Effect of silica on the mechanical properties of palm kernel shell based automotive brake pad

Olayide R. Adetunji, Ayodele M. Adedayo, Salami O. Ismailia, Olawale U. Dairo, Iliyas K. Okediran, Olanrewaju M. Adesusi

1 Department of Mechanical Engineering, Federal University of Agriculture, Abeokuta, P.M.B.2240, Abeokuta,110010, Nigeria
2 Department of Agricultural and Bio-resources Engineering, Federal University of Agriculture, Abeokuta, P.M.B.2240, Abeokuta,110010, Nigeria
3 Department of Mechanical Engineering, Osun State University, Osogbo, Oke Bale Street, Osogbo, 210001, Nigeria

This article contributes to:

Highlights:
• Inclusion of silica and steel slag in palm kernel shell brake pad composite materials will lower cost and improve performance.
• Lower silica content produced low flame resistance, water and oil absorption with increased hardness, compressive strength and wear resistance.
• Palm kernel shell particle size increase led to decreased hardness, compressive strength and coefficient of friction while oil and water absorption increased.
• Heavy metals that present health risk are absent in the developed brake pad.

Abstract
This research investigated the role of silica on palm kernel shell (PKS) as friction lining materials in automotive brake pad production. The friction materials were crushed, milled and sieved into four different particle sizes of 100, 150, 200 and 350 µm. The formulations weight percentages employed included Resin (20%), steel slag (15%) and carbon black (5%) while palm kernel shell and silica were varied for each particle size. Individual formulation was mixed for about 10 minutes until formation of homogeneous mixture. Homogeneous formulation A, B, C and D respectively, was compacted into mould and later sintered at 150 °C for 10 minutes in electric furnace and subsequently treated to enhance quality. Produced samples were characterized and evaluated for surface hardness (SH), compressive strength (CS), flame resistance (FR), oil absorption (OA), water absorption (WA) and wear rate (WR). The particles were also characterized using Scanning Electron Microscope. The results revealed that sample D had highest SH and CS values of 105.5 Brinell hardness number (BHN) and 115.2 N/mm² respectively with decreasing values as particle size increases. FR decreased from samples A to D, and also decreased as particle size increased. Deductively, Sample B with the sieved grade of 100 µm was the best with SH as 99.14 BHN, CS as 105.6 N/mm², WR as 4.15%, FR as 4.15%, and WA rate as 4.26% and CF as 0.45 and OA rate as 0.381%. Conclusively, this research developed a high quality eco-friendly PKS particle composite for the production of brake pad.

Keywords: Palm kernel shell, Silica, Friction, Surface hardness, Wear rate

1. Introduction

The quest for increasing local content of commercially available automotive spare parts coupled with the need for environmentally friendly products in Nigeria necessitate local sourcing of the parts such as brake pad from locally available waste materials. Brake pads are important parts of braking system for all types of vehicles that are equipped with disc brake [1]. Brake pads
are steel backing plates with friction material bound to the surface facing the brake disc. Different types of brake materials are used in different machines [2-3]. Although the use of asbestos for brake pads has not been banned completely, much of the brake pad industry is moving away from asbestos brake pads because of concerns regarding airborne particles in the factories, roads and disposal of wastes containing asbestos [4]. The significance of the brake pad is to transform the kinetic energy of a vehicle to heat energy via friction and ejecting the heat to the surrounding environment [5-6].

Composite brake pad constituents are often categorized into the following classes of ingredients; binders, fillers, friction modifiers, abrasive and reinforcement [7]. Selection of the constituents is often based on experience or trials and error method to make a new formulation. Some researches had been carried out in the area of development of asbestos-free brake pads [8-9]. The uses of bagasse and coconut shell have been investigated. Palm kernel shell is readily available in large quantities with little or no cost and is not toxic. When mixed with other constituents, it improves tensile strength, impact strength, hardness, compressive strength, low wear rate and low porosity [10]. However, from all available literature and expert opinion, Palm Kernel Shell (PKS) which has been handled for centuries especially in southern Nigeria up till date is not reported to have shown carcinogenic effects and its coefficient of friction is 0.41 which falls within the range 0.37-0.70 of friction material [11]. Coefficients of friction of PKS on metal surfaces were in the range of 0.37-0.52 [12-13] which led to lower wear rate and high friction of brake pad. In contrast, friction coefficient in the range of 0.30-0.70 is normally desirable when using brake lining material [14].

