



THERMAL AND TRIBOLOGICAL CHARACTERISATION OF ASBESTOS-FREE PARTICULATE CERAMIC MATRIX COMPOSITES

Stephen I. Durowaye¹, Olatunde I. Sekunowo² and Ganiyu I. Lawal³

¹University of Lagos, Faculty of Engineering, Department of Metallurgical and Materials Engineering, Lagos, Nigeria. Email: sdurowaye@unilag.edu.ng

²University of Lagos, Faculty of Engineering, Department of Metallurgical and Materials Engineering, Lagos, Nigeria. Email: osekunowo@unilag.edu.ng

³University of Lagos, Faculty of Engineering, Department of Metallurgical and Materials Engineering, Lagos, Nigeria. Email: glawal@unilag.edu.ng

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ABSTRACT

Production of friction particulate ceramic matrix composites (PCMCs) by powder metallurgy method was carried out using iron 105 μm millscale, 50 μm silica sand, 80 μm magnesia and 53 μm bentonite clay as input materials. Different formulation of the blend of these materials were prepared and the composites were produced. Microstructural, frictional, thermal and wear characterisation were determined using standardised methods. The ceramic composites exhibited very good properties in terms of thermal stability (heat resistance), coefficient of friction (COF) and resistance to wear. The specific values of these properties exhibited by sample D having 12 wt. % iron millscale addition in terms of high resistance to thermal decomposition in the temperature region (0 – 1600 $^{\circ}\text{C}$) indicating thermal stability, appreciable high COF (0.59) and very low wear rate of 1.9093×10^{-6} g/m are desirable. The uniform dispersion of the particles as observed in the microstructure and strong bonding/adhesion contributed to the enhancement of the properties. These results are indication that the composite is very suitable for application in areas where high resistance to thermal stress and abrasive wear are required such as the brake assembly of automobiles, specifically the brake pads.

KEYWORDS: Particulate ceramic matrix composites, powder metallurgy, coefficient of friction, thermal behaviour and wear characteristics.

1. INTRODUCTION

The brake system is an important part of automobiles because of the vital role it plays and the brake pads are vital components of the brake system. Without the brake system, driving vehicles could possibly result in road accidents which can endanger the lives of drivers, passengers, and also pedestrians. The function of the brake is to retard or stop the motion of vehicles. It has been reported that an average automobile driver uses the brake about 75,000 times a year, making the brake one of the most important and overworked parts of vehicles ([McPhee, 2007](#)).

The ban and gradual phasing-out of asbestos brake pads in many parts of the world due to health reasons have sparked the onset of extensive research and development into safer alternatives. Consumers demand and public awareness also led to extensive research and development of brake pads in the early 1990s to find suitable replacement for asbestos brake pads. These have promoted the production of different brake pad materials in the past decade of asbestos-free brake pad revolution. There are several categories of brake pads based on materials constituent such as metallic, semi-metallic and ceramic brake pads ([Ma et al., 2012](#)).

Despite the significance of the brake, it has its own challenges or problems. One of the reasons why automobiles brakes experience failure is due to the heat generated during braking causing heating of the brake pads which reduces braking efficiency. Accumulated heat within the brake system causes gradual thermal expansion and composition debonding of the brake pads resulting in impaired structural integrity and loss of friction. Further, these conditions aggravate the rate of wear of the brake pads surfaces which reduces its arresting capability resulting in brake-failure ([Aza, 2014](#)). The combination of these dysfunctional occurrences usually have adverse effect on brake pads in terms of performance and durability.

Attempts have been made to solve the problems associated with brake pads by using different types of materials to develop/produce brake pads. Some of these materials have problems associated with them. For instance, some less thermally stable resins in brake pads were identified as producing oily residues that contributed to brake fade ([Tanaka et al., 1973](#)). Certain phenolic resins were identified as evolving volatile gases during degradation causing a back pressure on the brake pad also resulting in brake fade ([Herring, 1990](#)). Of significant importance to the friction performance is the thermal stability and degradation reactions of the organic resin matrix in the brake pad ([Bark et al., 1975](#)). This highlights the necessity for thermally stable raw materials that will not evolve friction destabilising by-products during thermal degradation.

The friction between brake pads and discs converts kinetic energy of automobiles to thermal energy. The brake pads must be capable of absorbing heat to prevent distortion or cracking from

thermal stress until the heat is dissipated (Adebisi et al., 2011). The coefficient of friction between pads and discs and wear behaviour of the pads must satisfy standard requirements (Jafar et al., 2012). The materials for brake systems should have stable and reliable frictional and wear properties at different conditions of load, speed, temperature, environment, and high durability (Maleque et al., 2010). Brake pads must satisfy several desirable properties such as wear rate, thermal stability and low noise making the selection of a proper formulation to require many experimental trials/attempts (Kchaou et al., 2013; Sellami et al., 2014).

There are many studies on the development of brake pads. Ademoh (2015) developed maize husks (asbestos free) based brake pad. The results of the study showed that coefficient of friction, abrasion resistance, thermal conductivity, etc of maize husk filler based brake pads compared favourably with commercial brake pads and those produced in past related research works on asbestos free brake pads. Ma et al., (2012) investigated the effect of zirconium silicate as an abrasive on the brake friction performance. They reported that by increasing weight percent of zirconium silicate, the coefficient of friction of brake pad was enhanced but the wear rate was depressed. Some studies focused on the environment issues related to diffraction of hazardous constituted materials of brake pads such as copper (Straffellini et al., 2015; Lee and Filip, 2013). Straffellini et al., (2015) reported that similar to other heavy metal emissions, the release of copper into the atmosphere may have important environmental and health effects. So they introduced some replacements for copper in brake pads such as graphite. Singh et al., (2015) used lapinus-wollastonite fibers in the friction composite materials and carried out wear test of samples which were different in the amount of both fibers. They concluded that the increased lapinus fiber content enhanced the friction stability whereas, increased wollastonite fiber enhanced the wear performance.

Jafar et al., (2012) applied SAE J 661 to evaluate 10 formulations (15 types of materials) and select best formulation according to wear test results. The most important result of their study was dependency of wear results on materials constituted brake pads. That means, achieving proper wear properties needs several experiments with different chemical compositions to select the best formulation. Kchaou et al., (2013) carried out wear test of brake pads. They reported that in the high contact pressure, the friction coefficient was degraded relative to low contact pressure.

Thermal stress and wear being deleterious materials' performance phenomena, can be mitigated by the use of particulate ceramic matrix composites (PCMCs). Through the combination of certain materials composition modification and innovative processing, brake pads capable of

exhibiting enhanced thermal stability, low wear rate or high wear resistance can be developed. However, without proper materials selection and formulation, this may not be possible. This work is aimed at producing a friction composite material by powder metallurgy method using iron millscale, silica sand, magnesia and bentonite clay that is suitable for application as automobiles brake pad.

2. MATERIALS AND METHODS

2.1. Materials

The materials used in this study are iron millscale, silica sand, magnesia, and bentonite. Iron millscale was sourced from Universal Steel Nigeria Limited located in Ogba, Lagos. Silica sand was obtained from the beach of the Lagos Atlantic Ocean (bar beach). Magnesia and bentonite clay powders were obtained from a local vendor within the chemicals supplier trade group registered in Nigeria but were manufactured in China and Wyoming, USA respectively. The photographs of these materials and their chemical composition determined using an X-ray spectrometer model ARL9400XP+ Thermo are shown in [Fig. 1](#) and [Table 1](#) respectively.



