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TECHNICAL NOTE

Morphology and properties of periwinkle shell asbestos-free brake pad



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Abstract The development of asbestos-free automotive brake pad using periwinkle shell particles as frictional filler material is presented. This was with a view to exploiting the characteristics of the periwinkle shell, which is largely deposited as a waste, in replacing asbestos which has been found to be carcinogenic. Five sets of brake pads with different sieve size (710–125 μm) of periwinkle shell particles with 35% resin were produced using compressive moulding. The physical, mechanical and tribological properties of the periwinkle shell particle-based brake pads were evaluated and compared with the values for the asbestos-based brake pads. The results obtained showed that compressive strength, hardness and density of the developed brake pad samples increased with decreasing the particle size of periwinkle shell from 710 to 125 μm , while the oil soak, water soak and wear rate decreased with decreasing the particle size of periwinkle shell. The results obtained at 125 μm of periwinkle shell particles compared favourably with that of commercial brake pad. The results of this research indicate that periwinkle shell particles can be effectively used as a replacement for asbestos in brake pad manufacture.

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1. Introduction

Brake pads are an important part of braking systems for all types of vehicles that are equipped with disc brakes. Brake pads are steel backing plates with a friction material bound to the surface facing the brake disc (Anderson, 1992; Aigbodion et al., 2010). Brake pads convert the kinetic energy

of the car to thermal energy by friction. When a brake pad is heated up by coming into contact with either a drum or rotor, it starts to transfer small amounts of friction material to the disc or pad (that is the reason a brake disc is dull grey). The brake rotor and disc (both now with friction material on), will then “stick” to each other to provide stopping power. The friction of the pad against the disc is however responsible for the majority of stopping power. In disc brake applications, there are usually two brake pads per disc rotor, held in place and actuated by a calliper affixed to a wheel hub or suspension upright (Bhabani and Bijwe, 2005; Dagwa and Ibadode, 2005).

The brake pads presently used are generally made from asbestos fibre. Asbestos was widely used in pads for its heat resistance. In spite of its good properties asbestos is being withdrawn from all those applications where there is a

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possibility of man consuming or inhaling its dust, because of its carcinogenic nature. Due to this health risk, it is necessary to use alternative material for making non-carcinogenic brake pad (Dagwa and Ibhádode, 2006; Kallurin et al., 2008).

Dagwa and Ibhádode (2005) developed asbestos free friction lining material from palm kernel shell. In the study the mechanical and physical properties as well as the static and dynamic performance compared well with commercial asbestos-based lining material.

Olerie et al. (2007) investigated the changes induced by repeated brake applications and how much material modification affected friction and wear properties of the automotive brake disc. Surface films were investigated by transmission electron microscopy (TEM) after preparing thin cross-sections with a focused beam instrument (FIB). Since the observed friction layers revealed nanocrystalline structure, modelling with the method of movable cellular automata (MCA) was performed by assuming an array of linked nanometre size particles. It was observed that with a size of 10 nm, the coefficient of friction (COF) for oxide – on-oxide and metal – on – metal contact was 0.35 and 0.85, respectively.

Seong and Ho (2000) studied the friction and wear of friction materials containing two different phenolic resins reinforced with aramid pulp, investigated the friction and wear characteristics of automotive materials containing two different phenolic resins (a straight resin and a modified novolac resin) using a pad-on-disc type friction tester.

Peter et al. (2001) worked on friction layer formation in a polymer composite material for brake applications. The characterization of the properties of polymer matrix composite friction layer was carried out.

Satam and Bijwe (2004) carried out a study of the friction materials based on the variation in the nature of organic fibre fade and recovery behaviour. They investigated the influence of four selected organic fibres viz, Aramid (AF), PAN (polyacrylo-nitrile), carbon (COF) and cellulose (SF), on the N-fade and N-recovery behaviour.

