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Recycled ceramic tile composite for automobile applications, a comparative study with Nissan Jeep Cherokee brake pad

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Abstract

A ceramic tile/steel slag-graphite-Arabic gum composite was developed using conventional casting techniques for brake pad applications. The chemical properties of the phases present in the matrix of the developed composite were examined using X-ray diffractometry. Spatial configuration of the phases was viewed using scanning electron microscopy. Wear and thermal properties of the developed composite were also investigated. Comparisons of the properties of the developed composite and Nissan Jeep Cherokee brake pads were made. The results showed a partial homogeneity of second phase particles within a ceramic matrix. The developed composite brake pad showed better friction properties than commercial grade brake pads for up to 200 s. At longer times, the opposite was observed. The developed composite brake displayed lower wear rates (1.3×10^{-8}, 1.8×10^{-8}, 2.92×10^{-8} and 3.0×10^{-8} mm^3/Nm at 25, 30, 35 and 40 N, respectively) and better thermal stability than those (2.1×10^{-8}, 4.8×10^{-8}, 6.15×10^{-8} and 4.2×10^{-8} mm^3/Nm) of Nissan Jeep Cherokee brake pad. This implies an optimum combination of good wear resistance, friction properties and thermal stability for up to 200 s. Since brake application while driving is a short and intermittent process (< 3 minutes), the developed composite could serve as a replacement for asbestos brake pads for automobiles.

Keywords: Tile, Phases, Correlation, Replacement, Nissan Jeep Cherokee brake pad

1. Introduction

Over the years, improvement in braking systems has been one of top priorities in the automobile industry. Brake pads are vital components of braking systems for all categories of vehicles equipped with brake discs. This type of brake is made of steel backing plates with a material that can easily generate friction that is attached to a surface facing a brake disc [1]. Friction brakes decelerate a vehicle by transforming the kinetic energy of the vehicle into heat, via friction, and dissipating the heat to the surroundings [2]. Until the late 1960's, most cars used drum brakes on all four wheels. The pads for the drum brakes consisted of resins and asbestos, as well as a variety of other materials, to improve braking and wear. Later, automobile manufacturers began to incorporate disc brakes, especially for larger motor vehicles, because they had better braking performance. The Federal Motor Vehicle Safety Standard 105 required more stringent braking standards. It helped expedite the transition to disc front – drum rear braking systems [2-3]. Presently, disc brake systems have gained popularity because they dissipate heat and reduce the effect of wear better than drum brakes. Generally, the materials used for brake linings include metallic friction materials (Fe and Fe-Cu oxides), lubricants (graphite and Fe-Sb-Mo-Sn-Mn sulphides) and mineral fibers (barite, calcite, zircon and Al-silicates) used as fillers [3]. Brake pads traditionally consisted of asbestos fibers embedded in a polymer matrix along with several other components. However, exposure to asbestos carries health risks. That is asbestosis, an inflammation of lung tissue that is eventually fatal. It has also been implicated in mesothelioma, a cancer of the pleura. So, the use of asbestos fibre in brake pad systems is no longer acceptable. This development has necessitated a shift of attention to development of new asbestos-free brake pads made from agricultural and industrial waste products [4].

Researchers all over the world are currently exploring alternative materials for brake pads due to these environmental and health implications. Lately, agricultural residues or wastes have emerged as inexpensive filler materials that can be used for development of commercially viable composite materials. Agricultural wastes such as palm kernel shells, coconut shells, periwinkle shells, banana peels, egg shells, maize husks, coconut shells and bagasse, among others, have been studied as particulate reinforcements for eco-friendly brake pads. The results obtained from microstructural and mechanical (hardness, compressive, tensile and wear tests) characterisations showed that they can be effectively used as a replacement for asbestos materials in automotive brake pads. Unlike asbestos

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based brake pads, composite brake pad are eco-friendly with no known associated health hazards [5-10]. For instance, Aigbodion and Agunsoye (2010) [11] studied the development of asbestos-free brake pads using bagasse to replace asbestos. Compression moulding was used to make brake pads using 70% bagasse and 30% resin. Microstructural analysis, hardness, compressive strength, density, flame resistance, water and oil absorption were examined. Their findings showed uniform distribution of resin in the bagasse and the properties of the brake pads were improved with fineness of the bagasse particles. Moreover, Ibhadode *et al*. (2008) [6] used palm kernel shells (PKSs) in brake pads. It was reported that the mechanical and physical properties of the developed PKS composite brake pad compared well with those of commercial asbestos-based friction lining materials.