Fig. 1. Photographs of input materials (a) 105 μm iron millscale particles, (b) 53 μm bentonite clay particles, (c) 80 μm magnesia particles, (d) 50 μm silica sand particles.

2.2. Production of the Composites

The as-received silica sand and iron millscale were milled using a steel ball mill (model A50 43, Mashine, France) and sieved to 50 μm and 105 μm particle sizes respectively. These materials were manually blended with 80 μm magnesia and 53 μm bentonite according to the formulation presented in [Table 2](#) to obtained many formulations of samples A, B, C, D and E. Appropriate quantity of water was added to each blend. Thereafter, each of the wet-mixed blends was poured into the fabricated metallic mould. Green samples were obtained by uniaxial cold pressing at 0.33 MPa using a hydraulic press (Type P100EH, Model No. 38280, Weber

Hydraulik, Germany) to enhance surface smoothness of the samples. Little lubricant (Mobil engine oil XHP) was rubbed on the inner part of the moulds as a releasing agent for easy discharge of the samples from the moulds after compaction. The samples were sun dried (30-32⁰ C) for 24 hours followed by drying under a controlled atmosphere in an oven at 110⁰ C for 24 hours to remove any moisture left in the samples and to avoid cracking during high sintering temperature employed. Gradual sintering of the samples at 1200⁰ C was carried out in a muffle furnace pre-set at heating rate of 10⁰ C/min. After 3 hours of sintering, the samples were removed and cooled at room temperature. Sintering is heating the samples below the melting point of the materials but high enough to develop significant solid state diffusion. This is to enhance bonding of the particles for properties enhancement. This production process is similar to the one used by [Aigbodion et al., \(2010\)](#). The photographs of some of the composite samples and brake pads produced are shown in [Fig. 2](#).

Table 1. Composition of the input materials.

Compounds	Amount (wt. %)			
	Iron Millscale	Silica	Magnesia	Bentonite
FeO	69.57	0.04	-	-
Fe ₂ O ₃	24.08	0.16	-	3.89
Fe ₃ O ₄	6.06	0.001	-	-
SiO ₂	0.02	99.52	0.01	63.57
MgO	0.03	0.02	99.94	2.64
CaO	0.19	0.03	0.02	1.33
MnO	0.03	0.02	-	-
Al ₂ O ₃	0.02	0.19	-	25.63
Na ₂ O	-	0.006	-	2.31
K ₂ O	-	0.004	-	0.51
TiO ₂	-	-	-	0.12
L.O.I	-	0.001	0.03	-

L.O.I = Loss on ignition

Table 2. Materials formulation in weight percent (wt. %).

Sample	Iron millscale (wt. %)	Silica (wt. %)	Magnesia (wt. %)	Bentonite (wt. %)	Total (wt. %)
A	0	5	15	80	100
B	4	5	15	76	100
C	8	5	15	72	100
D	12	5	15	68	100
E	16	5	15	64	100

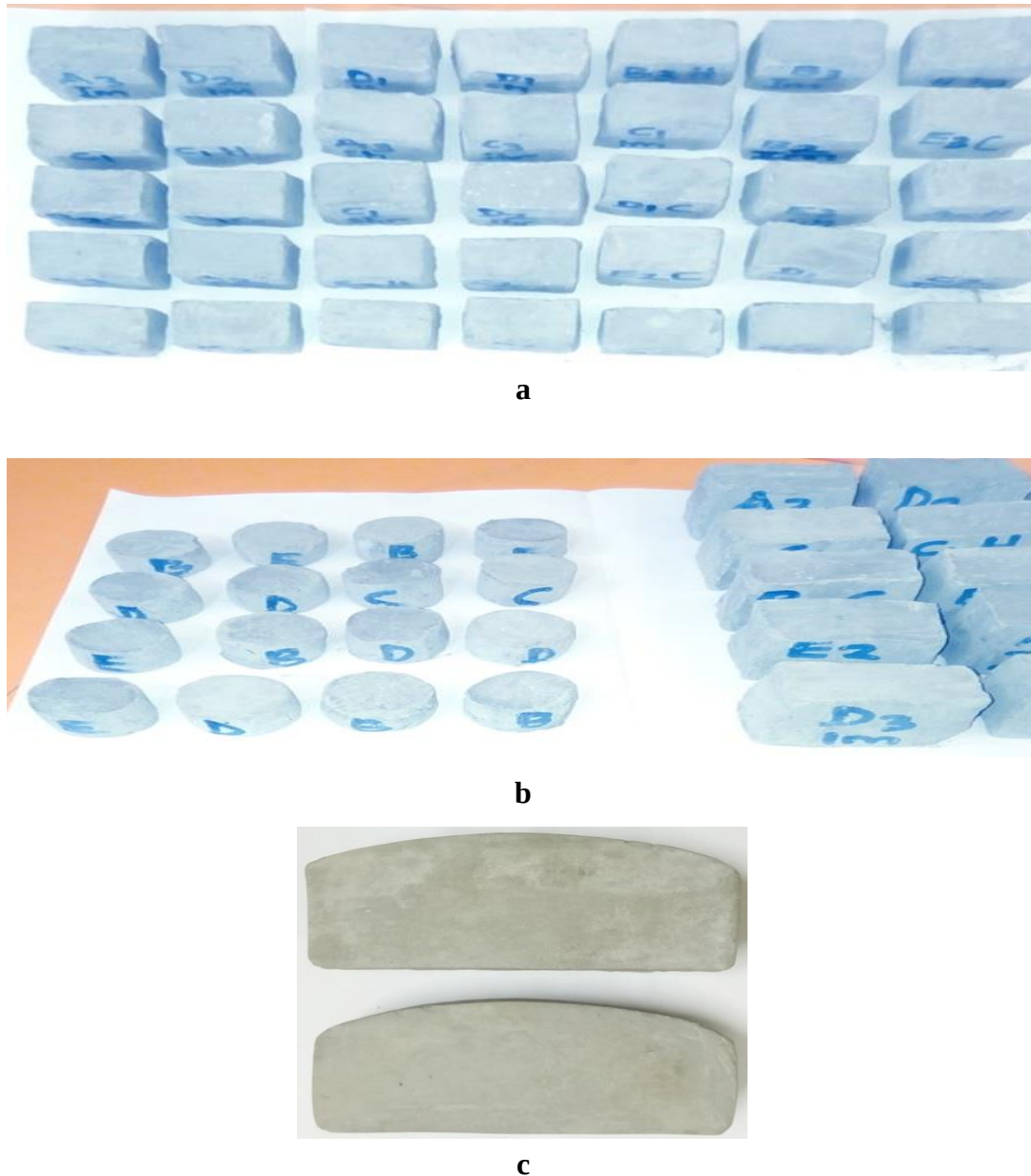


Fig. 2. a. Photograph of some of the composite samples produced.; b. Photograph of some of the composite samples produced; c. Photograph of two samples of composite brake pads produced.

2.3. Characterisation of the Composite

Two of the samples were subjected to qualitative analysis using an X-ray Diffractometer (XRD). They were sample A which did not have iron millscale addition and sample D representing samples with iron millscale addition. 10 g of sample was placed in the sample holder and was bombarded by a Cu K α radiation (40 kV and 40 mA) within the X-ray tube and the intensities of the reflected X-rays were recorded. The scan range, scan step size, time per step were evaluated simultaneously. The scan ranges were selected based on strong peak positions and limiting interference from excipients which lead scan range set to 10° to 70° (2 Theta) in a step scan mode of 0.05° , speed $2^{\circ}/\text{min}$, time/step 0.025° , wavelength $1.54056\text{\AA}$ (Cu).

