

REVIEW OF THE DEVELOPMENT OF PERIWINKLE/PALM KERNEL SHELL-REINFORCED EPOXY MATRIX HYBRID COMPOSITES FOR BRAKE PAD PRODUCTION

Oryina Mbaadega INJOR^{1*} [0009-0008-9453-0948], Emmanuel Rotimi SADIKU¹ [0000-0002-8504-1041], Moipone Linda TEFFO¹ [0000-0003-0347-7034], Munyadziwa Mercy RAMAKOKOVHU¹ [0000-0002-7196-9674], Victor Ugbetan AGBOGO¹ [0009-0002-3028-2005], Williams Kehinde KUPOLATI² [0000-0002-2574-2671]

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

² Department of Civil Engineering, Tshwane University of Technology, Pretoria 0001, South Africa

Abstract

Asbestos has traditionally been used in brake pad production due to its durability, but concerns over its health hazards, non-biodegradability, and high cost have prompted the search for safer alternatives. This study investigates the development of eco-friendly, asbestos-free brake pad composites using agro-waste materials, periwinkle shell (PS) and palm kernel shell (PKS) as reinforcements in an epoxy matrix. The uniqueness of the composites lies in combining the high thermal resistance of PS with the mechanical strength of PKS to create a cost-effective and sustainable friction material. The composites were produced with optimized particle sizes of 100–125 µm in various PS-PKS proportions, and their mechanical and tribological properties were evaluated. Results showed that finer particles reduced porosity, improved wear resistance and enhanced hardness up to 75 HRC for PS and 55.7 HRB for PKS. The best formulations achieved coefficients of friction between 0.35–0.44 and wear rates ranging from 0.017 to 0.170 mm/min, comparable to commercial brake pads. Thermal analysis confirmed that PS remains stable above 600 °C, while PKS decomposes in stages between 54–538 °C. These findings support the viability of PS/PKS-epoxy composites as high-performance, environmentally sustainable alternatives to asbestos-based materials.

Keywords: Asbestos, Hybrid composite, Periwinkle/palm kernel shell, Brake pad

Introduction

Brake pad materials are designed composites formulated to provide consistent friction, thermal stability, wear resistance, and durability under varying operating conditions to ensure safe and efficient braking performance. The increasing environmental concerns over non-biodegradable synthetic materials and the harmful emissions from traditional brake pads have driven interest in eco-friendly alternatives. Additionally, natural-based composites offer cost-effective solutions by utilizing abundant agro-waste products especially periwinkle and palm kernel shells, reducing raw material costs and promoting sustainable manufacturing practices [1]. Different machines employ different types of braking materials. All types/brands of vehicles with disc brakes, depend heavily on brake pads as part of their braking systems. Various car manufacturers, including Toyota, Nissan, Honda, Peugeot, Volkswagen, etc., employ brake pads in their braking systems. The steel back plate of these cars has frictional material that is affixed to the side that faces the brake disc [2-4].

*Corresponding author: InjorOM@tut.ac.za

The declaration of the average fuel efficiency standards, for corporations in the 1970's and 1980's, also drives the changes in the formulation of brake pads. This has compelled the auto industry to move away from the back wheel drive to front wheel drive vehicles. This move requires an added front braking scenario that often, leads to elevated temperatures and thus, a priority for the semi-metallic brakes [5]. The braking system plays a fundamental task in all vehicles, primarily functioning to reduce speed or bring the vehicle to a complete stop. Therefore, it must be highly reliable, and its performance should remain consistent throughout its service life [6, 7]. Developing sustainable friction materials is crucial for reducing environmental pollution, conserving non-renewable resources, and promoting the use of biodegradable, renewable alternatives in automobile applications. This study is focused on the development of a hybrid epoxy composite brake pad using periwinkle and palm kernel shells as dual agro-waste reinforcements, optimized for particle size and thermal-mechanical performance as a sustainable alternative to asbestos.

Material Complexity and Evolving Trends in Brake Pad Manufacturing

For an automotive brake system (ABS), the brake pads are friction-complex composites because they comprise of various components that vary in their mechanical, chemical and physical characteristics [8]. The lining material, which is a major part of the brake pad, based on the arrangement of the basic parts, is classified as metallic, semi-metallic and organic, or carbon-based, [9]. Among the many different components that make-up these brake pads, are binders, fibres, fillers, and frictional modifiers or additives (Fig. 1) [10]. The brake pad is typically, composed of several parts and asbestos fibres, encased in a polymeric matrix.

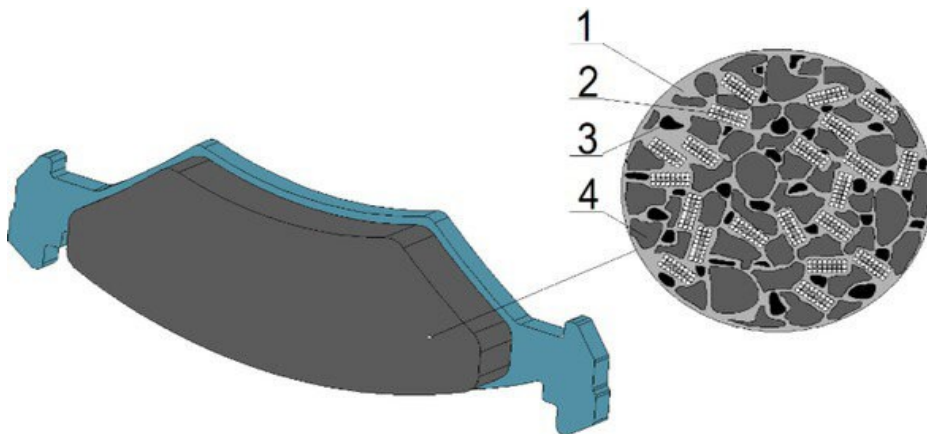


Fig. 1. Brake pad friction material configuration: 1 - Binder, 2 - Fibers, 3 - Filler, 4 – Additive.