Palm kernel shell is recovered as by-product in palm oil production. The shell thickness is the basis of their classification. There are three types of shells namely Dura (5-8 mm thick), Tenera (0.5-4 mm thick) and Pilfer (0.1-0.5 mm thick). The dura has thick shells surrounded by a fibre ring, large kernel and small proportion (20-40%) of the oil bearing mesocarp. The Tenera has thin shell specially been developed to yield high oil content. However, the oil palm exists in nature predominantly as the dura fruit form which constitutes over 95% of the world’s population. The pilfer is essentially female sterile thus having very little yield and occurs naturally at very low frequency of less than 1% as reported by Koya et al. [12]. Owning to these benefits associated with PKS, it is a viable filler material for automotive brake pad production.

The use of clay as filler material, resin for curing were studied by Choa et al. [15]. Steel slag, silica sand and activated carbon involvement in the brake pad composition were reported by Meng et al., Ren et al., Chen et al., and Gao et al. [16-19]. The PKS and other agricultural waste were used to replace asbestos in brake pad production. Such researchers included Leachate et al., Lawal et al., Idris et al. and Yawas et al. [20-23]. Chinedu [24] used coal ash with PKS to make brake pad. Fono-Tamo and Koya [25] reported poor fade features for non-asbestos organic materials as coefficient of friction (CoF) dropped below 0.1 but later experienced recovery to desired value with 0.3 mm particle size having best friction, friction fade, wear and mechanical performances. It was reported in the work of Soundararajan et al. [26] that Jute fibre and steel fibre reinforced polymer hybrid composite has high strength, increased friction coefficient and wear rate due to significant heat resistance and dissipation, reduced noise and excellent braking performance. The work of Achebe [27] show that palm kernel fibre content for brake pad production had water and oil adsorption decreased in comparison to results obtained from other researches where brake pads were prepared from other friction materials and asbestos. Ossia et al. [28] discovered that decreased particle size of coconut shell as substitute for asbestos brake pad production resulted in improvement in tested properties except thermal and conductivity stability. The 100 μm particle size as reported by Elakhame et al. [29], gave best properties for their formulated PKS based brake pad with values decreasing as the particle size increased to 1 mm. Anaidhuno et al. [30] revealed that decrease in bonding strength and hardness occurred as a result of increased grit size of PKS and coconut shell-based brake pad. Obtained results from the reported work of Afolabi et al. [31] showed that their formulated brake pad composition from PKS and cow bone were of poor percentage water and oil absorption capabilities which well above standard.

The quest for technological advancement, high cost of imported brake pads and poor quality of produced local brake pads has been the major concern in Nigeria asides the carcinogenic nature of the so-called asbestos brake pad. Much research is always carried out to find new and better lining materials and efforts are now being made into the design and manufacture of asbestos-free brake linings. This work therefore developed automobile brake pad from pulverized PKS, silica and steel slag composite.
2. Materials and Method

The main materials for this research work are; Palm kernel shell particle (PKS) as base material, steel slag, silica sand, carbon black and phenolic resin as binder. Equipment and materials used in the workshop and laboratory are crushing machine, engine mill, sieve of sizes 100 µm, 150 µm, 200 µm and 350 µm, weighing scale, electric furnace, electric oven, digital balance, cure mould, hydraulic press, Brinell hardness tester, compressive strength machine, universal material tester, polishing machine, scanning electron microscope, micrometre screw gauge. Other materials and substances used for this research are wire gauze, Bunsen burner, silicon carbide paper of grade 320, distilled water, SAE 20/50 grade oil and tripod stand.

The cracked PKS were thoroughly washed in warmed water with small quantity of detergent to remove impurities or residual oil in the shells which is rich in cellulose and lignin. Thereafter, all the cracked shells were sun dried for about 72 hours. About 20 kg quantities of the dried shells were pulverized into powder using crushing machine having electric motor power of 1.5/1.7 kW, which reduced the PKS sizes and subsequently grounded into fine particles with engine mill having an electric motor capacity of 0.75 kW. They were later sieved into different particle sizes of 100 µm, 150 µm, 200 µm and 350 µm according to the method used by Yawas et al. [23].