Industrial wastes are generated in significant quantities as waste materials or by-products from steel industries. They usually contain considerable quantities of valuable metals and materials. Converting these solid wastes from one form to another for reuse either by the same industry or in other applications is essential not only for conserving metals and mineral resources, but also for protecting the environment [12]. Moreover, the increasing accumulation of industrial waste is a major environmental challenge that has led to the promulgation of various environmental laws in recent years. These regulations have compelled the engineering sector to look for ways of harnessing such waste products to develop low cost composite materials that are environmentally acceptable [13-14]. Industrial wastes, such as ceramic oxides, are generated from the ceramic industry and from every day use of ceramic materials. These wastes are not efficiently and economically harnessed in Nigeria, creating a need to explore the possibility of using particulates of agricultural and industrial wastes as reinforcing materials in the manufacture of brake pads. This will fulfill the Federal Government of Nigeria's local content policy and increase foreign exchange savings.

The study seeks to compare the properties of a newly developed composite brake pad that uses particulate industrial wastes with those procured commercially in an attempt to develop an asbestos free brake pad for automobile applications.

2. Materials and methods

Epoxy resin (LY 556)/hardener (HT 975) and commercial brake pad used in this study were purchased locally at Ojota and Owode Onirin, Lagos, Nigeria. Steel slag, gum arabic, aluminium chips, graphite/bentonite and ceramic tiles were obtained from steel manufacturing industries in Lagos, a chemical shop at Zaria, the Mechanical Engineering Workshop at the University of Lagos, Nigerian Foundries in Oshodi, Lagos and an active construction site in Lagos, Nigeria. The commercial brake pads used as control were made for a Nissan Jeep Cherokee serial number QX56. They had dimensions of 142 x 48.3 x 17.2 mm and were procured at Owode Onirin in Lagos, Nigeria.

Steel slag, gum arabic, aluminium chips, graphite and clay were sundried at an average daily temperature of 25˚C for 10 days. Then, they were crushed manually using a hammer. The broken pieces of each were pulverised using a ball mill in accordance with [15]. The pulverised powder was size-sorted manually using a No. 150 BSS (106 µm) mesh sieve.