Asbestos is a hydrated magnesium silicate and its fibres, occurring genuinely, as mineral, have been used to strengthen the components of frictional materials to confer onto them, the necessary mechanical strength [11]. This is because of its excellent engineering properties, e.g., thermal stability at $\sim 500^{\circ}\text{C}$. However, because of the health risks, asbestos use has been discouraged [12]. This is because it can settle in the lungs and initiate certain harmful respiratory conditions. Therefore, the search for non-toxic and cheaper substitutes, becomes imperative. In as much as it is still legal to employ asbestos for brake pads, many brake pad manufacturing industries are pulling away from asbestos; due to concerns that are associated with flying particles in factories and the associated concerns about the waste disposal of the remnant materials that contain asbestos [13].

Brake pad manufacturers employ ~2000 various materials [14], in brake production, each differently, affecting the final product. A standard brake pad comprises between 10 and 20 different materials [15]. The determination of the optimal formulation and the anticipation of its effect on the brake performance, is a challenging process that demands extensive research and specialized knowledge [16]. This decision must also consider the brake pad's intended use and the conditions under which it will operate. Moreover, the manufacturing process plays a vital role in defining the final properties of the brake pad and it is often considered to be a highly protected trade secret, among manufacturers. The use of advanced manufacturing technologies can significantly enhance the tribological performance of brake pads, potentially by up to 100% [17, 18].

Constituents and Classification of Brake Pad Materials

Brake pad materials can be categorized into various ways, with the most critical classification, based on the function each material performs in the braking process. According to this approach, the key constituents are the: binders, additives, fillers, and abrasives [19-21]. The binder functions as the matrix which integrates other components. It must possess a consistently high friction coefficient, strong resistance to extreme or variable temperatures, and maintain a low overall weight [22]. Furthermore, the material should not interact with any other part of the pad, since this could result in alterations to the overall material properties or lead to delamination of the composite. Such issues can severely compromise the braking efficiency. Typically, binders are formulated by using epoxy or silicone resins [23]. Reinforcements are fibrous materials that are incorporated to enhance the mechanical properties of the binder thereby boosting its strength. The choice of reinforcement performs a crucial role in the determination of the brake pad's durability and structural integrity, and thus, must be produced with careful considerations.

Historically, asbestos was widely used as an effective reinforcing fiber. However, due to its harmful health effects [24], alternative materials to asbestos have been developed and they are now readily available, thereby offering safe and efficient performance [25, 26]. Fillers serve to fill the gaps between other constituents in brake pad composition. Since they can constitute as much as 10% of brake pad's volume, the selection of an appropriate filler material is essential to ensure optimal performance. The most frequently used fillers consist of vermiculite, perlite, mica, barium sulfate, and calcium carbonate, since these materials resist high temperatures, they do not react with other brake pad components, and they are cost-effective [27].

Depending on performance required, abrasives are incorporated into pads to modify the friction coefficient. Additives, such as steel, cast iron, flame-resistant oxides, silicates, and quartz are commonly used to boost friction between the brake pad and the disc, thereby improving durability due to their hardness. This effect is further strengthened by their interaction with the disc surface, particularly when metallic elements are involved. These materials also create contact zones, which act as primary friction interfaces between the braking components [28, 29]. However, these contact zones generate significant heat due to friction, which can compromise the structural integrity of the brake pad, thereby causing delamination of its components. To counteract this issue, lubricants are incorporated to enhance the pad's thermal conductivity. These lubricants help to dissipate heat more effectively, from the contact zones, hence, reducing the risk of overheating and therefore, maintaining the performance of friction materials [30-32]. The generally used lubricants include metallic sulfates, such as copper and tin, as well as graphite. The effectiveness of lubrication depends on both the content (typically around 10% of brake pad's volume, yields optimal performance) and the particle size of the lubricant [33].

Another method for the classification of brake pad materials is by their origin, either natural or synthetic. Natural materials comprise asbestos [34, 35], a mineral that has been historically used, but now banned due to its carcinogenic nature [36], and plant-based substances, derived from leaves, stems, or seeds. Others are animal-based materials, such as hair or fur, and several metals, such as steel, copper, bronze, and cast iron. In contrast, synthetic materials are typically

products of organic synthesis, such as aramids and acrylic fibers, or advanced materials, such as mineral fibers, ceramics, carbon fibers, and glass fibers.

Role and Performance of Resin Binders in Brake Pad Composites

Resins are commonly used as binders in brake pad production, thereby serving the essential role of holding all components together. However, they must be incorporated in precise measured quantities to perform effectively, since deviations can compromise the structural reliability and performance of brake pads [37, 38]. Insufficient resin diminishes pad durability, while excessive resin can lower the friction coefficient at higher temperatures and greatly enhance hardness [39, 40]. Resin plays a critical role in brake pad manufacturing by bonding the composite components into a cohesive structure. However, the intense heat generated during a braking scenario can lead to resin degradation, hence, compromising its adhesive strength and the overall integrity of the brake pad [41]. Therefore, thermal stability and the capability to retain the mechanical properties of the pad under harsh braking conditions, rely heavily on the performance of the resin [42]. To enhance both the mechanical and thermal characteristics, ongoing research focuses on the modification of resin formulations through the chemical composition adjustments of the constituents [43, 44].