Smart and Chugh (2007), worked on the development of fly-ash based automotive brake linings. They developed friction composite using fly ash obtained from a specific power plant in Illinois. Additives such as phenolic resin, aramid pulp, glass fibre, potassium titanate, graphite aluminium fibre and copper powder were used in the composite development phase in addition to fly ash. The developed brake lining composites exhibited consistent coefficient of friction in the range of 0.35–0.4 and wear rates lower than 12 wt%.

Recently Aku et al. (2012), studied the characterization of periwinkle shell as asbestos-free brake pad materials. They found out that periwinkle shell is an agricultural waste. The waste is produced in abundance globally and poses risks to the humans as well as environmental health. Thus their effective, conducive, and eco-friendly utilization has always been a challenge for scientific applications. The characterization of the periwinkle shell was investigated through X-ray diffractometer (XRD), thermogravimetric analysis (TGA/DTA), Fourier transform infra red density, hardness values and wear rate of the periwinkle shell were also found. There are various results obtained comparable with asbestos commonly used in brake pad production. Based on this background this work aimed at determining the morphology and properties of brake pad produced from a periwinkle shell waste.

2. Materials and methods

2.1. Materials/equipment

The materials and equipment used during the course of this work are: Phenolic resin (phenol formaldehyde), periwinkle shell, Engine oil (SEA 20/50), water, hydraulic press, brake pad mould, heater, cups, vernier calliper, grinder, digital weighing balance, hounsfield tensometer, hardness tester, scanning electron microscope (SEM), Denver cone crusher, Denver roll crusher, Denver ball milling machine, and a set of sieves of 125, 250, 355, 500 and 710 μm , digital weighing machine, digital balance pin on disc machine.

2.2. Method

The periwinkle shell was sun dried, followed by oven drying at 105 °C for 5 h until the moisture is ensured to have greatly reduced towards zero percent. This was then charged into Denver cone crusher and was reduced to between 4 and 3 mm. This was further charged into roll crusher that now reduces the size of periwinkle shell to between 2 and 1 mm (see Fig. 1).

The product of roll crusher was transferred into ball milling machine and was left in the mill for two (2) hours; after which the product was transferred into a set of sieves of; +710, +500, +355, +250, and +125 μm , and was sieved for 30 min using a sieve shaker machine for 30 min. While the oversize at +710 μm was returned or recycled for regrinding until it passes through the sieves.

The periwinkle shell sieve sizes of 125, 250, 335, 500 and 710 μm , were mixed with the phenolic (35%). Thirty (30) test samples from each of the sieve size were then produced. Each composition was blended homogeneously in a mixer for a period of five minutes before transferring it to a mould kept at a pressure of 40 kg/cm² at a temperature of 160 °C for 1.5 h. The samples were post cured at 140 °C for 4 h.

Density measurements carried out the formulated brake pad using Archimedes's principle. The buoyant force on a submerged object is equal to the weight of the fluid displaced. This principle is useful for determining the volume and therefore the density of an irregularly shaped object by measuring its mass in air and its effective mass when submerged in water (density = 1 g/cc). This effective mass under water was its actual mass minus the mass of the fluid displaced. The difference between the real and effective mass therefore gives the mass of



Figure 1 Photograph of periwinkle shell powder.

water displaced and allows the calculation of the volume of the irregularly shaped object. The mass divided by the volume thus determined gives a measure of the average density of the sample (Aigbodion et al., 2010).

The microstructure and the chemical compositions of the phases present in the test samples were studied using a JOEL JSM 5900LV scanning electron microscope. The SEM was operated at an accelerating voltage of 5–20 kV. The test was carried out in the University of the Witwatersrand, Johannesburg, South Africa

The 24-h water and oil soak test determines the water and oil absorption behaviour of the asbestos-free experimental brake pad and the effect of the absorbed water and oil on its dimensions. After oven drying of the specimens (0–25 °C), its weight was measured. Subsequently, the dimensions (thickness) of the specimens were measured using a vernier calliper after twenty-four hours of submersion in water and engine oil, SEA 20/50 at 26–30 °C. The specimens were weighed after the excess water and oil had drained off. Eq. (1) was used in calculation of the percentage of water and oil absorption (Dagwa and Ibadode, 2006):

$$\text{Absorption(\%)} = \frac{W_1 - W_0 \times 10\%}{W_0} \quad (1)$$

W_1 = is the weight after immersion.

