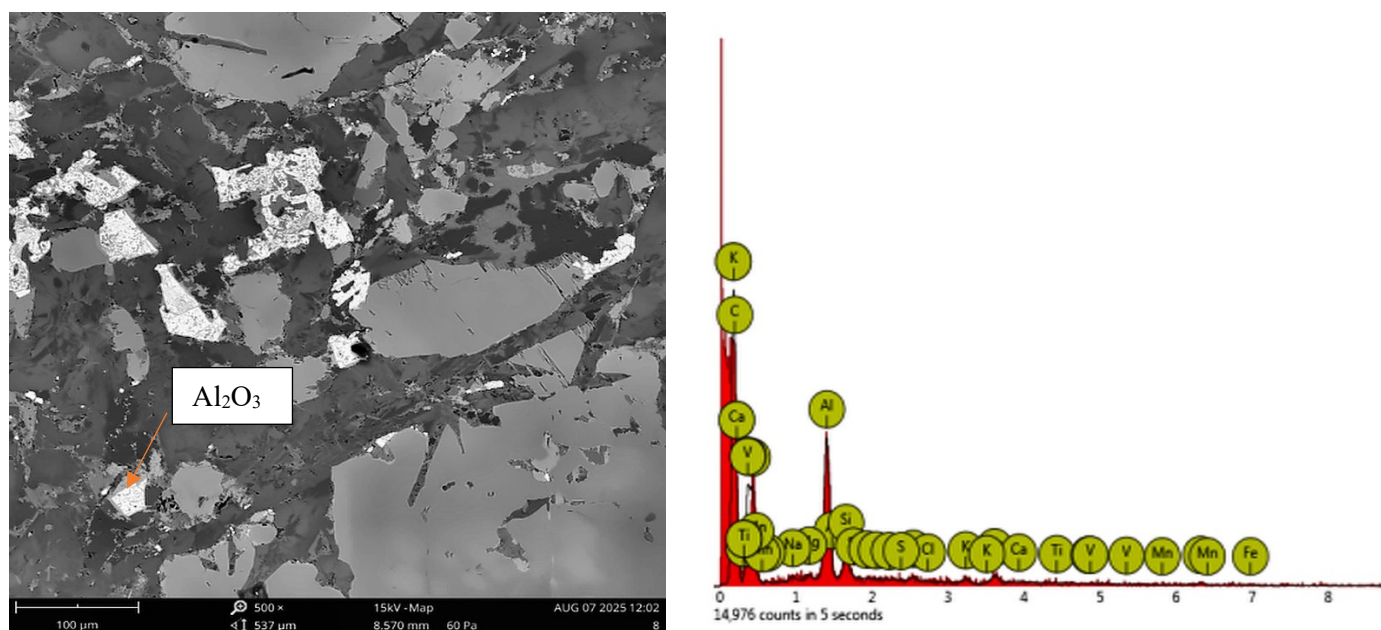


Figure 13. SEM/EDS images for the composition of sample D.

4. Conclusion

This study successfully developed hybrid epoxy composites reinforced with periwinkle shell (PWS) and raffia palm seed (RPS) particles for potential automotive brake pad applications. Four formulations (Samples A–D)

were produced with varying reinforcement contents, and their physical, mechanical, and tribological properties were assessed.

The results revealed that increasing the proportion of PWS and RPS particles considerably enhanced the

performance of the composites. The density increased from 2.60 g/cm³ in Sample A to 3.28 g/cm³ in Sample D, indicating improved compactness and reduced void content. Water absorption decreased from 0.1214% to 0.0388%, demonstrating enhanced moisture resistance. Hardness values increased progressively from 33.40 HV to 72 HV, confirming that higher reinforcement loading improves the resistance of the composite to plastic deformation.

Tribological analysis showed that Sample D exhibited the best wear performance, with the lowest wear depth of 140.297 µm compared to 257.859 µm for Sample A, and the lowest wear rate of 0.00000819 mm³/(N·m). In addition, the coefficient of friction decreased from 0.70 - 0.75 in Sample A to 0.57- 0.60 in Sample D, indicating improved frictional stability that may be considered for braking systems.

Generally, the hybrid composite containing higher contents of periwinkle shell and raffia palm seed particles (Sample D) exhibited improved hardness, enhanced wear resistance, higher density, and lower water absorption compared with the other formulations investigated. These results indicate that the developed composite may be considered a promising sustainable and environmentally friendly substitute to asbestos-based brake pad materials for automotive applications. The observed improvements indicate that increasing the reinforcement content certainly influenced the physical, mechanical, and tribological performance of the epoxy-based composite. The coefficient of friction obtained for the developed composites also fell closely to the ranges reported in the literature for certain non-asbestos friction materials. The study contributes to the growing body of research on sustainable friction materials by demonstrating the benefits of hybridizing periwinkle shell and raffia palm seed particles in an epoxy matrix and providing baseline data for future optimization and commercialization studies.