Phenolic resins are the most extensively used binders in the production of friction materials. While they have limited applications outside this domain, they are integral components to the brake pad formulations, often comprising between 5% and 35% of the pad's volume. Notably, the quantity of resin inversely affects the coefficient of friction (COF) as well as wear rate. Although a reduction of approximately 10% in resin content only resulted in a minor change in the COF, however, it still influenced the overall performance [45]. The extensive use of phenolic resins in the automotive industry is largely attributed to their high mechanical properties and cost-effectiveness [46]. Another important aspect is that this resin exhibits low thermal expansion. In comparison to other resins, it shows greater resistance to solvents, acids, and water, and produces minimal smoke when burned. The temperature at which early degradation occurs, which is significant due to its relationship with brake pad fading, drops from 354°C for pure resin to 303°C for cured friction material [47]. The addition of oil or silicone gum can help to decrease its susceptibility to cracking, resulting in a silicone resin. Nonetheless, during this process, various other mechanical properties are compromised [14]. Phenol resin is not the ideal choice for friction amelioration at elevated temperatures. In instances of abrupt braking, the pad temperature can hurriedly reach 700°C, which is by far, surpassing the degradation threshold of phenol resin [48, 49].

The cured condensed polynuclear aromatic (COPNA) resin, offers a slight improvement in thermal resistance. while its decomposition temperature is like that of other resins (400–500 °C), it releases considerably less gas during degradation. This lessens the likelihood of the brake fading. Furthermore, due to its graphite-like structure, the COPNA resin forms a strong bond with graphite particles, thereby enhancing the brake pad's resistance to friction [50, 51]. Cyanate ester resins, synthesized by using cyanide-based monomers, exhibit excellent chemical stability and they are known for their vibration-dampening properties. However, their most notable characteristic is their high thermal resistance. Studies have shown that the addition of ~5% zirconium diboride (ZrB_2), further improves its thermal stability and optimizes its tribological properties, particularly the COF and wear rate [52]. Additional research involving the combinations of graphite and molybdenum disulfide (MoS_2) has demonstrated improvements in the mechanical properties of the resultant product, along with reduced wear rates [38]. Despite their performance benefits, cyanate ester resins are rarely used in friction materials, due to their high cost, which results from a complex manufacturing process [53]. However, their superior properties have led to their growing usage in the electronics industry [54, 55].

Epoxy resins are widely used across various industries; however, their low thermal resistance (degrade at ~260 °C), makes them unsuitable for use in friction materials [56]. Research has

shown that at a relatively low temperature of 100 °C, epoxy resins can experience a mass reduction of ~7% [57]. This weight loss is attributed to the release of volatile compounds during the heating process. When exposed to prolonged heating above 400 °C, carbon formation and subsequent oxidation occur. Interestingly, the blending of epoxy with phenolic resins produces a composite material with significantly improved heat resistance and superior mechanical properties when compared to either of the resins, when used alone [56]. Another alternative, i.e., thermoplastic polyamide resin, produced by using fluorene and calcium carbonate, exhibits excellent wear resistance and thermal stability. In addition to its performance benefits, it offers a cost-effective and simple manufacturing process. Nevertheless, the resin serves as an effective thermal insulator, that is roughly three times, more efficient than phenol resin. This is the reason for its practicality in brake pad production, which is quite limited [56].

Fundamentals and Engineering Significance of Composite Materials

A composite is the arrangement of materials made of the combination/mixture of dissimilar components in a suitable arrangement, with a boundary separating and making them essentially, insoluble in one another with different forms and chemical compositions. Natural plant fibres in composites, have been in existence for centuries [58]. A composite material has engineering relevance when several distinctly different material constituents combine to form a unique material that have exceptional or outstanding characteristics when compared to the singular/individual components. Hence, composite materials are produced by lining-up exceptionally, formidable and rigid ingredients, e.g., fibres and tiny particles in a binder, known as matrix. This class of materials have excellent mechanical properties. One of the constituent parts, allows stress to be incorporated into the reinforcing phase, which creates a strong bond, referred to as matrix. Examples of matrix materials are polymers, ceramic and metals. Another component is the reinforcing phase, referred to as the reinforcement, which can be fibre, particulate or laminar. The distinctive features that composite materials possess, is as a result of the separate components as well as their boundary interaction.

Literatures have identified the factors that control the mechanical behaviour of particulate composites. It is generally, established that the particle size decreases with increased adherence to the matrix, as the reinforcing effect grows. Two polymers can be physically combined to change their properties, or a polymer can be combined with a non-polymeric component. Composites typically, exhibit both high strength and modulus, with a low density. They also exhibit resistance to wear, corrosion, fatigue, creep, and creep rupture [59].

Classification of Composite Materials in Automotive Systems

Composite materials can be generally categorized into three classes, depending on the matrix material. These include metal matrix composites, ceramic matrix composites and polymer matrix composites. Metal matrix composites have a metal, e.g., aluminium, magnesium and titanium as the matrix. Carbon and silicon carbide are used as fibres for reinforcement. The reinforcement is principally, to fit the need of the design. For instance, the addition of silicon carbide, as a fibre, increased the strength and the elastic stiffness of metals, with reduced electrical and thermal conductivities [60].

Metal matrix composites (MMCs) are gaining prominence in modern automotive innovations because of their superior mechanical and thermal properties. These composites are not restricted to braking systems, but likewise utilized in clutches [61], intake manifolds [62], engine blocks [63], and various fuel system components, including the traditional injectors that employ piezoelectric stacks [64, 65] and alternative fuel injectors [66]. Additionally, MMCs serve as substitutes for conventional materials in the fabrication of monocoque-type vehicle frames [67], thereby offering enhanced performance and reduced weight. In brake applications, MMCs are

used in the manufacture of brake drums and discs. These components are found, not only in passenger cars [68–70], but also across a range of other vehicles, including motorcycles [71]. In the brake pad friction linings, materials that are predominantly based on aluminum, often reinforced with silicon carbide, are utilized [72]. Their remarkable attributes, including low density, strong mechanical strength, high-temperature resistance, and efficient thermal conductivity [73–75], are the qualities that are often sought after in brake pads. Regrettably, this class of material also comes with its disadvantages.