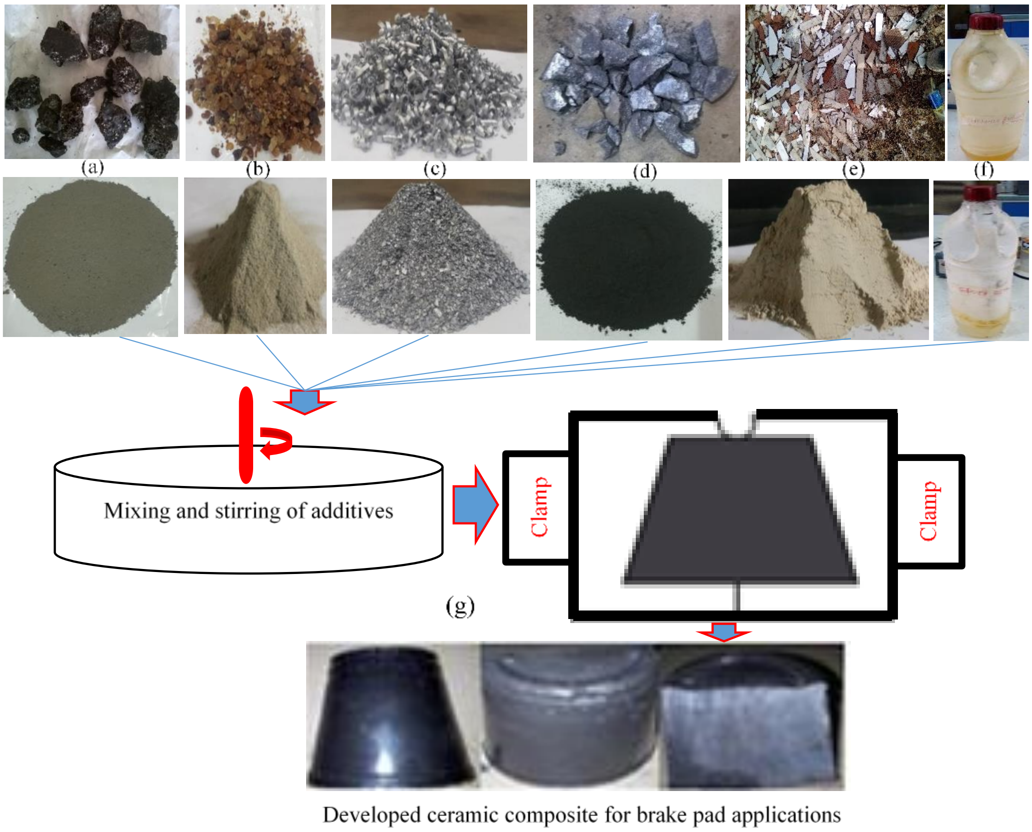

Figure 1 Materials used for development of composite brake pads: (a) steel slag (b) gum arabic (c) aluminium (d) graphite (e) ceramic powder before and after pulverisation (f) epoxy (g) sequential steps in the composite development

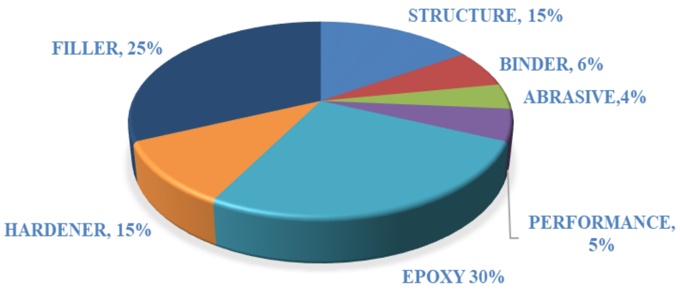

Figure 2 Formulation of the various categories of materials used in the developed brake pad

Table 1 Chemical composition of ceramic particles obtained from broken tiles

Compounds	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**CaO**	**Na_2O**	**K_2O**	**P_2O_5**	**TiO_2**	**MgO**	**Other**
% composition	64.30	19.20	2.51	1.85	0.23	1.80	0.05	0.81	2.15	7.10

A particulate steel slag (refractory filler), gum arabic (binder), aluminium chips (an abrasive material) and graphite (for performance) are presented in Figures 1a-e. Table 1 displays the chemical composition of the ceramic powders obtained from the broken tiles. 1.2 kg epoxy and 600 g hardener were mixed, equivalent to a 2:1 ratio. Weighed amounts of other additives (1 kg filler, 240 g gum arabic, 1.2 kg epoxy/0.6 kg hardener, abrasive, 200 g graphite, 400 g ceramic powder and 200 g bentonite), in the ratio shown in Figure 2, were added and stirred manually for 10 minutes using a wooden stick. The milled blend was poured into wooden moulds and clamped (see Figure 1g). The mixed blend was allowed to cure at room temperature for 24 hours after which the composite brake pads were removed from their moulds. Three different samples were prepared and examined for each analysis and an average of the readings was reported.

Microstructural and elemental analysis was done using a JEOL Scanning Electron Microscope (MODEL: JSM-6510**)** operated at an accelerating voltage of 2.0 kV. An X-ray diffractometer was used to identify the phases present. The analysis was done in the 2θ range between 8–90°. Thermal stability of the sample was measured in terms of global mass loss using a thermogravimetric analyser (model TGA Q50). The tests were performed in an inert atmosphere over the temperature range from 25 °C to 1000 ° C at a heating rate of 10 °C/min. The wear rate for the sample was measured with CETR UMT-2 Tribometer at loads varying from 25 to 40 N in 5 N intervals with sliding speed and distance of 2 m/s and of 2 m, respectively, using a tungsten carbide ball. Frictional force at the sliding interface was measured using a strain gauge while the wear rate was determined by weighing the specimen before and after each test. The formula used to convert the weight loss into wear rate is expressed as Equation 1:

$$\text{Wear rate} = \frac{\Delta W}{S*A} \quad (1)$$

where ΔW is the weight difference of the sample before and after the test in mg, S is total sliding distance (m) and A is the applied load (N).