The microstructure of the test samples was examined using an ASPEX 3020 model variable pressure scanning electron microscope (SEM) equipped with Noran-Voyager energy dispersive X-ray spectroscopy (EDS). Thermal stability of the samples was determined by the flame resistance method using a Bunsen burner and also by thermogravimetric method using a differential thermal analyser model NETZSCH DTA 404 PC having a furnace facility of 10⁰ C/min heating rate. For the determination of the coefficient of friction (COF), this experiment was carried out using a smooth inclined mild steel plane inclined at 35⁰. Each sample was attached to a string and placed on the mild steel inclined plane. The string was passed through a pulley which was connected to a mass hanger. Loads were added to the hanger which made the samples to slide along the surface of the steel plane. Varying loads were added to the hanger for sliding due to the fact that the load of the samples varies. The coefficient of friction is the ratio of the sliding force to the normal force. Hence, the coefficient of friction (μ) between each sample and the mild steel surface was calculated using Equations 1 and 2 ([Essays, 2018](#)).

$$\mu = \frac{\text{Sliding force}}{\text{Normal force}} \quad 1$$

$$\mu = \frac{P - W \sin \theta}{W \cos \theta} \quad 2$$

Where:

P = Load on the hanger (N)

W = Weight of sample (N)

θ = Angle of inclination

Dry sliding abrasive wear test was conducted on the samples using a pin-on-disc wear tester in accordance with ASTM G99 standard at room temperature. A load of 30 N was applied using dead weights. The samples were weighed and recorded as W_0 before the test using an electronic digital weighing balance (Model No. UW1020H, SHIMADZU, Japan) with sensitivity of ± 0.001 mg. A revolving disc upon which a Festool 496625 P36-grit sandpaper of 160 mm diameter surface has been mounted was used. The disc rotated with the help of a D.C. motor of speed 250 rpm and sliding occurred between the stationary sample and the rotating disc. As the disc rotated, the sand paper in direct contact with the sample also rotated at a relative speed to the disc and caused the sample to wear. After the pre-set time of 5 mins, the machine was turned off. The sample was cleaned and the final weight was measured and recorded as W_1 . The weight

loss of the sample was determined by finding the difference between the final and initial weights. The specific wear rate was determined using Equations 3 – 5 (Dan-Asabe et al., 2012; Olabisi et al., 2016). The worn surfaces of the test samples were also examined using the scanning electron microscope (SEM).

$$\text{Wear rate } (\omega) = \frac{\Delta w}{S} \quad 3$$

Where:

$$\Delta w = w_0 - w_1 \quad 4$$

$$S = 2\pi NDt \quad 5$$

Δw = weight loss in g

w_0 = weight of sample before wear in g

w_1 = weight of sample after wear in g

S = sliding distance in m.

D = diameter of the P36-grit sandpaper (abrasive) which is 16 cm

N = revolution per minute of the pin-on disc which is 250 rpm

t = time taken to expose each of the samples to wear which is 5 mins

$\pi = 3.142$.

3. RESULTS AND DISCUSSION

3.1. Microstructure

As presented in Fig. 3, mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) has the major phase in the X-ray diffraction (XRD) spectrum of sample A which did not contain iron millscale occurring at angles 22° , 27° , 46° and 60° with different intensities. Spinel (MgAl_2O_4) and magnesia (MgO) are other phases present at different diffraction angles and intensities. The XRD spectrum of sample D representing samples with iron millscale addition presented in Fig. 4 shows the presence of FeO, mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$), MgO and spinel (MgAl_2O_4) occurring at different diffraction angles and intensities. This is an indication that samples B, C and E will also have similar phases as sample D because they contain iron millscale in their formulation.

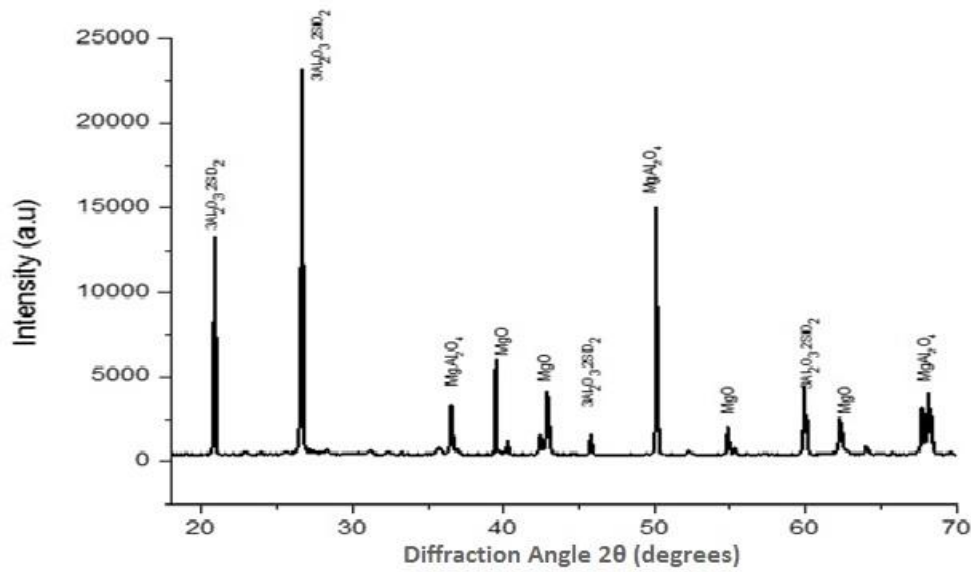


Fig. 3. XRD spectrum of sample A without iron millscale addition.

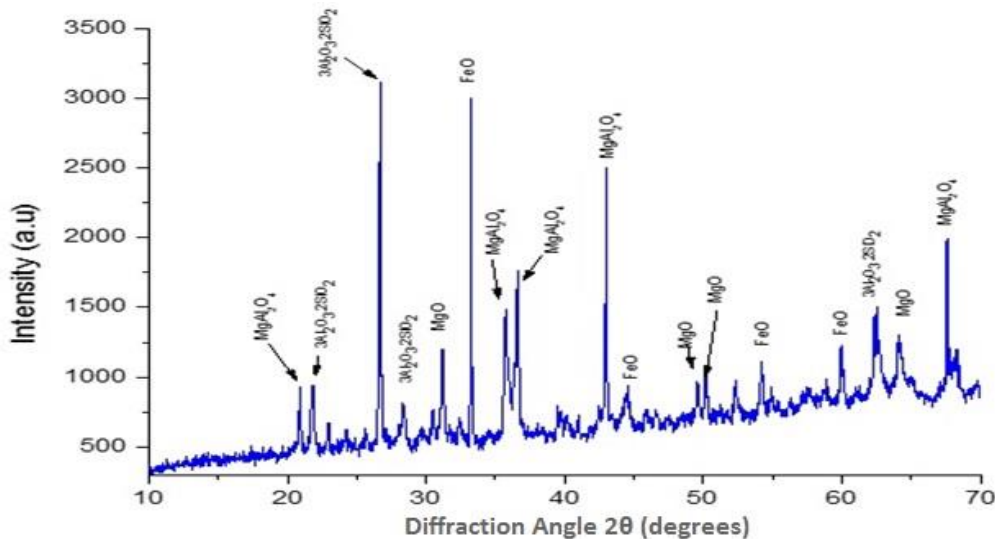


Fig. 4. XRD spectrum of sample D with iron millscale addition.