W_0 = the weight before immersion.

The Brinell hardness values were obtained using a digital hardness tester. The material of diameter 22.7 was used to carry out the test samples. The hardness values were determined according to the provisions in American Society of testing and materials (ASTM E18-79) using the Brinell hardness tester on “B” scale (Frank Welltest Brinell Hardness Tester, model 38506) with 1.56 mm steel ball indenter, minor load of 10 kg, major load of 100 kg and hardness value of 101.2HRB as the standard block (Aigbodion et al., 2010).

The compression test was conducted on Avery Denison strength testing machine of 500KN capacity with a strain rate of 0.002 s⁻¹. The sample was locked securely in the grips of the upper and lower crossbeams of the testing machine. A small load was initially applied to seat the sample in the grips and then the load was increased until failure occurred.

A pin-on-disc test apparatus was used to investigate the dry sliding wear characteristics of the samples as per ASTM G99-95 standards (Aigbodion et al., 2010). Wear sample 20 mm in diameter and 40 mm in height was used. Wear tests were conducted with loads ranging from 5 to 20 N, speed of 5.02 m/s and constant sliding distance of 5000 m.

3. Results and discussion

Macrostructural studies of the samples revealed a uniform distribution of periwinkle shell particles and the resin. The distribution of particles is influenced by good bonding of the resin and the periwinkle shell particles which resulted in good interfacial bonding (see Fig. 2). During the production of the formulation of the brake pad it was observed that proper bonding was achieved when decreasing the sieve size from 710 to 125 μm.

The results of density of the samples are shown in Fig. 3, the density of the samples increased as the sieve size decreased

from 710 to 125 μm. This can be attributed to the increases in bonding achieved i.e. increased packing of the particles. The 125 μm has the highest density which is a result of closer packing of periwinkle shell particles creating more homogeneity in the entire phase of the composite body. The levels of density obtained are within the recommended values for brake pad application (Hooton, 1969). The lower density shows that the periwinkle shell-based brake pad would be lighter than the asbestos brake pad. The result is in par with the earlier work of Aigbodion et al. (2010).

The surface morphology of the new pad materials was analysed using SEM followed by elemental analysis of the materials using EDS. Figs. 4–8 showed the SEM/EDS micrographs of the developed brake pad. The resin binder in the dark region can be seen in Figs. 4–8 along with periwinkle shell particle distribution in the white region. From the SEM study it can be postulated in general that microstructures of samples showed the homogeneous distribution as the sieve size of the periwinkle shell particles decreased (compare Fig. 4 with Fig. 8).

The EDS analysis results showed a very good combination of the elemental distribution which reflects on the heterogeneous distribution. Different sizes of particles or elements are also contributed to heterogeneous distribution. An overall observation shows the mixture of the brake pad materials is well dispersed even though there are some traces of pores due to the different particle sizes of each admixture material. It is confirmed that the presence of pores is controlled by the content of particle size. The pores decreased with a decreasing content of particle size. Good interfacial bonding between the resin and periwinkle shell particles was seen, this bonding increased as the particle size decreased. This is a result of proper bonding between the periwinkle shell particles and the resin as the sieve grade decrease and also closer inter-packing distance. This can be appreciable if one compares Fig. 4 with Fig. 8.

Figs. 9 and 10 show the water, oil soak absorption of the produced samples. These properties decreased as the particle size of periwinkle decreases, this may be attributed to the decrease in pores because of the close interface packing achieved (see Figs. 4–8). Also increased interfacial bonding between the resin and the periwinkle shell particles will lead to a decrease in the porosity level, hence the solubility values of the brake pad composites.