3. Results and discussion

3.1 Friction and wear resistance of the developed brake pad composites

The friction coefficient (CoF) of the commercial sample was compared with the developed composite brake pad as a function of increasing time (see Figure 3a-b). A steady state increasing then decreasing pattern was observed in the profile of the CoF with time. The developed composite brake pad exhibited better friction properties (0.06 μ) than the procured commercial grade brake pad (0.033) for up to 200 s. At times longer than 200 s, the friction properties of the procured commercial grade brake pad were better. A possible explanation for this degradation in the friction performance/property of the developed composite brake pad when it was used for longer periods could be linked to its inability to withstand increasing heat generation. Since heat generation due to the movement of surfaces in contact in the wear investigation is analogous to the contact of the brake pad with a rotor on brake application, continual brake application beyond 3 minutes (180 s) can lead to failure of the developed composite brake pad. Furthermore, for up to 3 minutes, the developed composite was a more reliable brake pad than those procured commercially.

Production techniques impact material properties [16]. Typically, a brake pad is fabricated under pressure. Conventional casting techniques were used in this study for the development of the composite brake pad. The degree of compaction may have influenced the structural integrity of the developed composite brake pad and led to poor friction properties at times longer than 200 s. Another prerequisite for brake system is an excellent resistance to wear. The wear characteristics of both the commercial grade and the developed composite brake pads were investigated under increasing loads and times to ascertain if a slight reduction in the CoF will translate into greater wear resistance.

An initially high loss of material may have been due to rupture of the uneven surfaces of the virgin brake pad, resulting in a third body between the interfaces. The presence of the extra material at the interface resulted in an increased coefficient of friction for up to 200 s. The decrease in the CoF can be attributed to chain scission of the binders due to frictional heating, leading to a decrease in their ability to remain joined with the additive as the friction induced heating increased. A material with a high coefficient of friction is a very good material for the production of brake pads. Good frictional property of the developed composite brake pads for up to 200 s is an affirmation that composite mix of epoxy, hardener, gum arabic, aluminium, graphite, ceramic clay and bentonite is a good substitute for production of asbestos free brake pads after an improvement on the

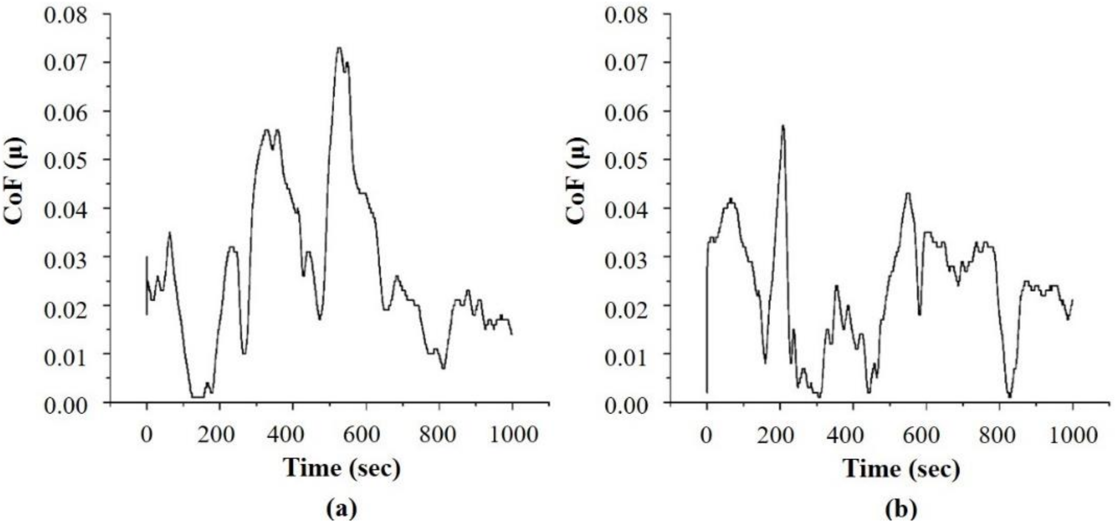

Figure 3 Coefficient of friction vs. time in (a) a commercial grade brake pad (b) the developed composite brake pad