This study did not evaluate thermal performance, dynamic braking behavior, long-term durability, environmental ageing, or compliance with commercial and industry standards. Future work should focus on thermal and fade-recovery characterization, friction stability, durability under service conditions, and benchmarking against commercial brake pads to validate

the practical and industrial applicability of PWS/RPS hybrid composites.

Author Contributions Statement

Oryina Mbaadega Injor: Development of ideas, Approach, Analysis, Writing - original draft; **Moipone Linda Teffo:** Development of ideas, Resources, Supervision, Writing - review & editing; **Emmanuel Rotimi Sadiku and Victor Ugbetan Agbogo:** Resources, Writing-review & editing.

Competing Interests Statement

The authors declare that they have no competing interests.

Data Availability Statement

All data analyzed during this study are included in this article.

Statement on the Ethical Use of AI Tools

During the preparation of this work, the author(s) used Grammarly to enhance the paper's readability. After using this tool, the author(s) revised and edited the content as needed and take(s) full responsibility for the content of the publication.

Acknowledgement

The authors acknowledge the Institute for Nano Engineering Research (INER) and the Department of Chemical, Metallurgical and Materials Engineering, Tshwane University of Technology for providing the necessary facilities and relevant information utilized in the preparation of this manuscript. The authors also acknowledge the use of Grammarly as an AI tool to fine tune and correct grammatical errors.