Challenges, such as uneven distribution of SiC particles, residual porosity, formation of undesirable phases, e.g., aluminum carbide, and exposure to higher braking temperatures, significantly hinder the broad application of metal matrix composites in braking systems [75]. The final characteristic pertains to the elevated friction coefficient when interacting with various materials, and excessive heat could result in material deterioration [76]. A critical challenge in the application of MMCs, lies in their production methods. This includes liquid-metal mixing techniques, where reinforcement particles are incorporated into a molten aluminum matrix, through mechanical stirring. Others are the liquid-metal penetration method, which involves the forcing of molten aluminum or its alloys, into a reinforcement preform, either as a compacted bed or a self-supporting structure; and the solid-state techniques, e.g., powder metallurgy [77]. When compared to the fabrication of resin-based friction materials, these processes require more sophisticated equipment and a deeper level of technical expertise.

Ceramic matrix composites have aluminium silicate, alumina and calcium as matrices, with silicon carbide as reinforcement. Ceramic matrix composites feature low density, high strength, hardness, and ceramic service temperature restrictions. Polymer matrix composites are highly sophisticated when compared to both the metal matrix composites and ceramic matrix composites. They are composed of thermoplastic polymers or thermosets, reinforced by natural carbon, which can be formed into various sizes and shapes. They produce superior properties, such as high stiffness and strength with high resistance to corrosion. Their tremendous strength, low cost, and ease of manufacturing, make them the most widely used composites [78].

Advantages, Applications and Limitations of Composite Materials

The strength, rigidity, and lightweight nature of composite materials are their greatest benefits. In selecting a suitable combination of reinforcement, the properties that precisely, match the specifications for certain uses, can be produced by manufacturers. For example, the modern aviation industry would be less effective without composites; this is because the requirement for materials that are both strong and lightweight, has been the major factor motivating the manufacture of composites. Advanced composites are commonly, used to make parts, such as rotor blades, propellers, wing and tail sections, and considerable numbers of the interior structures and accessories. A few of the smaller aircraft's airframes, as well as the stringers, tail, and body panels of big commercial airplanes, are made entirely, of composite materials [79].

Metals, such as aluminium are more prone, as opposed to composites, to entirely rupture when subjected to stress. This is because, minute fissure in a metal object can expand quickly, and may result to devastating consequences, particularly in aircrafts. However, the fibres in a composite, work to share the stress distributed in the composite and prevent any small crack from spreading. The appropriate composites, are often, very durable and resistant to heat and corrosion. This renders them to be perfectly and adequately ideal for usage in items that are subjected to harsh conditions, e.g., spacecraft, watercraft, and chemical-handling equipment [79]. Due to their ability to be moulded into extremely complicated shapes, e.g., surf, composite materials also provide design versatility.

The only drawback of composites is often, normally their costs. Even though the use of composites improves production processes, the raw materials are rather very costly. It is evident that composites will not completely, replace conventional materials, e.g., steel, but in many cases, they are just what is required. With the development of technology, new applications for composites, will undoubtedly emerge because what composites can do is not known yet [80].

Emerging Role of Natural Fibre Composites in Sustainable Brake Pad Development

The growing awareness in composite materials made of polymers reinforced with natural fibres, is associated to both their industrialized uses and the ultimate research endeavours on these materials. They are cheap, renewable, wholly or partly reusable, and they are eco-friendly. Plants, such as cotton, hemp, flax, jute, sisal, pineapple, kenaf, wood, banana, bamboo, etc., were used before now, as sources of lignocellulosic fibres, and they are more frequently, utilized to reinforce composites. Their accessibility, low densities, low prices and suitable mechanical properties, place them to be preferable candidates to glass, carbon and artificial fibres, often used to produce composites. The composites produced from natural fibre are more environmentally friendly, and they find applications in the transport industry (aerospace, rail system and automobiles), the defence sector, the construction and building sectors (ceilings, and partitioning of boards), the wrapping/packaging sectors, in the end-user goods, etc [81].

Considerable number of studies are in the domain of developing brake pads, free from asbestos. Agro-waste utilization, such as coconut shell, palm kernel shell (PKS), etc., were previously examined [82-85]. Table 1 shows the summary of some findings on the development of coconut shell to produce brake pad.

Table 1. Summary of some findings on coconut shell-based materials for brake pad production

S/No	Authors	Materials	Findings
1	Olumodeji [86]	Coconut shell and other agricultural waste	They exhibit better friction properties than imported pads
2	Abutu et al. [87]	Coconut shell and Seashell	All of the parameters considered have major impact on the brake pads' mechanical and tribological characteristics, as evidenced by their p-values of >0.010 (1%).
3	Bretotean et al. [88]	Coconut fiber	As the amount of coconut fiber increases, so do the hardness, compressive strength, and longitudinal elasticity modulus.
4	Abutu et al. [89]	Coconut shell	They exhibit steady friction coefficient, improved hardness and minimal wear rate.
5	Apasi et al. [90]	Coconut shell	As the coconut powder increases in the samples, there is increase in the tensile and impact strengths, with a reduction in wear
6	Abutu et al. [91]	Coconut shell and other agricultural waste (asbestos free materials)	The braking temperature of the developed coconut shell brake $\leq 350^{\circ}\text{C}$ and microstructural analyses confirmed the suitability of asbestos-free pads.
7	Datau et al. [92]	Coconut shell ash and Kyanite particles	The mean particle size in the coconut shell ash microstructure was $26.42\text{ }\mu\text{m}$, while utilizing it as reinforcing materials, will improve strengthening.
8	Kholil et al. [93]	Coconut fiber, wood powder, cow bone	Composite suitable for motorcycle and light vehicles brake pads with improved thermal and wear properties.
9	Simamora et al. [94]	Candlenut shell and coconut shell	Samples produced have higher hardness and high resistance to wear.
10	Shuaibu et al. [95]	Coconut shell powder & ash, gum arabic	Hardness and density increase while water and oil absorptions decrease as the particle sizes decrease from $450\text{ }\mu\text{m}$ - $154\mu\text{m}$.