The swelling that occurs during the water and oil absorption is the sum of two components, namely, swelling by hygroscopic particles and the release of compression stresses imparted to the brake pad composites during the pressing of mat in the hot press. The release of compression stresses, known as springback, is not recovered when the brake pad composites are in a dry state. The lower result values obtained from 250 to 125 μm periwinkle shell particles resulted in more resistance to absorbed water and blocked the void and decreased the porosity level.

Fig. 9, shows the graph for water absorption for different formulations of brake pad. From the graph, 710 μm shows the higher value while 125 μm shows the lower value of water content. Higher sieve size resulted in more water absorption because more pores are observed. This result was influenced by porosity and void formed in the brake pad samples. The water absorption decreased when decreasing the particle size of the periwinkle shell content. Theoretically, lower water absorbed to the brake pad will result in higher friction coefficient



Figure 2 Photograph of the test samples.

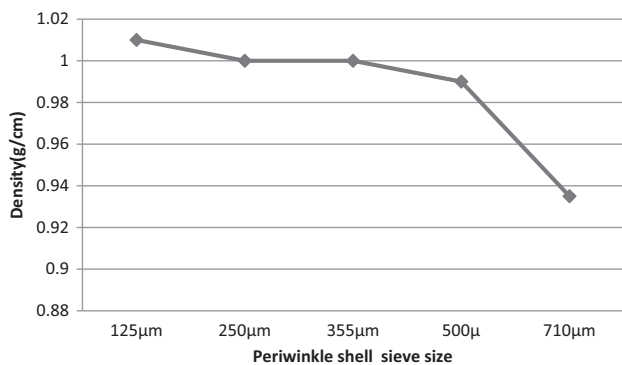


Figure 3 Variation of density with different sieve sizes of periwinkle shell particles.

and wear rate due to higher contact areas between the mating surfaces. The results obtained for the formulation of brake pad composites at 250–125 µm periwinkle shell particles are within the recommended standard (see Table 1). These results are in par with the earlier observation of the work of other researchers (Aigbodion et al., 2010; Dagwa and Ibadode, 2005; Kallurin et al., 2008).

The hardness values of the samples are shown in Fig. 11. From the results it is clear that the hardness increased with decreasing the particles size of periwinkle shell. The sample with 125 µm sieve grade has the highest hardness values of 112.5HBN. A decrease in hardness values was observed in the samples with higher sieve grades (250, 355, 500 and 710 µm). The high hardness values for the 125 µm sieve grade

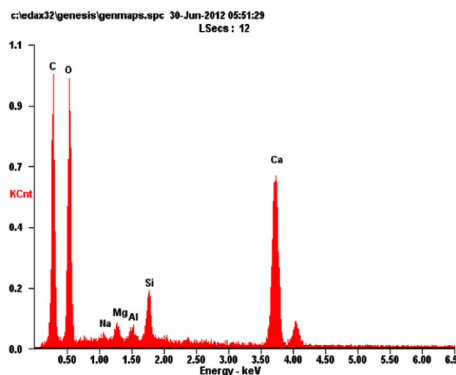
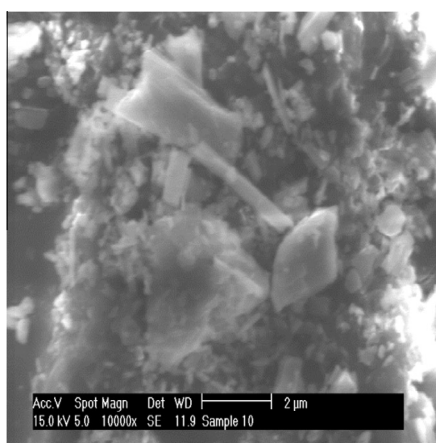


Figure 4 SEM/EDS microstructure of developed brake pad with 710 µm periwinkle shell particles.