References

- [1] S. Y. Aku, D. S. Yawas, P. B. Madakson, and S. G. Amaren, "Characterization of periwinkle shell as asbestos-free brake pad materials," *Pacific Journal of Science and Technology*, vol. 13, no. 2, pp. 57–63, 2012.
- [2] J. O. Dirisu, I. P. Okokpujie, P. B. Apiafi, S. O. Oyedepo, L. K. Tartibu, O. A. Omotosho, E. O. Ogunkolati, E. O. Oyeyemi, and J. O. Uwaishie, "Development of eco-friendly brake pads using industrial and agro-waste materials," *Journal of Engineering and Applied Science*, vol. 71, Art. no. 55, 2024, doi: 10.1186/s44147-023-00345-y.
- [3] M. S. K. Hemanth and J. E. R. Dhas, "Eco-friendly materials for brake pad: ANSYS overview," *Materials Today: Proceedings*, 2023, doi: 10.1016/j.matpr.2023.05.194.
- [4] A. Sellami and R. Elleuch, "Green composite friction materials: A review of a new generation of eco-friendly brake materials for sustainability," *Environmental Engineering Research*, vol. 29, no. 3, Art. no. 230226, 2024, doi: 10.4491/eer.2023.226.
- [5] O. M. Injor, E. R. Sadiku, M. L. Teffo, M. M. Ramakokovhu, V. U. Agbogo, and W. K. Kupolati, "Review of the development of periwinkle/palm kernel shell-reinforced epoxy matrix hybrid composites for brake pad production," *European Journal of Materials Science and Engineering*, vol. 10, no. 4, pp. 245–266, 2025, doi: 10.36868/ejmse.2025.10.04.245.
- [6] J. O. Dirisu, I. P. Okokpujie, O. O. Joseph, S. O. Oyedepo, O. Falodun, L. K. Tartibu, and F. D. Shehu, "Sustainable biocomposites materials for automotive brake pad application: An overview," *Journal of Renewable Materials*, vol. 12, no. 3, pp. 485–511, 2024, doi: 10.32604/jrm.2024.045188.
- [7] M. R. M. Asyraf, M. R. Ishak, A. Syamsir, N. M. Nurazzi, F. A. Sabaruddin, S. S. Shazleen, M. N. F. Norrrahim, M. Rafidah, R. A. Ilyas, M. Z. Abd Rashid, and M. R. Razman, "Mechanical properties of oil palm fibre-reinforced polymer composites: A review," *Journal of Materials Research and Technology*, vol. 17, pp. 33–65, 2022, doi: 10.1016/j.jmrt.2021.12.122.
- [8] H. O. Iwalola, S. C. Ikpeseni, S. O. Sada, and M. Ekpu, "Performance evaluation of the integration of periwinkle shell, cashew nut dust, and resin for manufacture of brake pad formulations excluding asbestos," *Journal of Applied Sciences and Environmental Management*, vol. 28, no. 10, pp. 2949–2955, 2024.
- [9] S. E. Mawuli, S. Anthony, and D. O. Akintunde, "Characterization of periwinkle shell powder as a possible replacement for asbestos in the production of automotive disc brake linings," *European Journal of Applied Sciences*, vol. 13, no. 2, pp. 106–118, 2025, doi: 10.14738/aivp.132.18429.
- [10] V. S. Aigbodion, U. Akadike, S. B. Hassan, F. Asuke, and J. O. Agunsoye, "Development of asbestos-free brake pad using bagasse," *Tribology in Industry*, vol. 32, no. 1, pp. 12–18, 2010.
- [11] H. Jariwala and P. Jain, "A review on mechanical behavior of natural fiber reinforced polymer composites and its applications," *Journal of Reinforced Plastics and Composites*, vol. 38, no. 10, pp. 441–453, 2019, doi: 10.1177/0731684419828524.
- [12] M. Ramesh, K. Palanikumar, and K. H. Reddy, "Mechanical property evaluation of sisal–jute–glass fiber reinforced polyester composites," *Composites Part B: Engineering*, vol. 48, pp. 1–9, 2013.
- [13] B. K. Satapathy and J. Bijwe, "Fade and recovery behavior of non-asbestos organic (NAO) composite friction materials based on combinations of rock fibers and organic fibers," *Journal of Reinforced Plastics and Composites*, vol. 24, no. 6, pp. 563–577, 2005, doi: 10.1177/0731684405043561.
- [14] C. M. Ruzaidi, H. Kamarudin, S. B. Jamaludin, M. M. A. Abdullah, and R. Abdulazak, "Mechanical properties and wear behavior of brake pads produced from palm slag," *Advanced Materials Research*, vols. 341–342, pp. 26–30, 2011.
- [15] D. S. Yawas, S. Y. Aku, and S. G. Amaren, "Morphology and properties of periwinkle shell asbestos-free brake pad," *Journal of King Saud University – Engineering Sciences*, vol. 28, no. 1, pp. 103–109, 2016, doi: 10.1016/j.jksues.2013.11.002.
- [16] A. Benson Bayode, O. Oluwamuyiwa Omoniyi, G. Daniel, O. Folasade Elizabeth, A. Olufemi Adegoke, A. John Oluwatayo, and A. Olusanya Tunde, "Property evaluation of raffia seeds reinforced epoxy matrix composite," *Journal of Materials Science Research and Reviews*, vol. 5, no. 2, pp. 175–185, 2022.
- [17] T. D. Ipilakyaa, S. E. Tile, G. B. Nyior, and D. T. Gundu, "Characterization of alkaline treated raffia palm fibres as reinforcement in polymer composite," *Engineering and Technology Journal*, vol. 42, no. 7, pp. 950–958, 2024, doi: 10.30684/etj.2024.147130.1704.
- [18] O. Fadele, I. N. A. Oguocha, A. Odeshi, M. Soleimani, and C. Karunakaran, "Characterization of raffia palm fiber for use in polymer composites," *Journal of Wood Science*, vol. 64, no. 5, pp. 650–663, 2018, doi: 10.1007/s10086-018-1748-2.
- [19] J. George, M. S. Sreekala, and S. Thomas, "A review on interface modification and characterization of natural fiber reinforced plastic composites," *Polymer Engineering & Science*, vol. 41, no. 9, pp. 1471–1485, 2001.
- [20] I. O. Oladele, I. O. Ibrahim, A. A. Adediran, A. D. Akinwekomi, Y. V. Adetula, and T. M. A. Olayanju, "Modified palm kernel shell fiber/particulate cassava peel hybrid reinforced epoxy composites," *Results in Materials*, vol. 5, Art. no. 100053, 2020, doi: 10.1016/j.rinma.2019.100053.
- [21] S. Shejkar, B. Agrawal, A. Agrawal, and G. Gupta, "Physical, mechanical, and sliding wear behavior of epoxy composites filled with surface-modified walnut shell particulate," *Polymer Composites*, vol. 43, no. 10, pp. 7247–7263, 2022, doi: 10.1002/pc.26847.
- [22] T. Popoola, A. B. Rabi, H. K. Ibrahim, P. O. Omoniyi, M. A. Babatunde, N. Muhammad, and F. O. Isiaq, "Production of automobile brake pads from palm kernel shell, coconut shell, seashell and cow bone," *Adeleke*