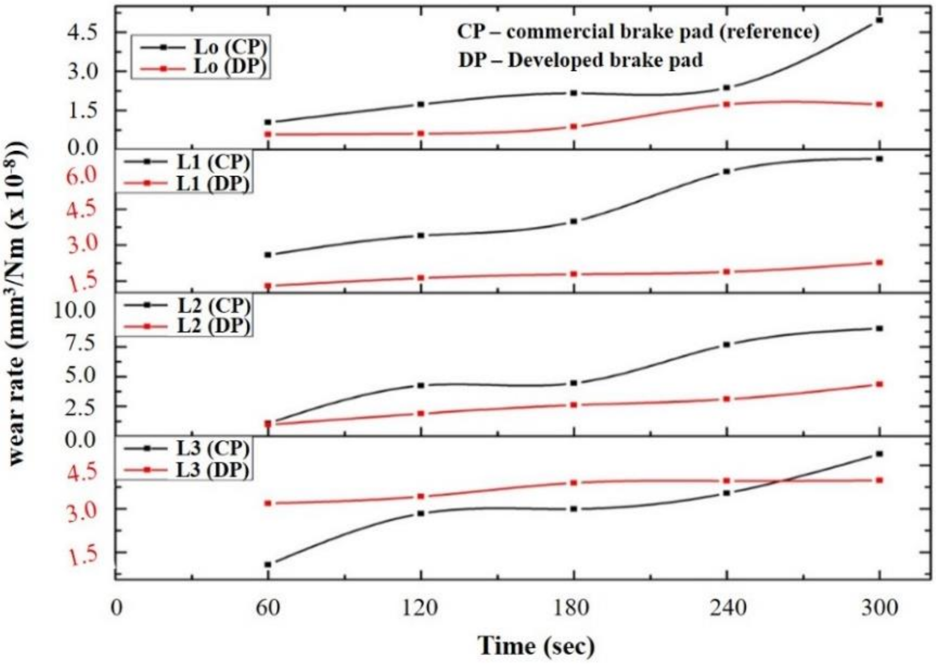

Figure 4 Matching of wear behaviour of the commercial grade and the developed composite brake pads

conventional casting method and optimisation of the additive composition.

Figure 4a-b compares the wear behaviour of the commercial brake pad (CP) and the developed composite brake pads (DP) under dry sliding conditions as well as, increased times and loads at a rotational speed of 150 rpm. Generally, under all the loading conditions, the wear rate of both pads increased with time. This observation shows that thermomechanical loading is a primary factor. Moreso, the developed composite brake pad showed a smaller wear rate compared to the commercial brake pad. For instance at 200s, wear rates of the developed composite brake pad were 1.3 x 10^{-8}, 1.8 x 10^{-8}, 2.92 x 10^{-8} and 3.0 x 10^{-8} mm^3/Nm at applied loads of 25, 30, 35 and 40 N respectively. The developed brake pads showed smaller wear rates, 2.1 x 10^{-8}, 4.8 x 10^{-8}, 6.15 x 10^{-8} and 4.2 x 10^{-8} mm^3/Nm, respectively, for the same times and applied loads. Typically, a brake pad should posses good friction stability and, wear rate over a considerable period of time [9]. Nevertheless, the developed composite brake pads showed an improved resistance to material loss under comparable test conditions. The improvement may have been related to binding properties of the epoxy and gum arabic. Therefore, both are suitable binders for use in brake pad production applications.

3.2 Microstructures of the developed composite brake pad

Figure 5 reveals microstructure and EDX spectrograph of a commercial brake pad before and after wear analysis. The microstructure in Figure 5a is homogeneous, implying that the component materials for making the commercial grade brake pad were thoroughly mixed to produce a single solid phase with no particle agglomeration. Since homogeneity, continuity and isotropy are some of materials properties determining a material's behaviour in service, such a structure in Figure 5a affirms the integrity of the