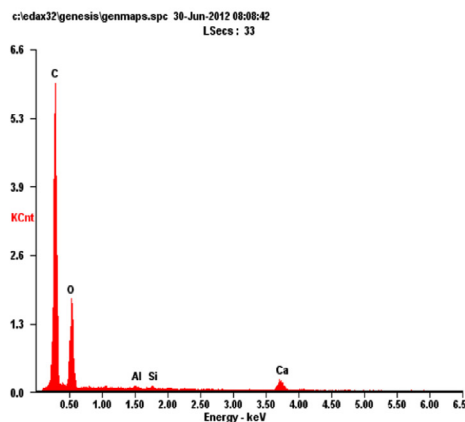
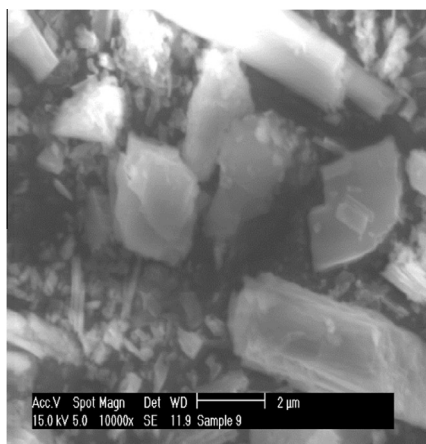


Figure 5 SEM/EDS microstructure of developed brake pad with 500 µm periwinkle shell particles.

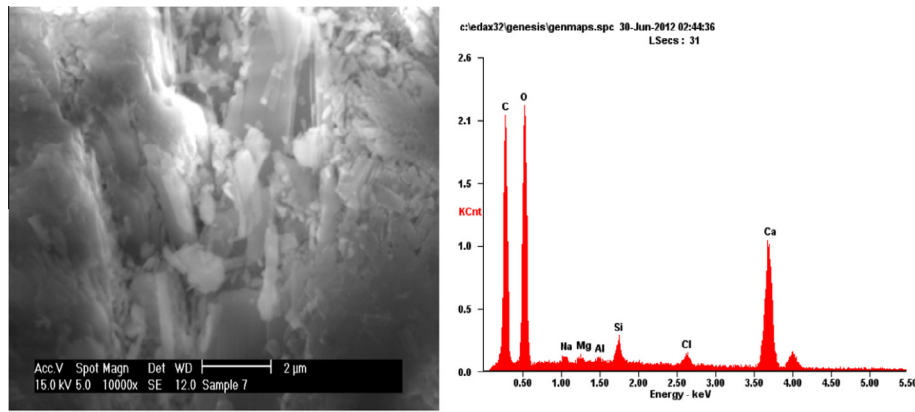


Figure 6 SEM/EDS microstructure of developed brake pad with 355 µm periwinkle shell particles.

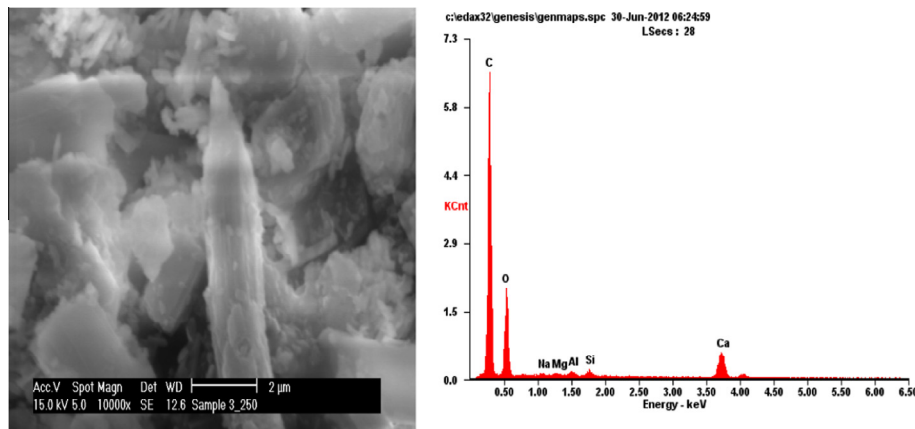


Figure 7 SEM/EDS microstructure of developed brake pad with 250 µm periwinkle shell particles.