The SEM micrographs and EDS spectra of the samples are shown in Fig. 5–9. The micrographs show that the samples are made up of different heterogeneous additives with differences in the geometry of the particles that are globular and needle-like. The elemental composition of the samples is shown by the EDS spectra. Sample A contains O, Si, Al, Mg and Ca while samples B, C, D and E also contain these elements and Fe (iron). There are also indistinguishable peaks in the spectra indicating the presence of other elements in very small amount. The white spots present in the microstructure are likely magnesia (MgO) phase while the dark spots are likely FeO phase from the millscale. The ash coloured needle-like portion is likely the mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) phase which is a highly crystalline ceramic compound from bentonite-silica mixture and the gray mixed with whitish spots are likely the spinel phase formed during

sintering of the samples. The various phases present in the microstructure of the samples are shown in Fig. 5 – 9 which is corroborated by the XRD spectra of Fig. 3 and 4.

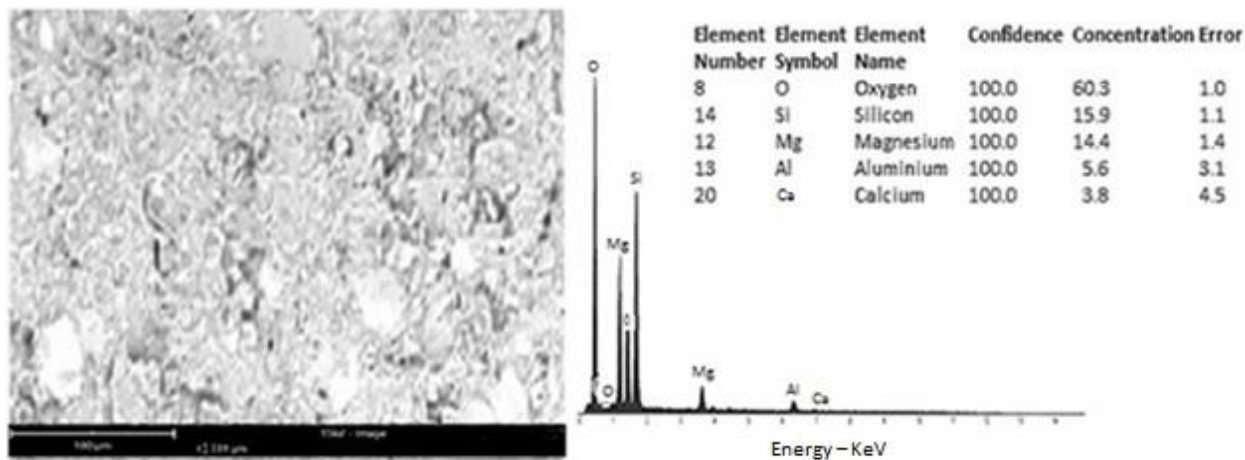


Fig. 5. Micrograph and EDS spectrum of composite sample A.

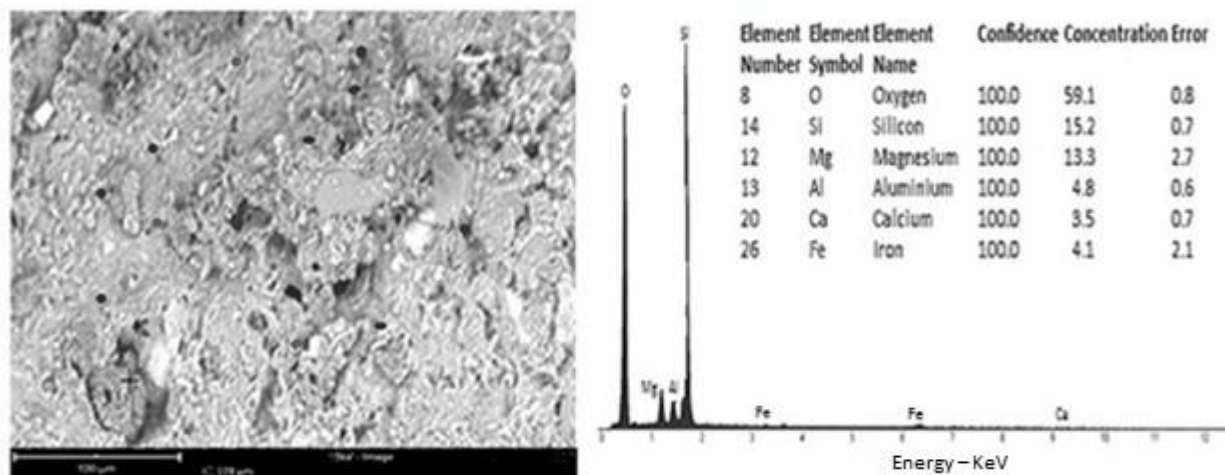


Fig. 6. Micrograph and EDS spectrum of composite sample B.

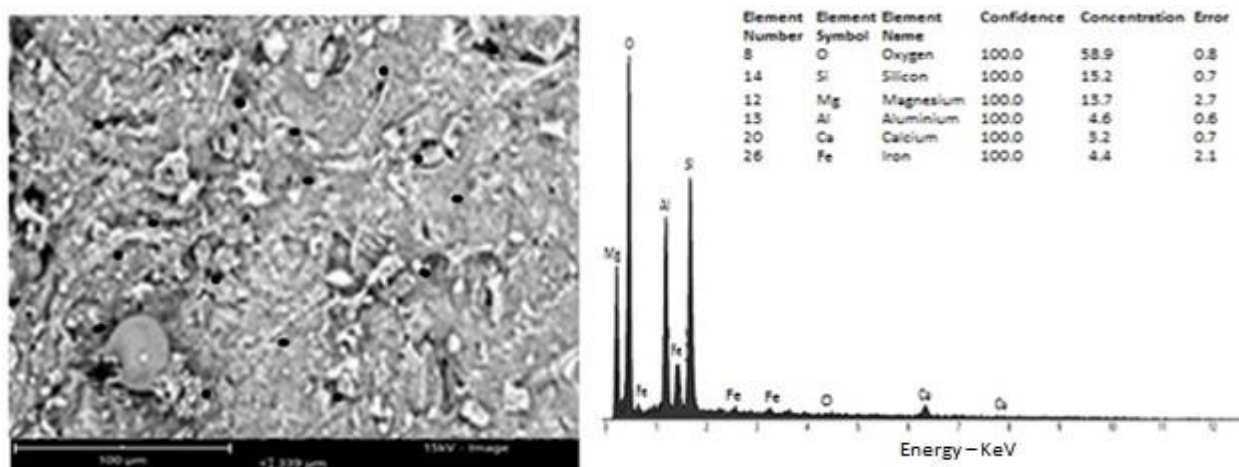


Fig. 7. Micrograph and EDS spectrum of composite sample C.