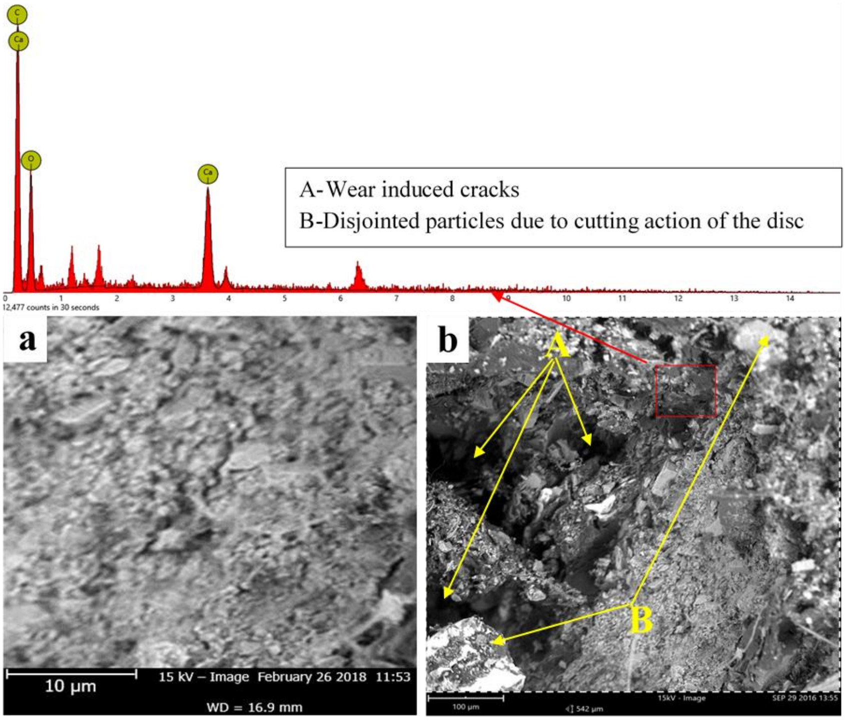

Figure 5 SEM/EDX of a commercial brake pad (a) before wear analysis, and, (b) a worn-out surface

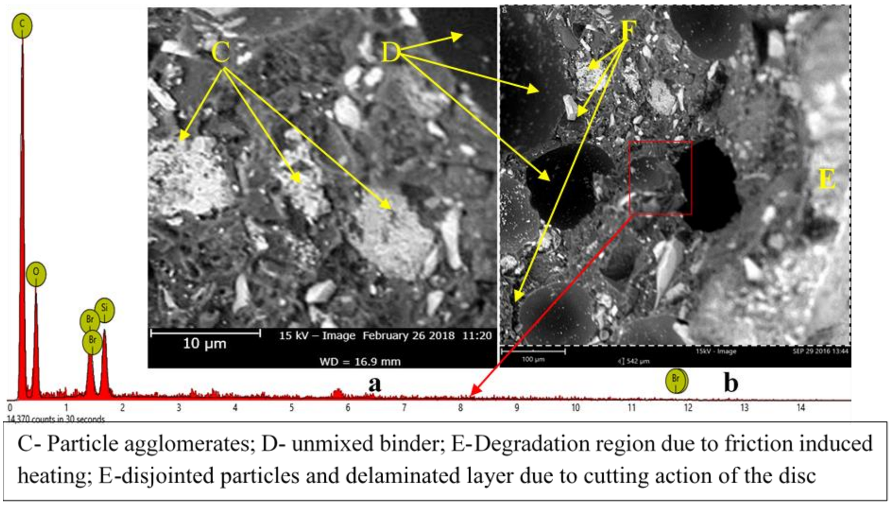

Figure 6 SEM/EDX of the developed composite brake pad (a) before wear, and, (b) a worn-out surface

commercial brake pad. After wear analysis, two regions A and B were discernably different as shown in Figure 5b. Symbol A depicts cracks developed from fusion of wear induced voids, symbol B shows disjointed particles (wear debris) due to the abrasion of the moving disc in contact with the surface of the commercial brake pads during wear analysis. The presence of C, Ca and O confirmed that the commercial brake pad was produced from both organic and inorganic minerals.

Particle agglomerates and the epoxy/binder was not completely mixed with other additives at regions C and D in Figure 6a, revealing that the microstructure of the developed ceramic composite brake pad was not perfectly homogeneous. This indicates that manual stirring alone is not sufficient for achieving homogeneous mixing of the component materials. Agglomerates within the developed composite created a region of weak bonding due to absence of or insufficient amount of binder to join individual particles. These regions served as a areas of stress concentration and interfered with stress transfer and distribution during wear analysis [17]. The microstructure seen in Figure 5a had better structural integrity than that in Figure 6a. Surprisingly, the epoxy composites displayed higher wear resistance than its commercial grade counterpart. This observation can be attributed to various combinations of materials used in developing each of the brake pads. There were various phases, as shown in Tables 1 and 3 in the developed composite brake pads, Table 2 gives data for commercial brake pad. Figure 6b shows the topography of the developed composite after wear analysis. Two regions E and F were identified. Symbol E reveals a degraded region due to combined mechanical and thermal stresses from the applied load and friction induced heating. Elemental analysis of the developed composite brake pads