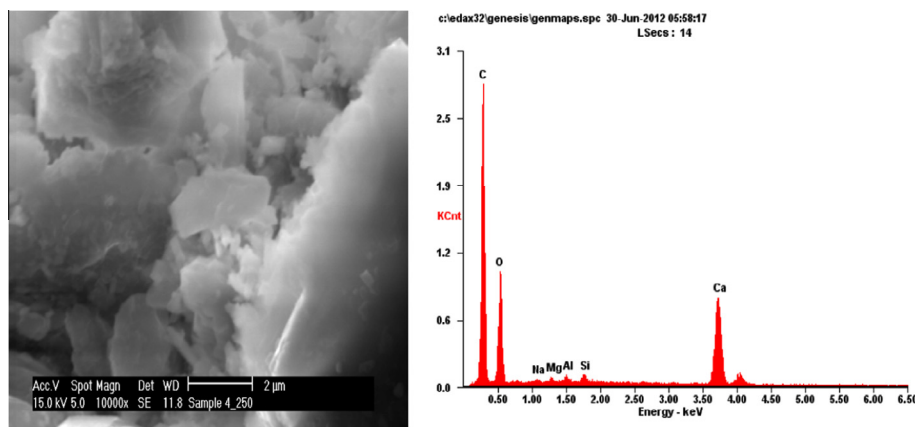


Figure 8 SEM/EDS microstructure of developed brake pad with 125 µm periwinkle shell particles.

was a result of reduced particle size of periwinkle shell i.e. an increase in surface area which resulted in an increase in bonding ability with the resin. The hardness values for this material were compared with other materials from other researches as shown in [Table 1](#) which indicated an acceptable result with the findings of other researchers. It was found that the hardness values of periwinkle shell particles based brake pad are higher than that of asbestos at 125 µm size (Aigbodion et al.,

2010). This is probably due to the presence of Fe_2O_3 , CaO and SiO_2 of the chemical made up of periwinkle shell particles (Aku et al., 2012).

Compressive properties of the samples are presented in [Fig. 12](#). It was clear that the compressive strength of the developed brake pad increased as the particle size of periwinkle shell. This may be attributed to the hardening of the resin by periwinkle shell particles (Smart and Chugh, 2007). Brake

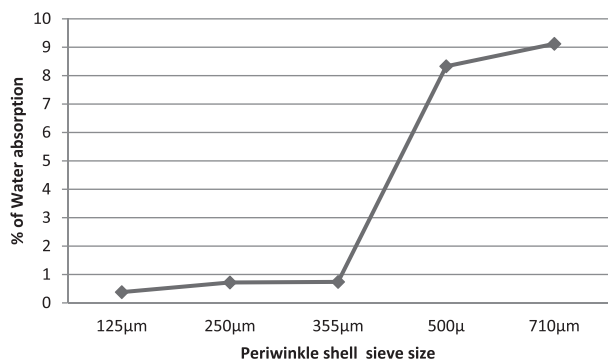


Figure 9 Variation of thickness swelling in water with different sieve sizes of periwinkle shell particles.

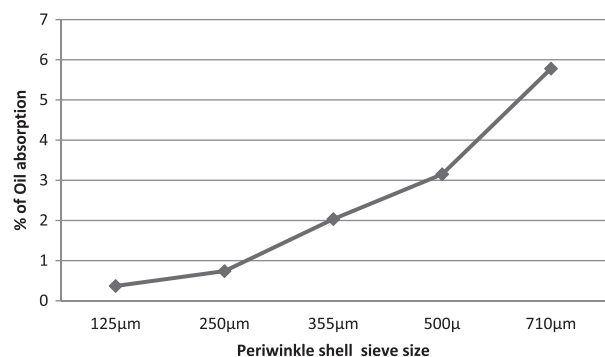


Figure 10 Variation of thickness swelling in oil with different sieve sizes of periwinkle shell particles.

pad formulation with 125 µm periwinkle shell particles showed the highest compression strength compared to other sieve size used. The less pores and the more compact mixture in 125 µm periwinkle shell particles showed a significant effect on the compression strength, and more pores in high sieve size decreased the compression strength of the brake pad.