Table 1. Formulated compositions of the brake pad

| Raw materials         | Samples’ nominal composition (%) |
|-----------------------|----------------------------------|
|                       | A   | B   | C   | D   |
| Palm kernel shell     | 45  | 50  | 55  | 60  |
| Steel slag            | 15  | 15  | 15  | 15  |
| Silica sand           | 15  | 10  | 5   | 0   |
| Carbon black          | 5   | 5   | 5   | 5   |
| Phenolic resin        | 20  | 20  | 20  | 20  |
| Total                 | 100 | 100 | 100 | 100 |

Each formulation was mixed for about 10 minutes until a homogeneous mixture was formed. Thereafter the mixed composite samples were compacted into a cylindrical mould chambers of 29.44 mm diameter under reference until the shape was formed and later sintered (subjected to heat) at 150 °C for about 10 minutes inside an electric furnace model GEMCO furnace REX-900 type CFR with capacity 40 kW working at temperature 1200 °C where curing process took place. It was then hot pressed immediately after being removed from the furnace using hydraulic press model HMS 060 to a certain pressure for about 30 to 45 seconds of pressing which induced curing at that pressure and temperature. This procedure described above was repeated for all the experimental sets.

The produced samples with different formulations and particles sizes were characterized and subjected to mechanical, physical and wear examination. The best sample of the formulations was later used to produce the brake pads. Steel backing plate of pathfinder jeep was selected, surface smoothened, smeared with binder to ensure proper sticking of lining to the backing plate and positioned into the mould. The mixture for the brake pad was compacted on backing plate inside the fabricated mould cavity of pathfinder jeep until the shape was formed and later subjected to heat at 150 °C for about 10 minutes inside an electric furnace where curing process took place. It was hot pressed immediately after being removed from the furnace using hydraulic press to a certain pressure with about 30 to 45 seconds of pressing which induced curing at that pressure and temperature. It was re-sintered for another 10 minutes, removed from furnace and hot pressed second time.

The brake pad was later removed and the edges trimmed to a desirable shape and subsequently allowed cool naturally for about 3 days. The finishing of the brake pad, including grinding and smoothening was carried out to a standard size of 16 mm thickness and all resinous layers were removed using emery cloth made of silicon carbide. The brake pad was then tempered at a temperature 25 °C for about ten minutes to remove the residual stresses induced during finishing process. This process and procedure described above was repeated for all the experimental sets. The produced samples were examined for Brinell hardness test using a universal tester model Gunt WP 300 with maximum test force 20 kN and compressive test using equipment of BS240, a Tensometer M500-25 kN, wear resistance test using a polishing machine model DAP-2
struers with variable speed 0-250 rpm, coefficient of friction test using an incline plane apparatus model 12558, flame resistance, water absorption and oil absorption. SEM were studied using a TESCAN JEOL JEM-7600F scanning electron microscope (SEM), operated at an accelerating voltage of 5-20 kV.

The wear rate experiment was accomplished by consistently pressing the samples against a pin on disc rotating machine (silicon carbide paper) of grade 800 fixed on the rotating polishing machine of 180 mm diameter at a constant speed 125 rpm for 2 minutes, under a load of 10 N through a sliding distance of 2000 m. The initial weight of the samples was measured using a single pan electronic weighing machine with an accuracy of 0.01 g. During the test, the pin was pressed against the counterpart rotating cast iron disc of Rockwell hardness 65 HRC and counter surface roughness 0.3μm, by applying the load. A friction detecting arm connected to a strain gauge held and loaded the pin samples vertically into the rotating hardened cast iron disc. After running through a fixed sliding distance, the samples were removed, cleaned with acetone, dried, and weighed to determine the weight loss due to wear. The difference in weight measured before and after the tests give the wear of the samples and the wear rate was calculated using Equation 1.

\[
\text{Wear rate} = \frac{\Delta W}{S} = \frac{\Delta W}{2\pi NDt} \left( \frac{g}{m} \right)
\]

Where, \(\Delta W\) = weight loss (weight difference before and after wear); \(S\) = sliding distance; \(D\) = diameter of the disc; \(N\) = rpm; \(t\) = time it takes each sample (exposed) on grinding distance.

The coefficient of friction between the linings and steel was determined using a steel inclined plane. The plane was kept at 180° (horizontal position). Each sample A, B, C and D of 100 µm particle sizes were attached to a string and placed on the plane. The string was passed through a pulley, which was connected to a mass hanger. Standard masses 23 g, 24 g, 22 g and 21 g for 100 µm formulations A, B, C and D respectively were added to the mass hanger until the lining began to slide along the surface of the steel plane. The coefficient of friction is the ratio of frictional force (equivalent to mass at hanger to initiate sliding with the stated masses value above) to the normal reaction (weight of brake lining which are 55.78 g, 55.65 g, 55.13 g and 55.13 g for 100 µm formulations A, B, C and D, respectively).