Research around the globe, concentrates on the approaches that employ wastes from agriculture or industry, as sources of raw materials for the sector. The use of these waste materials

will be both economical, and result to foreign exchange earnings (in regions where they are available in abundance) and in negative environmental amelioration. Another agro waste is the bagasse, which is the fiber that is left upon forcefully pressing sugar cane to extract the sugar [96]. Bagasse is freely obtainable, and it is not harmful [4]. Some of the bagasse is burnt to provide heat for sugar processing operations. Before now, ~90 percent of empty bunches, fibers, branches, stalks were disposed as trash and burned outside or dumped in waste ponds. As a result, the sugar cane manufacturing industry wastes, adds substantially, to the carbon dioxide emission [13].

Periwinkle Shells as Sustainable Reinforcement for Brake Pad Production

The periwinkle (*Turritella communis*) is a dark, oval, and hard-shelled aquatic snail that is edible. The outer shell that covers a periwinkle is a naturally occurring structure. This exterior structure protects the winkles from mechanical harm and potential assailants. This structure has many segments, and it is naturally composed of conchiolin, an organic matrix, stuck together with calcium carbonate precipitates [97, 98]. The water-resistant nature of these organic matrix shells, loaded with calcium carbonate, allows the periwinkle shells and their byproducts to have a wide range of uses [99]. Periwinkles are plenty on the rocky coastlines in the surrounding regions of South-South Nigeria, namely: Cross-River, Rivers, Akwa-Ibom and Bayelsa States of Nigeria. There are numerous markets in Nigeria, where they are available for sale. The shells are disposed after consumption, contributing to solid waste in the cities where they are produced, which poses substantial risks to humans and the environment [100]. Consequently, their efficient, favourable, and environmentally friendly use, has long portrayed a problem for scientific applications.

One of the most unconventional materials employed in brake pad production is the periwinkle shell, a food processing by-product that poses environmental and health risks due to the large volumes of waste that it generates. The utilization of crushed periwinkle shells as an alternative for asbestos in brake pad manufacturing, presents a promising solution to the disposal challenge. Studies conducted in Nigeria, evaluated the mechanical properties of periwinkle shell particles smaller than 255 µm, revealed that the material has a lower density compared to asbestos and it demonstrated significant resistance to high temperatures, with its thermal degradation only beginning at temperature beyond 600 °C. Moreover, the material demonstrated significantly, greater hardness, measuring approximately 75 HRC, indicating its superior rigidity when compared to asbestos [101, 102]. Further research conducted in Nigeria, expanded on the earlier findings, by developing multiple brake pad prototypes that incorporated periwinkle shell particles of varying sizes, ranging from 100 - 350 µm. The study identified that particles sized at 100 µm, offered the most favourable properties. These prototypes demonstrated the highest compressive strength and hardness, along with the lowest levels of water and oil absorption. According to the researchers, these performance metrics surpassed those of the commercially available brake pads [103]. Table 2 shows the composition of periwinkle shell particles, while Fig. 2 shows the photograph of the periwinkle shells.

Table 2. Analysis of the elements in the periwinkle shell particles [103]

S/No	Element	Periwinkle shell	Unit
I	SO3	0.30	Wt %
II	CaO	96.09	Wt %
III	Fe2O3	0.79	Wt %
IV	K2O	0.52	Wt %
V	MgO	1.54	Wt %
VI	Na2O	0.10	Wt %
VII	SiO2	0.09	Wt %
VIII	MnO	0.06	Wt %
IX	Cr2O3	0.003	Wt %



Fig. 2. Periwinkle Shells

Properties and Potential of Palm Kernel Shell (PKS) as a Sustainable Reinforcement in Brake Pad Production

Plant fibres, such as shells of coconuts and palm kernels, are hydrophilic because they are made of lignocellulose, which has hydroxyl groups that are highly polarized [104]. When compared to glass fibres, lignocellulosic fibres have several advantages due to their biodegradability, renewable nature, and appropriate unique qualities. Essentially, these fibres have intriguing physical and mechanical characteristics [105].

The Palm Kernel Shells (PKS) are solid rocky endocarps that enclose the kernel and the shells, with varying sizes and structures [106, 107]. There exist two types of these shells, which are “Tenera” and “Dura”. In contrast to the Dura type, the hybrid Tenera has a small shell thickness and was created specifically to produce a high oil content [108]. The shell is composed of approximately 21% combustible gas, 45% pyroligneous liquor, and 33% charcoal [109]. The palm kernel shell itself is a material with low moisture product and high density, and it can burn, thereby producing combustion Calorific heat of about 4,200 kcal/kg. The PKS are gotten after the palm oil has been extracted. After breaking the nuts and removing the kernels, the shells are typically abandoned as wastes.

Palm trees are primarily, cultivated in plantations across West Africa. Between 15-18 tons output per hectare of palm tree plantation, renders the PKS a plentiful and low-cost resource. Its minimal density in relation to other organic waste, allows for easy and cost-effective transportation [110]. Due to its fibrous structure, PKS has been investigated as a viable alternative to asbestos in brake pad manufacturing. PKS can make up as much as 40% of the friction layer volume [111]. When processed into powdered form, PKS exhibits low hygroscopicity, along with high rigidity and toughness, which are beneficial in braking applications [9]. Additionally, PKS demonstrates a high coefficient of friction when combined with cast iron brake discs. However, its effectiveness diminishes at elevated temperatures because during a high-speed braking the coefficient of friction drops considerably, as a result of its limited thermal resistance. Furthermore, oxidation heightened the wear of the pad, which occurred drastically faster when compared to the conventional brake pads [112, 113].