Fig. 13 shows the variation of wear rate of the developed brake pad.

It can be seen that as the load increases, the wear of developed brake pad also increased (see Fig. 13). The wear of the 710 µm particle size is more than that of the other samples. However the wear rate decreases with decreasing the periwinkle shell particle content. From Fig. 13, the positive effect of the periwinkle shell particle size in reducing the wear rate of materials can be seen. When the load applied is low, the wear loss is quite small, which increases with an increase in applied load. It is quite natural for the wear rate to increase with applied load.

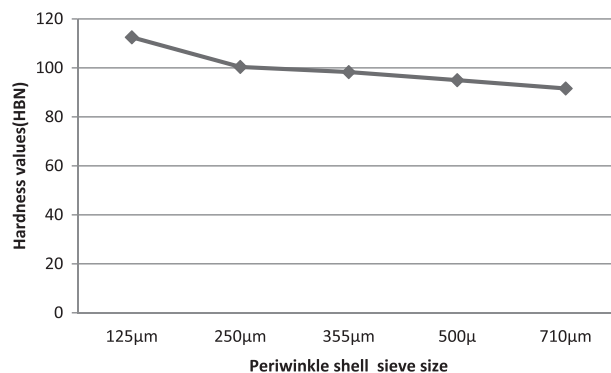


Figure 11 Variation of hardness values with different sieve sizes of periwinkle shell particles.

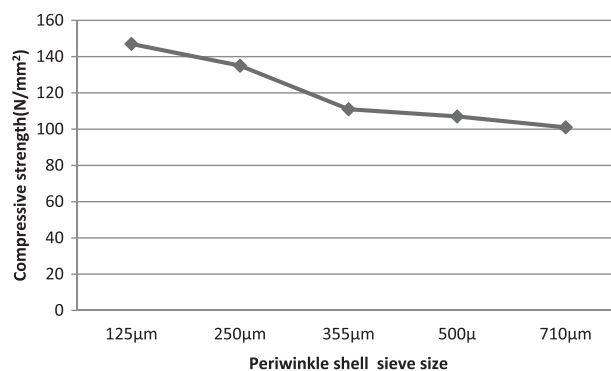


Figure 12 Variation of compressive strength with different sieve sizes of periwinkle shell particles.

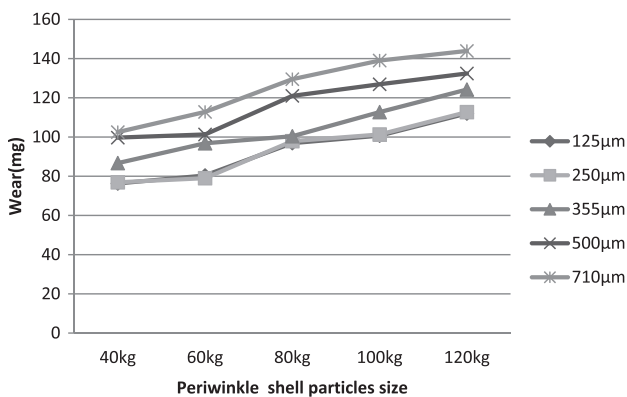


Figure 13 Wear (mg) at varying loads at a constant speed of 5.06 m/s.

Table 1 Summary of result findings compared with existing ones.