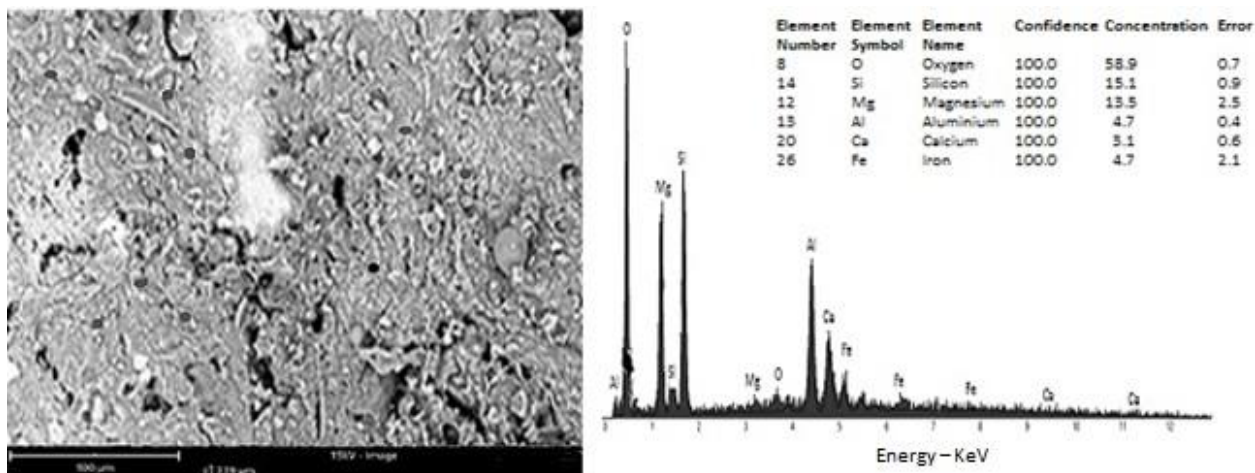


Fig. 8. Micrograph and EDS spectrum of composite sample D.

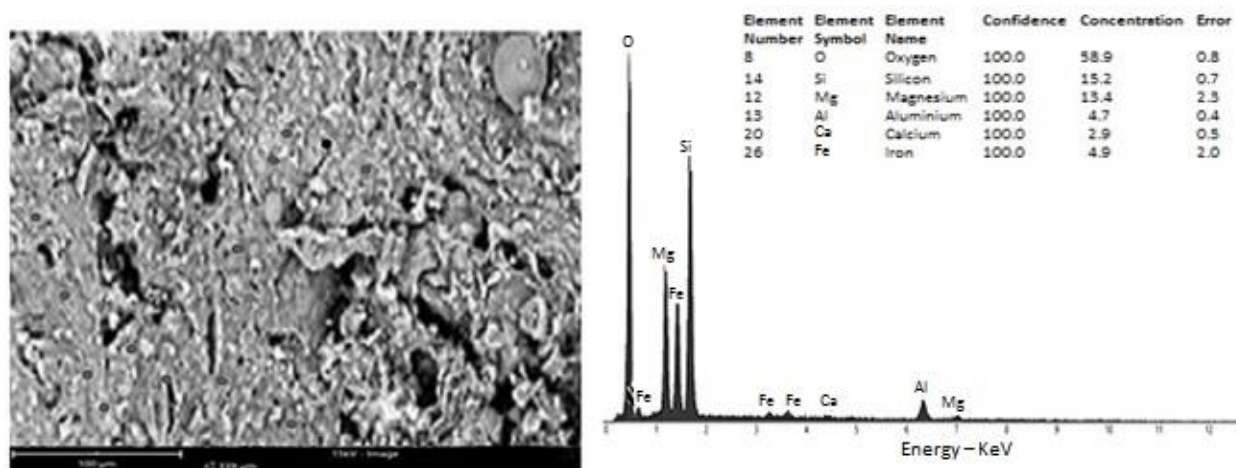


Fig. 9. Micrograph and EDS spectrum of composite sample E.

3.2. Thermal Stability

The mass (weight) losses of decomposition of the ceramic composite samples A, B, C, D and E were evaluated using thermogravimetric method as illustrated in Fig. 10(a – e). The figures show the weight loss percentage as a function of temperature. Sample A which was not blended with iron millscale exhibited the highest weight loss percent of 0.9 while sample D which was blended with 12 wt. % of iron millscale exhibited the lowest weight loss percent of 0.2. There was no loss in weight of the samples from temperature 0 – 1600⁰ C. However, beyond 1600⁰ C to the peak (1800⁰ C), there was weight loss of 0.9 %, 0.7 %, 0.5 %, 0.2 % and 0.3 % in samples A, B, C, D and E respectively. The increase in weight loss of sample E when compared to sample D could be due to weak bonding of the particles. The bonding of the particles of sample E during sintering may be less than that of sample D thereby resulting in higher weight loss.

The onset of thermal decomposition of the samples occurred in the temperature region 1600 – 1800⁰ C. These temperatures were quite high indicating that the composites exhibited high

ability to absorb heat before disintegration implying that they are thermally stable. This may be due to the refractoriness of the ceramic materials coupled with the strong interfacial bonding of the particles. The strong interfacial bonding of the particles effectively acted as barriers to decomposition of the samples during heating, thereby enhancing the thermal stability of the composites. This is similar to what was reported by [Subasinghe et al., \(2016\)](#).

As shown in [Table 3](#), after one hour of exposure to the Bunsen burner flame, none of the samples got burst into flame or charred. Virtually all the samples had the same final weights as the initial weights except sample A which lost 0.01 g representing 0.015 %. This indicates that 99.985 % of the weight of samples A was retained while other samples had 100 % weight (no loss). This shows that all the samples exhibited high ability to absorb heat indicating thermal stability. This may be due to the refractoriness of the ceramic materials coupled with the strong interfacial bonding of the particles. The strong interfacial bonding of the particles effectively acted as barriers to decomposition of the samples during heating, thereby enhancing the thermal stability of the composites. This is similar to what was reported by [Subasinghe et al., \(2016\)](#). In the earlier studies of [Dagwa and Ibadode, \(2006\)](#) and [Idris et al., \(2015\)](#), 43.55 g sample of asbestos based commercial automobile brake pad was left for 10 minutes on the Bunsen burner flame after which the flame was removed. The brake pad did not burst into flame but charred and when weighed, it was 38.64 g. This gave a mass loss of 11.3 % (ash/volatile materials). When compared with the result of this study, the ceramic composites were more thermally stable than the asbestos based commercial brake pad.

3.3. Coefficient of Friction of the Ceramic Composites

Coefficient of friction (COF) is a dimensionless property indicating the friction quality of one material sliding over another. As illustrated in [Fig. 11](#), samples A, B, C, D and E exhibited COF values of 0.52, 0.54, 0.57, 0.59 and 0.59 respectively which indicates that there were asperities between the surfaces of the mild steel inclined plane and samples. The progressive increase in the COF values from sample A – D in the range of 0.52 – 0.59 could be the influence of the increasing weight of samples.

The COF of the composite samples (0.52 – 0.59) is higher than that of the Nigerian industrial standard specification of 0.3 – 0.38 ([NIS 1997](#)) for road vehicles brake linings and pads which suggests that the samples could be more effective because a higher coefficient of friction would offer a better performance. Brake pads must ensure a stable friction with a sufficiently high coefficient of friction. They must not decompose or break down in such a way that the friction

coefficient with the brake disc is not compromised at high temperatures (Djafri et al., 2014; Ikpambese et al., 2016).

The values of the COF of the composite samples is also within the industrial standard of 0.3 – 0.6 (Dagwa and Ibadode, 2006; Aigbodion and Akadike, 2010) recommended for automobiles brake pads.