- University Journal of Engineering and Technology*, vol. 4, no. 2, pp. 92–101, 2021.
- [23] ASTM International, *Standard Test Method for Water Absorption of Plastics*, ASTM D570-98, West Conshohocken, PA, USA, 1998, doi: 10.1520/D0570-98.
- [24] Z. U. Elakhame, O. Alhassan, and A. Samuel, "Development and production of brake pads from palm kernel shell composites," *International Journal of Scientific & Engineering Research*, vol. 5, no. 10, pp. 735–744, 2014.
- [25] K. K. Ikpambese, D. T. Gundu, and L. T. Tuleun, "Evaluation of palm kernel fibers for production of asbestos-free automotive brake pads," *Journal of King Saud University – Engineering Sciences*, vol. 28, no. 1, pp. 110–118, 2016, doi: 10.1016/j.jksues.2014.02.001.
- [26] A. Athijayamani, M. Thiruchitrabalam, U. Natarajan, and B. Pazhanivel, "Effect of moisture absorption on the mechanical properties of randomly oriented natural fibers/polyester hybrid composite," *Materials Science and Engineering A*, vol. 517, no. 1–2, pp. 344–353, 2009.
- [27] S. D. S. Koppaarthi and A. N. Netravali, "Green composites for structural applications," *Composites Part C: Open Access*, vol. 6, Art. no. 100169, 2021, doi: 10.1016/j.jcomc.2021.100169.
- [28] P. J. Blau, "Compositions, functions, and testing of friction brake materials and their additives," Oak Ridge National Laboratory, Oak Ridge, TN, USA, Rep. ORNL/TM-2001/64, Aug. 2001, doi: 10.2172/788356.
- [29] J. Ajith, K. N. Bharath, and S. Basavarajappa, "Recent progress in the research on natural composite brake pads: A comprehensive review," *Tribology – Materials, Surfaces & Interfaces*, vol. 17, no. 4, pp. 259–281, 2023, doi: 10.1080/17515831.2023.2237810.
- [30] S. S. Lawal, N. A. Ademoh, K. C. Bala, and A. S. Abdulrahman, "A review of the compositions, processing, materials and properties of brake pad production," *Journal of Physics: Conference Series*, vol. 1378, no. 3, Art. no. 032103, 2019, doi: 10.1088/1742-6596/1378/3/032103.
- [31] A. Sadiq, A. A. Abdelmalik, U. Sadiq, A. Abdulraheem, and M. Balogun, "Influence of periwinkle shell microparticle on the mechanical properties of epoxy polymer composite," *Nigerian Research Journal of Chemical Sciences*, vol. 8, no. 1, pp. 25–36, 2020.
- [32] P. U. Adah, A. A. Nuhu, A. A. Salawu, A. B. Hassan, and P. A. Ubi, "Characterization of periwinkle shell ash reinforced polymer composite for automotive application," *FUDMA Journal of Sciences*, vol. 8, no. 1, pp. 83–92, 2024, doi: 10.33003/fjs-2024-0801-2158.
- [33] C. Onuoha, O. O. Onyemaobi, C. N. Anyakwo, and G. C. Onuegbu, "Physical and morphological properties of periwinkle shell-filled recycled polypropylene composites," *International Journal of Innovative Science, Engineering & Technology*, vol. 4, no. 5, pp. 186–196, 2017.
- [34] O. P. Okafor, E. N. Idenyi, and A. O. Agbo, "Fabrication and performance assessment of eco-friendly brake pad using palm kernel shell," *International Research Journal of Applied Sciences, Engineering and Technology*, vol. 8, no. 6, pp. 28–44, 2022.
- [35] A. O. A. Ibhado and I. M. Dagwa, "Development of asbestos-free friction lining material from palm kernel shell," *Journal of the Brazilian Society of Mechanical Sciences and Engineering*, vol. 30, no. 2, pp. 166–173, 2008.
- [36] F. Asuke and V. S. Aigbodion, "Experiment numerical study of dry sliding wear behavior of epoxy/periwinkle shell particulate composites," *Journal of the Chinese Advanced Materials Society*, vol. 4, no. 2, pp. 123–139, 2016, doi: 10.1080/22243682.2015.1124736.
- [37] M. I. Uzochukwu, M. O. Aiyejagbara, and M. Ugbaja, "Property investigation of cowhorn/periwinkle shell epoxy composite for automobile brake pad linings," *Journal of Emerging Trends in Engineering and Applied Sciences*, vol. 10, no. 3, pp. 86–91, 2019.