Properties	Commercial brake pad (asbestos based)	Laboratory formulation (Palm kernel shell)	Laboratory formulation (Bagasse)	New laboratory formulation (periwinkles shell at 125 µm)
Specific gravity (g/cm ³)	1.89	1.65	1.43	1.01
Friction coefficient	0.3–0.4	0.440	0.420	0.35–0.41
Thickness swell in water (%)	0.9	5.03	3.48	0.39
Thickness swell in SEA oil (%)	0.30	0.44	1.11	0.37
Hardness values (HRB)	101	92.0	100.5	116.7
Compressive strength (N/mm ²)	110	103.5	105.6	147

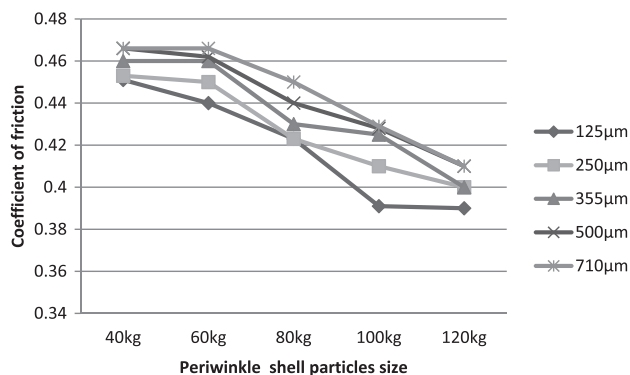


Figure 14 Coefficient of friction at varying loads at a constant speed.

A similar trend was also observed independently for different wear distances as a function of load and speed (Aigbodion et al., 2010). Consequently, the effect of particle size on the brake pad composite wear resistance is better for low loads. With higher loads contact temperatures become high and plastic deformation occurs with consequence of very high wear.

Fig. 14 shows the variation of friction coefficient with different applied loads. The coefficient of friction of the samples decreases as the applied load increases, and also decreases with an increase in periwinkle shell particle size.

The decrease in wear rate of the periwinkle based brake pad composites may also be attributed to higher load bearing capacity of hard material and better interfacial bond between the particle and the resin reducing the possibility of particle pull out which may result in higher wear (see Figs. 4–8). The improvement in wear resistance accompanying the presence of decreasing particle size of the periwinkle shell in the resin is due to an increase in average hardness values and compressive strength. The wear rate obtained for these brake pads composite at 125–250 µm fall within the automotive standard ranges for the production of automotive brake pad (Hooton, 1969).

The developed brake pads with 125 µm periwinkle shell particle size provided greater friction coefficient than the 710 µm periwinkle shell particle size but offered lower wear rate than the 125 µm periwinkle shell particle size. Increases in friction coefficient do not tend to have higher wear rate, the above results are consistent with the one reported by Hooton (1969). The friction coefficient falls within the industrial standard ranges of 0.3–0.45 for automotive brake pad system (Satam and Bijwe, 2004).

The results of this work indicate that sample containing 125 µm periwinkle shell particles gave better properties than other samples tested. Hence, the decreasing periwinkle shell particles in the resin lead to better properties. The result of formulation of 125 µm periwinkle shell particles were compared with that of commercial brake pad (asbestos based) and optimum formulation laboratory brake pad (Palm Kernel Shell based (PKS)) and Bagasse as shown in Table 1, which were tested under similar conditions (Aigbodion et al., 2010; Dagwa and Ibhado, 2005).

4. Conclusions

From the results and discussion in this work the following conclusions can be made:

1. Periwinkle shell particle brake pad was successfully developed using a compressive moulding.
2. There was good interfacial bonding as the particle size of periwinkle shell was decreased from 710 to 125 µm.
3. Compressive strength, hardness and density of the developed brake pad samples were seen to be increasing with decreasing the particle size of periwinkle shell from 710 to 125 µm, while the oil soak, water soak and wear rate decreased with decreasing the particle size of periwinkle shell.
4. The sample containing 125 µm of periwinkle shell particles gave the best properties in all.
5. The results obtained at 125 µm of periwinkle shell particles compared favourably with that of commercial brake pad. The results of this research indicate that periwinkle shell particles can be effectively used as a replacement for asbestos in brake pad manufacture.

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