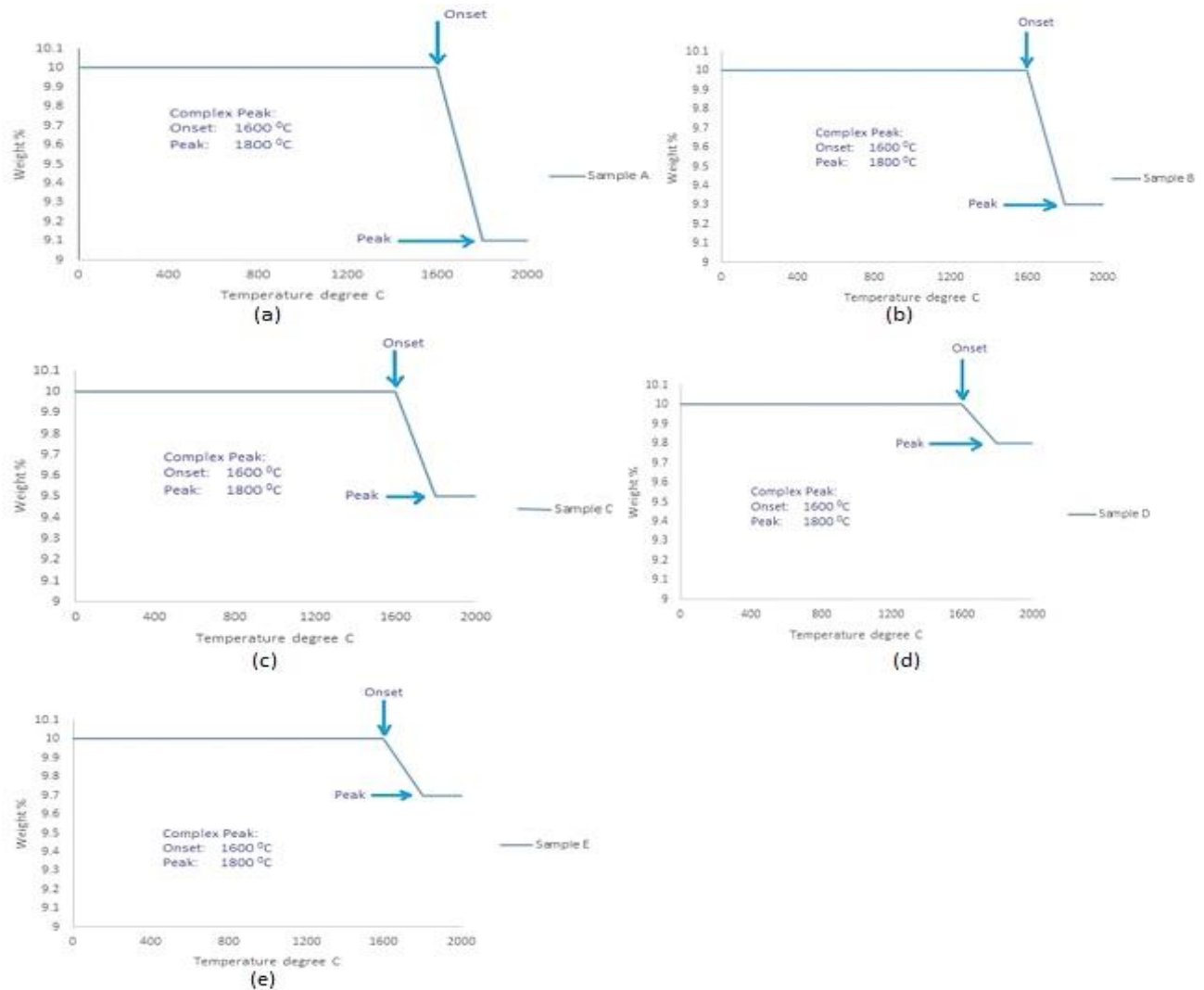


Fig. 10. Thermogravimetric chart of ceramic composite (a) sample A, (b) sample B, (c) sample C, (d) sample D, (e) sample E.

Table 3. Effect of flame on the composite samples.

Sample	Details	Initial Weight (g)	Final Weight (g)	Weight loss (g)	Weight loss (%)	Weight Retained (%)
A	Unreinforced CMC (Control)	67.19	67.18	0.01	0.015	99.985
B	Blended with 4 wt. % millscale	156.53	156.53	-	-	100
C	Blended with 8 wt. % millscale	158.11	158.11	-	-	100
D	Blended with 12 wt. % millscale	159.77	159.77	-	-	100
E	Blended with 16 wt. % millscale	162.25	162.25	-	-	100

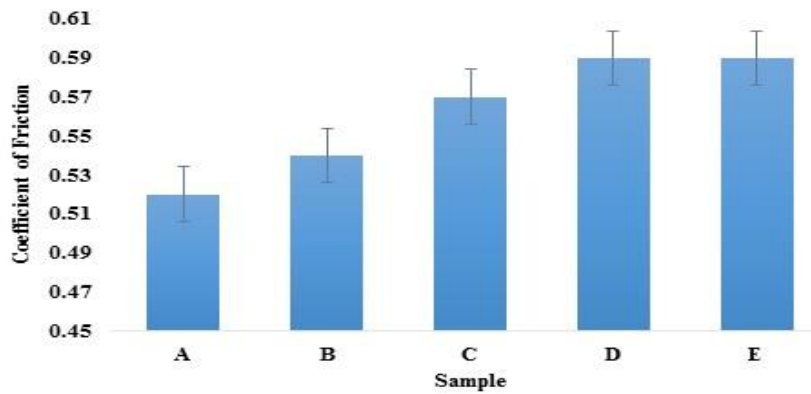


Fig. 11. Coefficient of friction of the different samples.

3.4. Weight Loss and Wear Rate of the Composites

As illustrated in Fig. 12a, there was weight loss in all the composite samples due to plastic deformation of materials on the friction surface as a result of increase in the shear force between asperities and the ploughing force of asperities. Similar observation was also reported by Djafri et al., (2014) and Yawas et al., (2016). The wear rate of the samples shown in Fig. 12b is similar to the trend observed in Fig. 12a because wear rate is directly related to weight loss due to wear. Samples B to E which contain millscale in their formulation (Table 2) exhibited lower weight loss and wear rate than sample A. This indicates the effect of concentration of millscale addition in reducing wear. The lower weight loss and wear rate exhibited by samples B – E could be attributed to the strong interfacial bonding between the particles as shown in the microstructure (Fig. 6 – 9) which reduced the possibility of particle pull out. The decrease in wear rate was also enhanced by the decrease in the number of microstructural flaws which are stress concentrators needed for nucleation and propagation of cracks resulting in removal of materials (Hussainova, 2005). Also, decrease in wear rate of the composites was due to the high hardness and compressive strength values of the composites.

The wear rate of commercial conventional asbestos based brake pad is 3.8×10^{-6} g/m (Olabisi et al., 2016) while the range of wear rate of all the composites obtained in this study is $(1.909 - 2.705) \times 10^{-6}$ g/m. This is much lower than that of asbestos based conventional brake pad and is an indication that the composites have the potential of better performance when used as brake pads.

3.5. Fractography of Worn Surfaces of the Composites

As shown in Fig. 13(a – e), there was formation of grooves on the surfaces of the worn samples as a result of abrasion. The surfaces of the worn samples showed a typical abrasive wear mode in which abrasive particles (debris), scratches, shallow, and deep grooves were formed. The

wear grooves are indication of plastic deformation. A similar observation was reported by Djafri et al., (2014). The findings of this study are also supported by the fact that abrasive wear in contacting surfaces resulted in scratching, furrows, work hardening, or grooves (Holmberg and Matthews, 2009; Kumar and Singh, 2011).