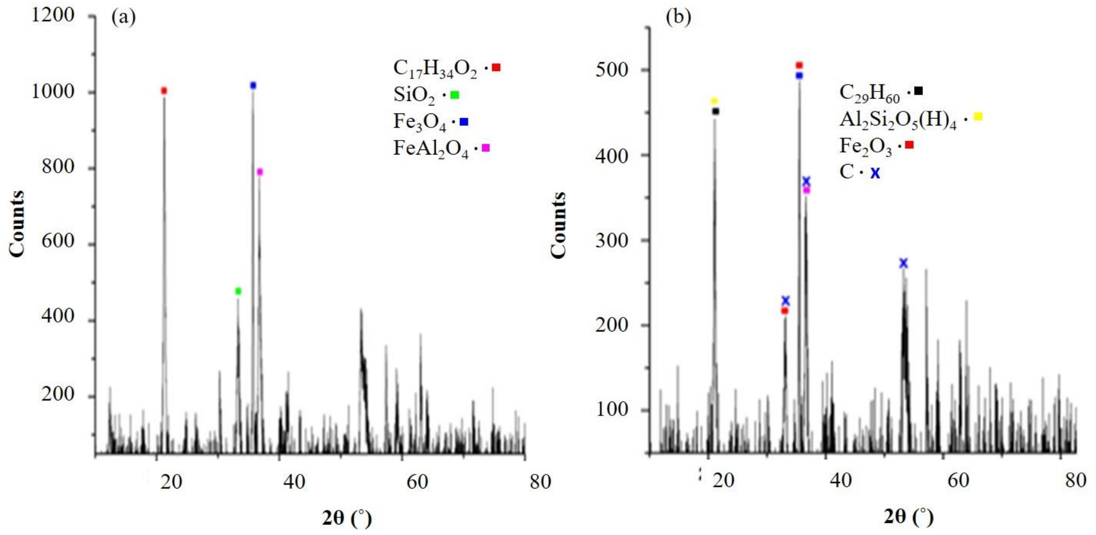

Figure 7 XRD of: (a) commercial grade brake pad (b) cast epoxy resin based brake pad

Table 2 Compounds present in the commercial grade brake pad

Score	Compound Name	Chemical Formula
41	Magnetite	Fe_3O_4
27	Heptadecanoic Acid	$C_{17}H_{34}O_2$
29	Silicon Oxide	SiO_2
24	Hercynite	$FeAl_2O_4$

Table 3 Phases of the developed composite brake pad

Score	Compound Name	Chemical Formula
57	n-Nonacosane	$C_{29}H_{60}$
40	Hematite	Fe_2O_3
23	Nacrite	$Al_2Si_2O_5(H)_4$
24	Lignite	C

showed that they contained C, O, Br and Si, as shown by the EDX in Figure 6.

3.3 X-ray diffractometry of the developed composite brake pads

The XRD profile of the commercial brake pad is presented in Figure 7a. The major peaks at 21.11, 33.312, 35.53 and 36.62° indicate the presence of Magnetite, (Fe_3O_4), Heptadecanoic Acid ($C_{17}H_{34}O_2$), Silicon Oxide (SiO_2) and Hercynite, ($FeAl_2O_4$), respectively. The score count of each of these phases is presented in Table 2. The observed phases in Table 2 confirm that the commercial brake pads were made from organic and inorganic materials not found in asbestos brake pads. $C_{17}H_{34}O_2$ is a solid organic compound and can easily burn in the presence of heat. Other phases are hard and refractory. Their presence in commercial grade brake pads enhanced both their wear resistance and thermal stability while reducing the thermal conductivity of the organic phase. Figure 7b show the phases of the developed brake pad with their corresponding peaks. The count score and chemical formula of the compounds is presented in Table 3. Comparison of the XRD profiles of the developed composite brake pad with those of the commercially available ones shows that the phases of the developed composite brake pad were entirely different than those in the commercial brake pad since they were made from different materials. Consequently, they behaved differently under the same conditions. The phases in Table 3 are also organic and inorganic. However, n-nonacosane in Table 3 is less thermally active than heptadecanoic acid in Table 2. Hematite (Fe_2O_3) and lignite (C) are hard and refractory, while nacrite ($Al_2Si_2O_5H_4$) is refractory and relatively soft. A combination of these phases had a tendency to enhance the thermal stability, hardness and toughness of the developed composite brake pads. Wear resistance is a function of hardness and toughness of the phases making up the composites. Hard phases blunt the teeth of the cutting disc while the tough phases firmly hold the hard phases together and prevented easy pullout or delamination of the hard phases from their binder. Similar explanations have been presented in the literature [18-20]. Therefore, higher wear resistances (lower wear rates) of the developed composite brake pad than those of the commercial grade can be linked to the combined hard and tough phases of the former since the latter phases are completely hard. Similarly, the presence of elemental carbon (lignite) in addition to other refractories (see Table 3) may account for higher thermal stability of the developed composite brake pad.