Equation 2 below was used to calculate the coefficient of friction (\(\mu\)) between the linings and steel.

\[
\mu = \frac{F_r}{F_n}
\]

Where, \(F_r\) = friction force (N); \(F_n\) = normal reaction (N).

3. Results and Discussion

The formulated samples were grouped into A, B, C and D respectively for the purpose of comparison on the test carried out. PKS and silica sand are the only materials varied while others were kept constant throughout the formulation. All the samples A, B, C and D with different particle sizes 100 µm, 150 µm, 200 µm and 350 µm were subjected to physical, chemical, tribological and mechanical tests as shown in Table 2 to Table 7.

3.1. Results of physical, mechanical, chemical and tribological tests

The physical, mechanical, chemical and tribological tests carried out on the samples are hardness surface, compressive strength; wear test, flame resistance, oil and water absorption test.

3.2. Brinell hardness, compressive strength, and wear resistance

Brinell values of four hardness tested samples were recorded. The results clearly showed the comparison on the effect of the surface hardness on samples as shown in Table 2. Generally, PKS concentration increased from sample A to D which corresponded to increase in Brinell hardness values from 79.55 to 105.50 BHN where sample D has the highest while considering particle size 100 µm. These values decreased with increase in particle size for each sample A, B, C and D respectively. Similarly, as concentration of PKS increased from sample A to D, compressive strength and wear rate resistance increased as shown in Figure 1 and Figure 2 while both parameters decre-
steel slag, 20% resin and 5% carbon black with the finest sieve grade size 100 µm PKS, produced best result. The BHN of 99.14 is not too hard to damage the rotor disk of the brake pad. This is followed by the particle size of sample B with 150 and 200 µm having hardness values of 95.48 BHN and 88.65 BHN respectively. Mathur et al. [1] and Dagwa [2] made use of medium hardness and low wear rate properties of ideal automotive brake pad. Formulation A with surface hardness of 79.55 BHN is the lowest which could lead to short life span of the pad and make the pad to become indented easily while formulation C and D results of 103.5 BHN and 105.5 BHN respectively are too hard which may damage the rotor disc of the brake pad. The Brinell hardness of 100 µm sieve grade in sample B suggested that PKS of 50% proportion with silica 10% sand and steel slag 15% all play a role in determining the friction characteristics of the sample.

Compressive strength test results are presented in Figure 1. Sample D had the highest strength of 115.2 N/mm² which also decreased as particle size increased. Wear test results are contained in Figure 2. Sample D had the highest wear resistance which reduced as particle size increased. Figure 1 shows the variation of compressive strength values of each formulation with sieve grade 100, 150, 200, 350 µm which indicates that the formulation B, C and D of particle size 100 µm are better with values of 105.6, 108.5, and 115.2 N/mm² while formulation A of 92.55 N/mm² is relatively low. The 100 µm size grade had the best compressive strength of 105.6 N/mm². The gradual decrease in compressive strength as the size increased can be attributed to the decreasing surface area and pore packaging capability of the PKS particles in the resin.

Wear test results of the produced samples A, B, C and D in Figure 2 showed increase in wear rate as the sieve grade sizes increased. This could be as a result of higher/closer packing which affected stronger binding of PKS, silica and steel slag within the compositions. This may also be due to high hardness values and compressive strength of the samples B, C and D as sieve size decreased. Considering the particle size 100, formulation D of 3.92% had the best wear resistance followed by C and B of 4.12% and 4.15% respectively but sample A had the highest wear rate of 6.25% which could shorten the life span of the pad. Since sample C and D have failed in surface hardness test, then formulation B is being considered as the most preferred. Earlier researchers (Ibhadohe and Dagwa [10] and Koya et al. [12]) agreed in their reports that toughness, low wear, and porosity are essential factors in selecting good automotive brake pad. It is used to determine the life span of the brake pads and as a major determinant that could be used as a prototype factor during production of the pads.