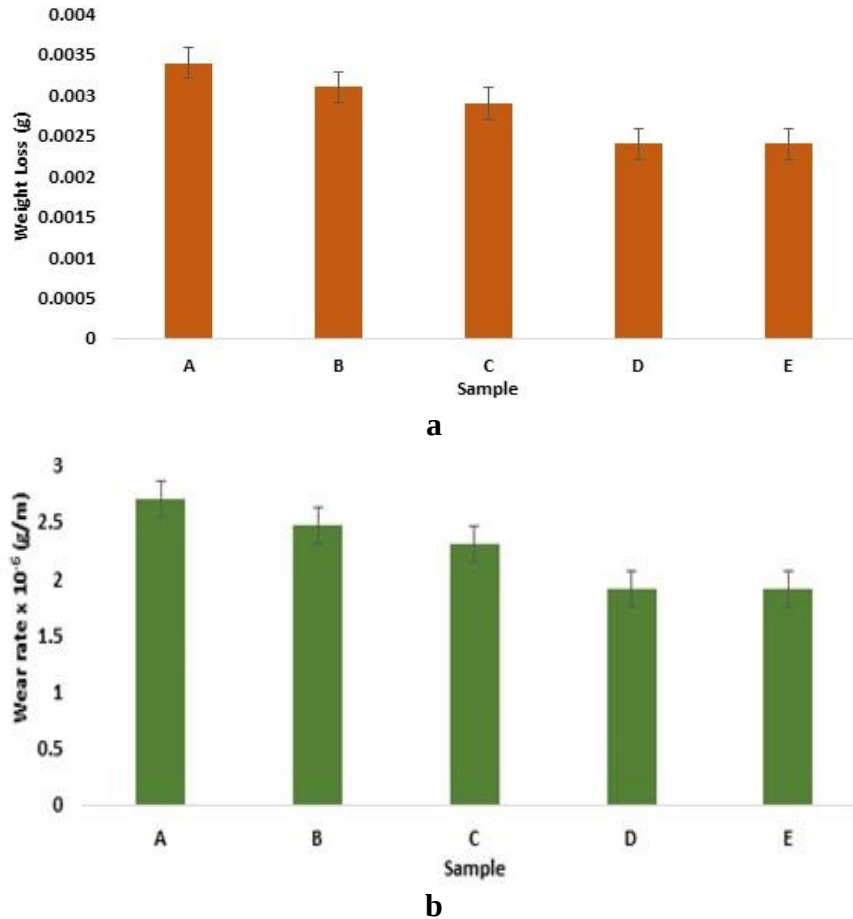


Fig. 12. a. Weight loss due to wear graph of the composite samples; b. Wear rate graph of the composite samples.

The microstructure of worn surface of sample A (Fig. 13a) shows more groove defects than that of Fig. 13(b – e). The worn surfaces appeared to be rough on the microscopic scale. During the wear process, wear debris were generated and they were mainly fine powders that were either released into the environment or trapped between the contact areas. Occasionally, some of the wear debris may also be trapped in any available porous or void area of the samples. The wear debris accumulated and piled up against the contact areas. This phenomenon resulted in the contact areas increasing in size to form a friction layer and patches due to the compaction of wear debris trapped between the sliding surfaces. The debris in the microstructures enhanced the rate of wear of the samples. Similar observations were also reported by Sloan, (2008) and Ruzaidi et al., (2013).

The microstructures of the worn samples revealed that abrasion, adhesion, and grooves formation are the wear types that were in operation during the wear process. Abrasion is always characterised by the presence of shallow grooves or ploughed marks on the friction layers. This wear mechanism was observed in all the microstructures and a similar observation was reported by [Talib et al., \(2008\)](#). This phenomenon could be due to the hard particles of the ceramic materials which existed during the wear process that ploughed into the worn surfaces. Wear of friction materials should be minimized as much as possible. A higher wear rate means shorter friction material life and thus, the incurring of more material and maintenance costs. A lower wear rate would increase the life of the brake pad and a higher friction coefficient would offer a better performance.

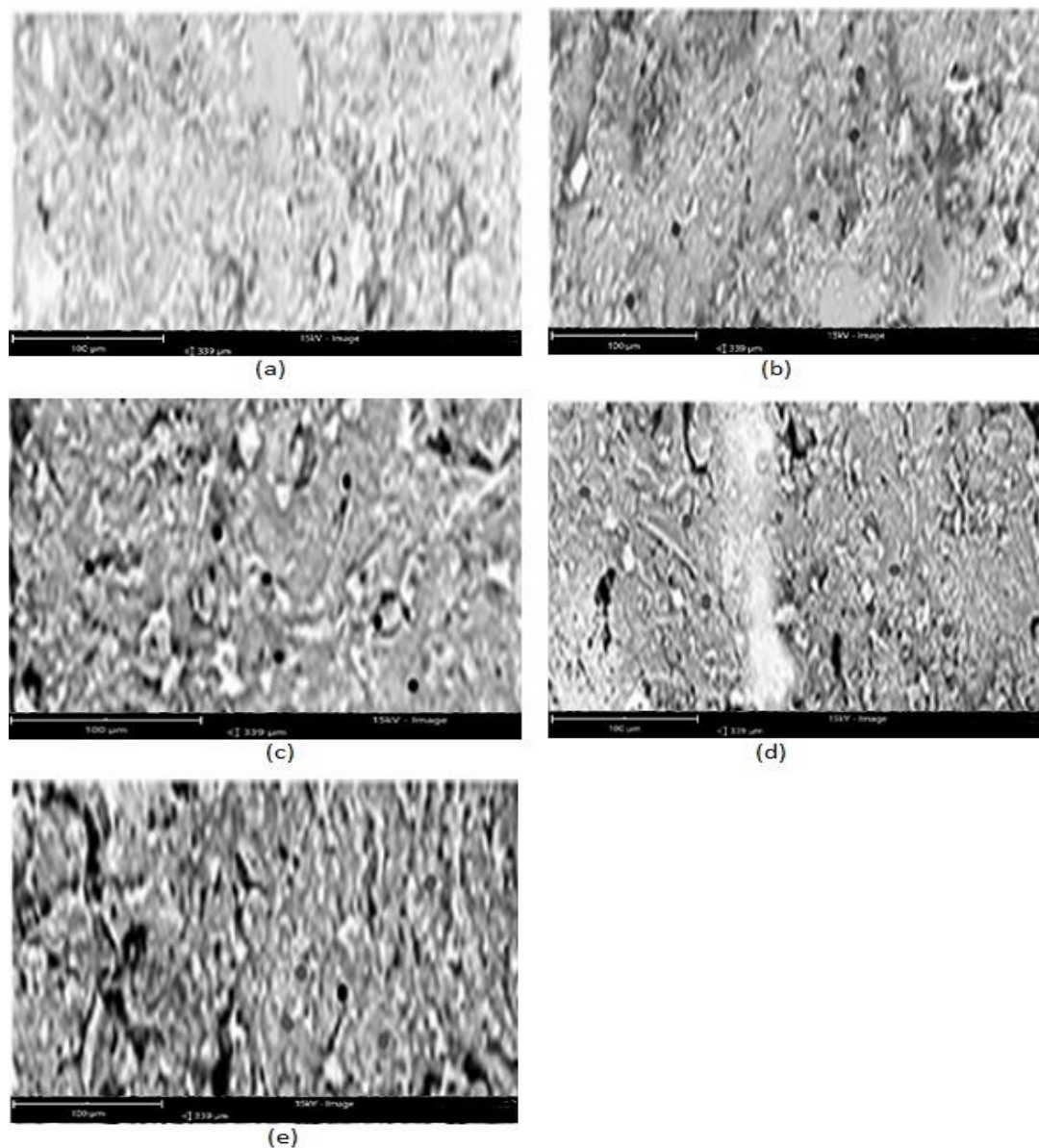


Fig. 13. Microstructure of the worn surface of (a) sample A, (b) sample B, (c) sample C, (d) sample D, (e) sample E.