3.4 Thermal properties

The thermogravimetric (TG) curves in Figure 8 described the thermal behaviour and stability of the commercial as well as the developed composite brake pads. Both TG profiles are characterised by step-wise degradation. An initial oxidation resulted in about a 7 % mass loss below ~100 °C. This mass loss can be associated with the formation of volatiles during firing. Continued heating above ~400 °C led to the oxidation of carbon. However, the developed composite brake pad showed a slightly better stability to degradation in an oxidising environment compared to the commercial pad. The residual mass of the CP was 68% compared to the developed composite brake pad, 72%, after heating to 1000 °C. The improved overall stability can be connected to the reinforcing additives. From Figure 8b, maximal degradation occurred at 100, 400 and 700 °C. Although TG curves have shown the better thermal stability of the developed composite than those of commercial grade, the ability of the developed composite brake pad to remain thermally stable under abrasion for longer than 200 s is lower. Moreover, an optimal combination of resistance to

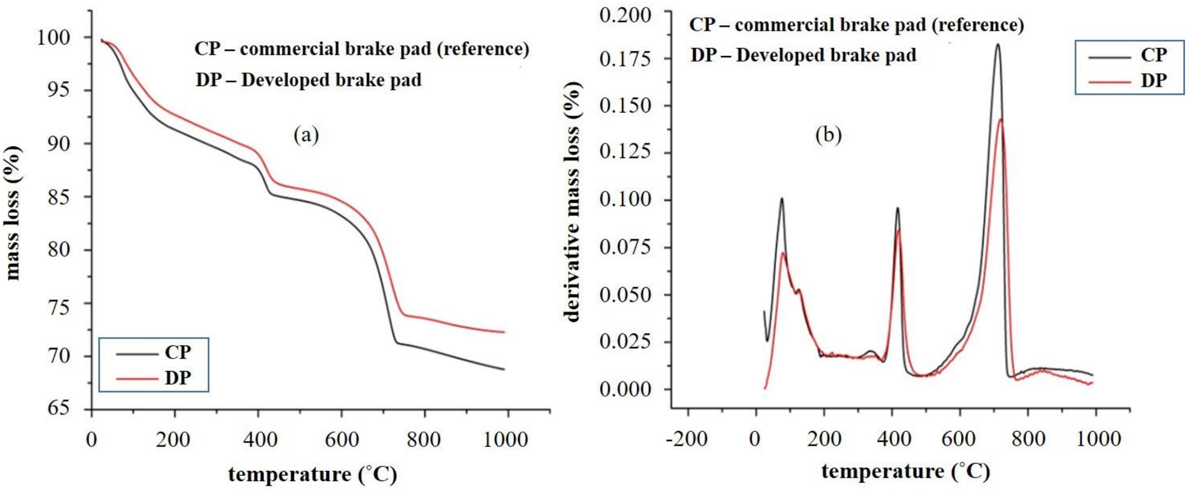

Figure 8 Thermogravimetric analysis of the commercial brake pad and the developed composite brake pad as (a) percent mass loss (b) percent derivative mass loss

abrasion/wear and thermal stability is prerequisite for superior performance in brake pad applications. Inability of the developed composite brake pad to retain its friction property beyond 200 s limits its performance. Since brake application while driving is short and intermittent (< 3 minutes), the developed composite can be used for automobiles.

4. Conclusions

An asbestos free brake pad was developed from recycled clay tiles, epoxy resin/hardener, gum arabic, graphite and foundry slag using a casting method. The performance of the developed composite brake pads was compared with that of the commercially available imported brake pads obtained in Nigeria. Comparison revealed a fair suitability of the developed composite for automobile brake pad applications. Moreover, the properties of the developed epoxy composite for brake pad applications can be improved through modification of fabrication techniques prior to full curing of the epoxy used as a binder for the other additives in this novel composite brake pad.

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