| Grade size (µm) | Load (kg) | Area (mm²) | Brinell Hardness (BHN) | A | B | C | D |
|-----------------|-----------|------------|------------------------|---|---|---|---|
| 100             | 3000      | 680        | 79.55                  | 99.14 | 103.50 | 105.50 |
| 150             | 3000      | 680        | 72.48                  | 95.48 | 101.62 | 102.48 |
| 200             | 3000      | 680        | 64.32                  | 88.65 | 97.18 | 99.39 |
| 350             | 3000      | 680        | 46.25                  | 59.68 | 69.35 | 72.50 |

Figure 1. Sample compressive stress versus particle size

Figure 2. Sample wear rate versus particle size

Table 2. Brinell hardness values of samples
3.3. Flame resistance and coefficient of friction

Flame resistance test results shown in Table 3 with sample A having the lowest resistance which increased from sample A to D and increased as particle size increased. It revealed that formulations A and B have the best flame resistance of 32.56% and 38.98% respectively. The result is due to high silica and steel slag weights in the compositions which made them resist heat and also of high abrasiveness. In the case of sample C, it is 45.57% and D is 66.51% which is considered to be high. This could be as a result of high percentage of PKS 60%, 15% steel slag but 0% of silica sand which has negative effect on the heat resistance. The properties increased as the sieve grades increased which can eventually be attributed to the increased pores as the sieve size increased. It can also be observed from the results that samples with 100 µm gave best properties because of a very good dispersion of decreased particles size.

Table 4 shows the sieve grade 100, 150, 200 and 350 µm for the values of coefficient of friction for formulation A, B, C and D with highest values of 0.41, 0.44, 0.39 and 0.38 respectively obtained at 100 µm. The table indicates that the sieve grade size 100 for formulation A and B gave the best property in terms of coefficient of friction. This coefficient of friction property reduced as the sieve grade increased which can eventually be attributed to the increase pore size as sieve grade size increased with sample B having the highest coefficient of friction 0.44. Each sample coefficient of friction decrease as the particle size increased. The values vary from 0.36 to 0.44 for sample B.

The coefficients of friction of all the samples fall within the SAE class F industrial standard ranges of 0.30 - 0.45 for automotive brake linings system as reported by Idris et al. [22].

3.4. Oil and water absorption

The oil and water absorption result are presented in Table 5 and Table 6 with sample D having the least value. The developed brake pad was observed to have experienced increase in oil and water absorption with increasing grain size, irrespective of the ratio of weight percentage of the other constituents of the brake pad.

Table 5 shows the sieve grade 100, 150, 200 and 350 µm which indicated that the sieve grade size for formulation B, C and D gave the best property in terms of oil absorption with absorption rate of 0.381%, 0.365% and 0.312% respectively, compared with sample A of 0.423%. This property increased as the sieve grade increased which can eventually be attributed to the increased pores as sieve size increased [26-27].

Table 6 also shows the sieve grade 100, 150, 200 and 350 µm which indicated that the sieve grade size for formulation B, C and D gave the best property in terms of water absorption rate of 4.26%, 4.19% and 3.72% respectively, which absorbed the least water unlike sample A of 6.67% considered to be the highest. The water absorption rate of the developed brake pad was observed to increase with increasing grain size irrespective of the ratio of weight percentage of the other constituents of the brake pad. As a result, 100 µm grade sizes of the PKS produced the least water absorption capacity. With increasing grain size
size 100 to 350 μm and at the least 45% wt. composition of PKS, 15% silica and 15% steel slag, the porosity to water after 24 hrs was 6.67% in sample A.

3.5. Scanning electron microscopy test

The microstructural test carried out on sample B with 100 μm particle size to determine the morphology and elemental composition of the lining are shown in Figure 3. It is clear that before and after the wear, the EDX in selected samples revealed no traces of heavy metals like copper dust, lead, mercury, arsenic, cadmium and chromium which are of high degree of toxicity and health risk as compared to the existing PKS-based brake pads which contained traces of copper. From the SEM and EDX result, it was discovered that the microstructure of 100 μm of sample B as shown in Figure 3 is more homogenous, hard to differentiate between white region of PKS and other constituents from the dark region of the resin and carbon black which made it give a better interfacial bonding between the PKS, silica, steel slag particles and the resin resulting into closer inter-packing surface. The EDX analysis in 100 μm of sample B shows about 70% high content of aluminium but low content in other constituent elements.