4. CONCLUSIONS

In this study, the development via powder metallurgy method and characterisation of particulate ceramic matrix composites (PCMCs) have been successfully carried out. The conclusions from the study are highlighted as follows:

1. The ceramic composites exhibited very good properties in terms of high thermal stability (heat resistance), stable coefficient of friction and resistance to wear.
2. The specific values of the properties exhibited by composite sample D in terms of resistance to thermal decomposition in the temperature region (0 – 1600⁰ C), appreciable and high coefficient of friction (0.59) and very low wear rate (1.9093×10^{-6} g/m) are desirable.
3. The uniform dispersion of the particles as observed in the microstructure and strong bonding/adhesion of the particles contributed to the enhancement of the properties.
4. These are indications that the composite is very suitable for application in areas where high resistance to thermal stress and abrasive wear are required such as the brake assembly of automobiles, specifically the brake pads.

5. ACKNOWLEDGEMENTS

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6. REFERENCES

- Adebisi A.A., Maleque M., Shah Q.H. (2011) "Surface temperature distribution in a composite brake rotor" *International Journal of Mechanical and Materials Engineering*, 6(3), 356-361.
- Ademoh N.A. (2015) "Development and evaluation of maize husks (asbestos-free) based brake pad" *Industrial Engineering Letters*, 5(2), 67-80.
- Aigbodion V.S., Agunsoye J.O., Kalu V., Asuke F., Ola S. (2010) "Microstructure and mechanical properties of ceramic composites" *Journal of Minerals & Materials Characterization & Engineering*, 9(6), 527-538.
- Aigbodion V.S., Akadike U. (2010) "Development of asbestos-free brake pad using bagasse" *Tribology in Industry*, 32(1), 45-50.
- Aza C.A. (2014) "Composites in automotive applications: review on brake pads and disc, research development: literature review, Advanced Composites Centre for Innovation and Science (accis), University of Bristol, United Kingdom, 1-14. <http://www.bristol.ac.uk/engineering/media/accis/cdt/news/aza.pdf>.
- Bark L.S., Moran D., Percival S.J. (1975) "Chemical changes in asbestos based friction materials during performance - a review" *Wear*, 34, 131-139.

- Dagwa I.M., Ibhadowe A.O. (2006) "Some physical and mechanical properties of asbestos free experimental brake pad" *Journal of Raw Materials Research (Jormar)*, 3(2), 94-103.
- Dan-Asabe B., Madakson P.B., Manji J. (2012) "Material selection and production of a cold-worked composite brake pad" *World Journal of Engineering and Pure and Applied Science (WJEPAS)*, 2(3), 96.
- Djafri M., Bouchetara M., Busch C., Weber S. (2014) "Experimental study of the tribological behaviour of materials of brake" *MECHANIKA*, 20(4), 420-425.
- Essays UK. (2018) "Experiment to Determine the Coefficient of Friction" <https://www.ukessays.com/essays/physics/experiment-determine-coefficient4293.php?vref=1>
- Herring J.M. (1990) "SAE Automotive Engineers Congress. Society of Automotive Engineers" Paper Number 670146, Detroit, USA.
- Holmberg K., Matthews A. (2009) "Coatings tribology, properties, mechanisms, techniques and applications in surface engineering" (Briscoe, B.Ed.), *Tribology and Interface Engineering*, 2nd edition, Series 56, 560.
- Hussainova I. (2005) "Microstructure and erosive wear in ceramic-based composites" *Elsevier, Wear*, 258, 357-365.
- Idris U.D., Aigbodion V.S., Abubakar I.J., Nwoye C.I. (2015) "Eco-friendly asbestos free brake-pad using banana peels" *Journal of King Saud University-Engineering Sciences*, 27, 185–192.
- Ikpambese K.K., Gundu D.T., Tuleun L.T. (2016) "Evaluation of palm kernel fibers (PKFs) for production of asbestos-free automotive brake pads" *Journal of King Saud University-Engineering Sciences*, 28, 110–118.
- Jafar T.R., Selamat M.S., Kasiran R. (2012) "Selection of best formulation for semi-metallic brake friction materials development" *Powder Metallurgy*, 1-30.
- Kchaou M., Sellami A., Elleuch R., Singh H. (2013) "Friction characteristics of a brake friction material under different braking conditions" *Elsevier, Materials and Design*, 52, 533-540.
- Kumar A., Singh S. (2011) "Wear property of metal matrix composite" B.Tech. Thesis, National Institute of Technology, Rourkela, Odisha 769008, India, 1-45.
- Lee P.W., Filip P. (2013) "Friction and wear of Cu-free and Sb-free environmental friendly automotive brake materials" *Wear*, 302, 1404-1413.
- Ma Y, Martynkova G.S., Vala S.M., Matejka V., Lu Y. (2012) "Effects of ZrSiO₄ in non-metallic brake friction materials on friction performance" *Tribology International*, 41(3), 166-174.

- Maleque M.A., Dyuti S., Rahman M.M. (2010) "Material selection method in design of automotive brake disc" *Proceedings of the World Congress on Engineering (WCE)*, 3, 1-5.
- McPhee A.D., Johnson D.A. (2007) "Experimental heat transfer and flow analysis of vented brake rotor" *International Journal Thermal Science*, 47(4), 458–467.
- Nigerian Industrial Standards, (1997) "Specification for friction materials for road vehicles brake lining and pads" *Standard Organisation of Nigeria (SON)*, NIS 323:1997.
- Olabisi A.I., Ademoh N.A., Okechukwu O.M. (2016) "Development and assessment of composite brake pad using pulverized cocoa beans shells filler" *International Journal of Materials Science and Applications*, 5(2), 66-78.
- Ruzaidi C.M., Kamarudin H., Shamsul J.B., Mustafa, A.M., Liyana, J. (2013) "Mechanical properties and morphology of palm slag, calcium carbonate, and dolomite filler in brake pad composites" *Applied Mechanics and Materials*, Trans Tech Publications, 313-314, 174-178.
- Sellami A., Kchaou M., Elleuch R., Cristol AL., Desplanques Y. (2014) "Study of the interaction between microstructure, mechanical and tribo-performance of a commercial brake lining material" *Elsevier, Materials and Design*, 59, 84–93.
- Singh T., Patnaik A., Chauhan R., Rishiraj A. (2015) "Assessment of braking performance of lapinus-wollastonite fiber reinforced friction composite materials" *Journal of King Saud University- Engineering Sciences*, Article in press.
- Sloan M.R. (2008) "Eco-efficient friction materials" *Ph.D. Thesis*, University of Exeter, United Kingdom, 1-107.
- Straffelini G., Ciudin R., Ciotti R., Gialanella S. (2015) "Present knowledge and perspectives on the role of copper in brake materials and related environmental issues: a critical assessment" *Environmental Pollution*, 207, 211-219.
- Subasinghe A., Das R., Bhattacharyya D. (2016) "Study of thermal, flammability, and mechanical properties of intumescent flame retardant pp/kenaf nanocomposites" *Taylor & Francis Group, International Journal of Smart and Nano Materials*, 7(3), 202–220.
- Talib R.J., Azimah M.A.B., Yuslina J., Arif S.M., Ramlan K. (2008) "Analysis on the hardness characteristics of semi-metallic friction materials" *Journal of Solid State Science& Technology*, 16(1), 124-129.
- Tanaka K., Ueda S., Noguchi N. (1973) "Fundamental studies on the brake friction of resin based friction materials" *Wear*, 23, 349-365.
- Yawas D.S., Aku S.Y., Amaren S.G. (2016) "Morphology and properties of periwinkle shell asbestos-free brake pad. *Journal of King Saud University-Engineering Sciences*, 28, 103-109.