Research carried out by Mgbemena et al., [114] identified the initial temperatures of thermal decomposition in three distinct phases, viz: 54.28 °C, 237.14 °C, and 538.57 °C, accompanied by notable weight losses of: 6%, 27%, and 42.37%, respectively. Brake pads designed for cold braking applications that do not reach extremely high temperatures, performed significantly better under these conditions. The particle size of PKS used in pad production, also plays a critical role. Pads that incorporate large PKS particles, exhibited increased hygroscopic absorption and

improved wear resistance. However, most favourable mechanical properties, including high hardness, enhanced durability, and low porosity, were achieved when finely grounded PKS material was used [60]. In this modern technological era, bio-based composites provide the potential for environmental benefits, low energy consumption, light weight, insulation, and the capacity to absorb sound and acoustics [115]. Table 3 shows the composition of palm kernel shell particles, while Fig. 3 shows the palm kernel shells.

Table 3. Analysis of the elements in palm kernel shell particles [60]

S/No.	Element	Level Detected (Palm kernel shell)	Unit
I	Br	0.005	Wt %
II	Ca	0.028	Wt %
III	Cu	0.009	Wt %
IV	Cr	0.002	Wt %
V	Fe	0.006	Wt %
VI	K	0.005	Wt %
VII	Mn	0.002	Wt %
VIII	Se	0.003	Wt %



Fig. 3. Palm Kernel Shells

Preparation and Molding of Periwinkle and Palm Kernel Shell-Based Composites

Onyeneke et al [115] reported that crushed periwinkle shell (PWS) and cracked palm kernel shell (PKS) were soaked separately, in distilled water, diluted with 1% (v/v) sodium hydroxide (NaOH) at ambient temperature. After three (3) hours of mechanical stirring, the mixture was filtered, cleaned with distilled water, and dried for twenty-four (24) hours at 80oC in an oven. The cleaned shells of PWS and PKS were burnt separately, in the furnace at a temperature of 500 - 600°C; thereafter the shells were removed from the furnace and cooled for some time before being cut with a hammer into small fragments. These small fragments were thereafter, grounded/pulverized into powder and sieved according to the BS 1377; 1990 standard, with

particle sizes ranging from 80 to 150 μm [115]. A wooden rectangular mould was prepared to carry out the moulding operation. The grounded PWS and PKS powder prepared were then, mixed in different proportions. Each proportion was mixed with epoxy resin in the desired amount and aluminium filings were added to obtain the required strength and hardness to the mixture. After that, the mixture was transferred into the prepared mould and mechanically pressed. After about 30 minutes, the laminate was taken out of the mould and cut to the required sizes for mechanical testing.

Mathematical Formulation of Volume and Weight Fractions in Composite Laminates

The volume fraction for a particulate (v_p) and for the matrix (v_m) and the weight fraction of a particle (w_p), as well as for the matrix (w_m) of a composite laminate, are defined by using Eq. 1 [99].

$$v_p = \frac{V_p}{V_c} \text{ and } v_m = \frac{V_m}{V_c} \quad (1)$$

where V is the volume of the constituent under consideration, and the particle, composite, and matrix are denoted by the subscripts p , c and m , respectively.

Comparably in Eq. 2:

$$w_p = \frac{W_p}{W_c} \text{ and } w_m = \frac{W_m}{W_c} \quad (2)$$

where w_p and w_m are the weight fractions of particle and matrix respectively, while w_c is the weight of the constituent under consideration, taking into cognizance the subscripts.

A correlation between the weight and volume fractions is shown by bringing forth the density (ρ) of the composite and its components, as shown in Eq. 3 [99].

$$\rho_c = \rho_p v_p + \rho_m v_m \quad (3)$$

The weight fractions of the particle w_p and the matrix w_m , are as shown in Eq. 4 to Eq. 5. [36].

$$w_p = \frac{W_p}{W_c} = \frac{\rho_p V_p}{\rho_c V_c} = \left(\frac{\rho_p}{\rho_c} v_p \right) \quad (4)$$

$$w_m = \frac{W_m}{W_c} = \frac{\rho_m V_m}{\rho_c V_c} = \left(\frac{\rho_m}{\rho_c} v_m \right) \quad (5)$$

At any composite strain, ε_c the preceding fracture, the stresses in the matrix (σ_m) and particle (σ_p) are obtainable, as shown in Eq. 6 to Eq. 7.

$$\sigma_m = E_m \varepsilon_c \quad (6)$$

$$\sigma_p = E_p \varepsilon_c \quad (7)$$

where E_m and E_p represents Youngs moduli of matrix and particle, respectively, while the stress of the composite (σ_c) is given by Eq. 8.

$$\sigma_c = \sigma_p v_p + \sigma_m (1 - v_p) \quad (8)$$

and the components' axial Youngs Modulus is given by Eq. 9.

$$E_c = E_p v_p + E_m (1 - v_p) \quad (9)$$

Development and Performance of Periwinkle/Palm Kernel Shell Agro-waste Reinforced Composites for Brake Pad Production