3.6. Appraisal of the developed pks-silica and steel slag brake pad with others

Comparison of the physical and mechanical properties of the developed PKS-silica and steel slag brake pad with existing brake pads in literature is shown in Table 7. The wear rate of the newly developed PKS-silica brake pad shows improved wear rate, compressive strength, water absorption rate and hardness compared to existing PKS, other laboratory developed brake pads and commercial brake pad and in addition, the constituents of the developed PKS-silica brake pads have no adverse effect on the environment and pose no health risks as compared to asbestos-based brake pad. The best particle size obtained from the work of Yawas et al. [23] and Chinedu [24] was 125 μm while this research achieved 100 μm, tending towards nano size which enhanced quality performance. Aside that, with the composition of PKS which is half of the lining constituent materials, including silica and steel slag of relative weights will reduce its cost and improve the quality compare to the existing local PKS based pads in the market having up to eight (8) lining materials. As a matter of fact, the eco-friendly nature of PKS has tremendously encouraged its usage in many areas such as asphalt stabilization in road and construction works, and so on. However, steel slag with less percentage composition of vanadium is encouraged for the development of brake pads in order to prevent the formation of toxic pentavalent oxide form of vanadium (V_2O_5) in the environment.
Table 7. Comparison of new formulated laboratory brake pad with the existing brake pads

| Property                              | New formulated brake pad (PKS-Silica based) [This work] | Commercial brake pad (asbestos based) [5, 10, 22] | Formulated brake pad (PKS based) [10, 22] | Formulated brake pad (banana peels based) [22] | Formulated brake pad (bagasse based) [5, 22, 23] |
|---------------------------------------|---------------------------------------------------------|-------------------------------------------------|------------------------------------------|-----------------------------------------------|-----------------------------------------------|
| Brinell Hardness (at 3000 kg)         | 99.14                                                   | 101                                             | 92                                       | 98.8                                          | 100.5                                         |
| Compressive strength (N/mm²)          | 105.6                                                   | 110                                             | 103.5                                    | 95.6                                          | 105.6                                         |
| Average wear (%)                      | 4.15                                                    | 3.80                                            | 4.40                                     | 4.15                                          | 4.20                                          |
| Flame resistance test after 10 minutes (%) | Charred with 39% ash                                | Charred with 9% ash                             | Charred with 46% ash                     | Charred with 24.67% ash                       | Charred with 34% ash                         |
| Coefficient of friction               | 0.44                                                    | 0.40                                            | 0.44                                     | 0.40                                          | 0.42                                          |
| Thickness swell in water after 24hrs (%) | 4.26                                                  | 0.90                                            | 5.03                                     | 3.21                                          | 3.48                                          |
| Thickness swell in oil (SAE 20/50) after 24hrs (%) | 0.38                                                  | 0.30                                            | 0.44                                     | 1.15                                          | 1.11                                          |

4. Conclusion

This research investigated the role of palm kernel shell (PKS) and silica as friction lining materials among steel slag and phenolic resin particles in automotive brake pad production. Hence, this work reached conclusions that the higher the PKS and the lower the silica sand concentrations of the formulated brake pads, the higher the brinell hardness, compressive strength and wear rate while flame resistance, water and oil absorption decreased. PKS, silica sand, steel slag, carbon black and phenolic resin with composition 50%, 10%, 15%, 5% and 20% respectively in formulation B showed the best mechanical property with the lowest particle size 100 µm as compared to others previously obtained. Hardness, compressive strength and wear resistance decreased with increase in particle size, while, flame resistance, oil and water absorption increased with increase in particle size. The coefficient of friction decreased as the particle size increased, the highest coefficient of friction with 100 µm of 0.44 was established. Scanning electron microscope structures of the produced brake pads were analyzed and found to conform to the standard as they show a uniform distribution of resin and the lining materials. The energy dispersive X-ray showed no traces of heavy metal and contain the essential elements such as Al, Fe, Na, O and C among others which are the predominant elements of a good brake pad.

5. Recommendations

Based on the results obtained from this research work, it is hereby recommended that the Nigerian government should invest massively in the production of automotive brake pad using composite from palm kernel shell base material, silica sand and slag in order to reduce importation of asbestos brake pads, create employment and diversion of steel slag from landfill.

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Authors’ Declaration

Authors’ contributions and responsibilities - The authors made substantial contributions to the conception and design of the study. The authors took responsibility for data analysis, interpretation, and discussion of results. The authors read and approved the final manuscript.

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