Many researchers have examined agro waste materials for brake pad application. Anaidhuno et al., [116] utilized the palm seed, coconut and palm kernel shells as constituents in a composite to produce brake linings. Their result showed improved wear resistance from the wear test result of between 0.025mm/min–0.06mm/min. Rajmohan et al., [117] also used the followings: coconut shells, sugar cane, and snail shells as composite materials to develop brake pad linings. Their findings showed an improved wear resistance, resulting from a wear test result, of between 35.5×10^{-5} mg/m– 41.3×10^{-5} mg/m. Amaren et al., [81] examined how the size of periwinkle shell particles affected the asbestos-free brake pad's wear behaviour. Their findings revealed that particles of periwinkle shell can successfully, substitute asbestos in brake pad manufacture because the coefficient of friction obtained in their investigation, fell within the permissible range for automobile brake pads. Afolabi et al., [118] investigated the production of brake pads by utilizing polymer composites reinforced with cow bone and PKS. The study found that both materials exhibited sufficient thermal stability to withstand typical braking temperatures without decomposition. Additionally, hardness tests revealed values of 46 HRB for the cow bone and 55.7 HRB for the PKS, which are comparable to those of conventional materials currently in use. Dagwa et al., [119] characterized powdered PKS for use in composites made of a polymer matrix. Their findings revealed that the properties exhibited by palm kernel shell, encouraged its usage as a friction material. Those properties, include density (1.58 ± 0.07 g/cm³), porosity (6.76 ± 0.42 %) and moisture content (11.16 ± 0.16 %). Anshuman and Maurya [120] carried out the mechanical analysis of an epoxy-based composite made from biowaste. Their findings indicated that the composite showed excellent abrasion and wear resistance as the hardness increased from 13 Hv to 35 Hv.

In a study conducted by Deepika et al., [113], the development and performance assessment of a composite material for wear-resistant applications was explored. PKS was used as a filler, in combination with sulphur and other conventional brake pad constituents, including calcium carbonate, quartz, iron ore, brass chips, ceramics, cashew nut-shell liquid (CNSL), and carbon black. The PKS filler was pulverized to a particle size of 125 µm. The results demonstrated that PKS exhibited performance characteristics comparable to those of asbestos-based brake linings under a range of speed and inertial conditions.

Fono-Tamo and Koya [9] developed advanced automotive brake pad material, following the normal practices involving palm kernel shell (PKS) in achieving quality properties that are comparable to those of the commercially produced brake pads. Their results showed a shear strength of 40.95 MPa, a hardness of 32.34 MPa, and a notable friction coefficient of ~0.43. These values fall within the acceptable range proposed by Roubíček et al., [12], who suggested that the friction coefficient for brake pad materials should lie between 0.30 and 0.70.

The potential of PKS agro-waste as a substitute for asbestos in brake pad production was effectively, demonstrated by Ibadode and Dagwa [112]. By using the Taguchi optimization technique, they identified the optimal process parameters, including a moulding temperature of between 150–170 °C, pressure of between 16.74–27.90 MPa, curing times of between 6–10 minutes, and heat treatment durations of between 1–3 hours. The material formulation comprised of 56% reinforcement, 14% abrasives, 24% binder, and 6% friction modifier. The fabricated brake pad was subjected to performance testing, yielding results that are comparable to those of the traditional asbestos-based pads. The measured surface hardness ranged from 64 to 89 HRB, with a coefficient of friction of between 0.35 and 0.44. The wear rates fell into the range of between 0.017 and 0.170, that is consistent with increased wear at speeds above 80 km/h. These

findings confirm the effectiveness of PKS-based pads as a sustainable and efficient alternative to the asbestos-based brake materials.

Elakhame et al., [121] explored the use of PKS at varying particle sizes of 100 μm , 350 μm , 710 μm , and 1 mm for the development of asbestos-free brake pads. The brake pad formulations included the followings: between 35%–55% PKS, 20% resin, 10% graphite, 15% steel, and ~20% silicon carbide (SiC), and processed by using the compression moulding technique. Major properties, such as: hardness, density, compressive strength, flame resistance, and fluid absorption were evaluated. Among the tested samples across multiple metrics, the formulation with 100 μm PKS demonstrated the most favorable results. Microstructural analysis confirmed a uniform dispersion of the resin used, indicating improved interfacial bonding, at small particle sizes. When compared to conventional asbestos-based pads, the PKS-based variants exhibited a highly competitive performance, highlighting their potential as eco-friendly, cost effective and efficient alternatives in brake pad production.

Mgbemena et al., [114] developed an asbestos-free friction lining for automotive brake pads by using ground palm kernel shell (PKS) as the primary filler and metallic cutting shavings from workshops as abrasives. Phenolic and alkyd resins were utilized as binders in the formulation. The fabricated samples were subjected to physical, thermal Optical, and tribological tests, which were conducted by using a Simultaneous Thermal Analyzer and Stereomicroscopy (ZEISS). When compared with commercial brake linings, manufactured by the original equipment manufacturers (OEMs), the PKS-based material showed a slightly higher wear rate of 0.24 μm , in contrast to the 0.16 μm observed in the OEM-produced samples. Despite this, the results demonstrated the fact that the PKS-based friction linings possessed promising characteristics for application in eco-friendly, asbestos-free brake pads. However, the PKS-based lining demonstrated enhanced thermal stability and a greater pulverized yield, hence, indicating its resilience under elevated temperature conditions. These findings suggest that, despite the higher wear rate, the PKS-based formulations offered good potential as thermally robust alternatives to the asbestos-containing brake linings.

Singh, et al., [122] studied the absorption characteristics and mechanical properties of coconut shell powder-epoxy composite. Their findings indicated that the composite made with between 20-30% coconut shell powder occupied volume fraction, finds application in the automobile's interior because of its improved strength and low density. Ofem and Umar, [123] researched into how filler the content affected the mechanical properties of CNSL resin composite reinforced with periwinkle shells. Their results showed maximum flexural and tensile strengths at 30% filler content and 400 μm particle sizes, while the maximum impact strength and tensile modulus were observed at an 800 μm particle size with 30% and 40% filler contents, respectively.

Egeonu et al., [124] utilized locally sourced palm kernels and coconut shells to develop high quality asbestos-free disc brake friction lining material that met the Mitsubishi L-300 geometrical specifications. A brake pad that was purchased commercially, served as the control. A trio of separate samples, coded: A, B and C were developed by varying the mass compositions of coconut and palm kernel shells. The result obtained from all the three different samples, compared favourably with the commercially purchased brake pad.

Aku et al., [101] conducted out an in-depth investigation into the suitability of periwinkle shell as an asbestos-free alternative in brake pad production, through both spectroscopic and wear analysis techniques. The study incorporated analytical tools, such as X-ray diffraction (XRD), thermogravimetric/differential thermal analysis (TGA/DTA), Fourier-transform infrared spectroscopy (FTIR), and X-ray fluorescence spectroscopy (XRF), to characterize the material properties. Major properties, such as density, hardness, and wear rate were measured and compared with those of conventional asbestos-based brake pad materials. The findings confirmed the fact that periwinkle shell exhibits favorable mechanical and thermal characteristics, making it a viable candidate for brake pad applications. Furthermore, Obota et al., [125] carried out a comparative study between the different types of abrasive sandpaper that were produced by using

palm kernel and periwinkle shells that were locally sourced. This was done to assess their potentials as replacements for imported abrasives sandpaper. Their results revealed that the mechanical and physical properties obtained, were closely at par to the acceptable standards when compared to the imported sandpapers.

Yawas et al., [126] examined the morphological and mechanical properties of asbestos-free brake pads, reinforced with periwinkle shell particles, focusing on the influence of particle size. Their findings revealed that by reducing the particle sizes to between 710 μm to 125 μm , led to notable improvements in the asbestos-contained brake pad compressive strength, hardness, and density. This enhancement in the mechanical properties with finer particles, underscores the capability of periwinkle shell as an effective reinforcement material. The study supports the effectiveness of periwinkle shell as a sustainable and efficient substitute to asbestos, in the production of high-performance brake pads. In the same vein, Yakubu et al., [127] designed an asbestos-free brake pad using periwinkle shell particles (PSP) as reinforcement and thermoset resin as a binder and investigated how different PSP particle sizes (710 μm -125 μm) affected the brake pad performance. The study found that smaller particle sizes enhanced the interfacial bonding between the PSP and the resin matrix, thereby resulting in a better material integrity. While wear rates increased with higher loads and coarser particles, the friction coefficient remained within the acceptable range for standard brake pads. Moreover, the PSP demonstrated a higher decomposition temperature than asbestos, hence, indicating a superior thermal resistance. These findings highlight its potential as an environmentally sustainable and effective replacement for asbestos in brake pad manufacturing. Furthermore, Iwalola et al., [128], Mawuli et al., [129], and Uzochukwu et al., [130], each conducted independent studies on the development of asbestos-free brake pads by using periwinkle shell particles as reinforcement. Their findings consistently confirmed the fact that periwinkle shell is a viable and effective substitute for asbestos, by offering favorable mechanical and thermal properties that are desirably suitable for brake pad applications.

Current Research Gaps in Periwinkle/Palm Kernel Shell Epoxy Reinforced Composites

Despite significant progress in the development of periwinkle shell (PS) and palm kernel shell (PKS) reinforced epoxy matrix hybrid composites for brake pad applications, several critical research gaps do persist. A thorough understanding of their long-term operation under real-life operating conditions, such as thermal cycling and high-speed braking alternatives are still lacking. The interfacial bonding behavior between these natural fillers and the epoxy matrix, also needs deeper exploration to improve durability and mechanical properties. Moreover, there are insufficient modeling and optimization scenarios of multi-filler systems and resin compositions, particularly under varying environmental influences, e.g., humidity and temperature. Standardized testing protocols and consistent benchmarking against the commercial brake pads remain underdeveloped, thereby underscoring the need for uniform evaluation methods and regulatory validation.

Future Trends for Periwinkle/Palm Kernel Shell Epoxy Reinforced Composites

The growing need for sustainable, high-performance brake pad materials has driven major interest in hybrid composites reinforced with agro waste, such as periwinkle shell (PS) and PKS. Future research in this area is expected to focus on the nano-engineering of the reinforcements employed, surface modification techniques, and hybridization with other natural or industrial fillers to enhance the composites mechanical and tribological properties. Advances in epoxy matrix systems, particularly through toughening or bio-based resin modifications, will play a key role in improving the composites thermal and structural stability. The use of advanced manufacturing techniques, e.g., hot pressing and additive manufacturing, combined with

computational tools, such as finite element analysis and AI-based modelling, will streamline the optimization of composite formulations. Additionally, efforts should be made to integrate thermally conductive and vibration-damping additives for better heat dissipation and operational safety. Emphasis on life-cycle assessment and cost-performance analysis will also guide the sustainable commercialization of these asbestos-free alternatives. These research trends promise to usher-in a new generation of eco-friendly, efficient, and durable brake pad materials.

Conclusion

The development of periwinkle shell (PS) and palm kernel shell (PKS) reinforced epoxy matrix hybrid composites offers a sustainable and performance-oriented alternative to asbestos-based brake pads. Composites produced with particle sizes between 100–125 μm exhibited the most favorable mechanical and tribological properties, including improved hardness and wear resistance. This particle size range was identified as optimal for interfacial bonding.

The hybrid composite brake pads demonstrated hardness values up to 75 HRC for PS and 55.7 HRB for PKS and compressive strength between 32–41 MPa. The coefficient of friction (COF) was within the acceptable range of 0.35–0.44, satisfying automotive performance requirements.

Thermal analysis revealed that PS maintains structural integrity above 600 °C, while PKS exhibits thermal decomposition between 54–538 °C, indicating their suitability for braking systems subjected to moderate-to-high temperatures.

Future research should focus on long-term durability testing under real-world conditions, finite element modeling for stress distribution, and the incorporation of nano-fillers for improved thermal dissipation.

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