

Production of Asbestos Free Brake Pad Using Periwinkle Shell as Filler Material

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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 (100-350 μ m) of periwinkle shell particles with 13% resin were produced using compressive molding. The physical and mechanical 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 350-100 μ m, while the oil soak, water soak and wear rate decreased with decreasing the particle size of periwinkle shell. The results obtained at 100 μ m of periwinkle shell particles compared favorably 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.

Keywords—Compressive strength, Flame Resistance, Hardness, Micro-structure, Porosity and Wear

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 [1]. 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 caliper affixed to a wheel hub or suspension upright [2,3]. The brake pads presently used are generally made from asbestos fiber. The major component in the brake pad is the lining materials, which are categorized as metallic, semi-metallic, organic and carbon-based, depending on the composition of the constituent elements. Typical formulations consist of more than 10 ingredients, and more than 300 materials are in different brands [4]. These ingredients are classified into four broad groups: binders, reinforcing fibres or structural materials, fillers, and frictional additives/modifiers, based on the major function they perform apart from controlling friction and wear performance. The binders hold the ingredients together, to maintain structural integrity of the brake lining

under varying mechanical and thermal stresses. The structural materials provide the structural reinforcement to the composite matrix; fillers make up the volume of the brake lining, while keeping the cost down; and friction modifiers stabilize the coefficient of friction and wear rates. These components perform synergistically in controlling friction and wear performance of the brake pad.

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 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 [6].

1.1 Description of Periwinkle

The common periwinkle or winkle (*Littorina littorea*) is a species of small edible sea snail, a marine gastropod mollusc that has gills and an operculum, and is classified within the family Littorinidae, the periwinkles. This is a robust intertidal species with a dark and sometimes banded shell. It is native to the rocky shores of the northeastern, and introduced to the northwestern, Atlantic Ocean.

The shell is broadly ovate, thick, and sharply pointed except when eroded. The shell contains six to seven whorls with some fine threads and wrinkles. The color is variable from grayish to gray-brown, often with dark spiral bands. The base of the columella is white. The shell lacks an umbilicus.

The white outer lip is sometimes checkered with brown patches. The inside of the shell has a chocolate-brown color.

The width of the shell ranges from 10 to 12 mm at maturity, with an average length of 16–38 mm. Shell height can reach up to 30 mm, 43 mm or 52 mm (Figure 1).

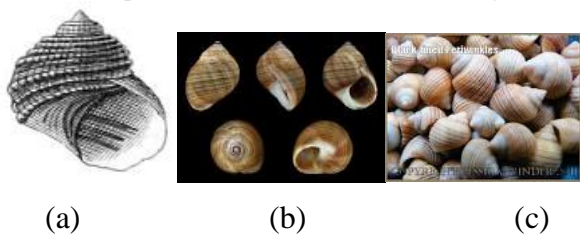


Figure 1. Periwinkle shell

Brake pads were generally made from asbestos fibers. Despite its good properties, asbestos is being withdrawn from all its applications, because of its carcinogenic effect on human, so where there is a possibility of alternative material for making non-carcinogenic brake pad.

Aim

The overall aim of the study is to develop and produce a brake pads made from periwinkle shell composite.

Specific Objectives:

1. To characterize periwinkle shell material to ascertain their chemical composition and mechanical properties.
2. To run series of laboratory tests and analysis which includes ash, porosity, bulk density, compressive test, wear rate and microstructural analysis
3. To conduct performance evaluation.

Materials and Methods

Materials

The materials and equipment used during the course of this work are: Epoxin Resin, periwinkle shell, steel slag dust, carbon black, Engine oil (SEA 20/50), water, hydraulic press, brake pad mould, base metals, vernier caliper, grinder, digital weighing balance, tensiometer, hardness tester, spec-electron, hammer crusher [TYPE: 000T, PUISSANE: 1,5KV, NO: 13634], ball milling machine (Model 87002 Limoges-France, A50.....43), and a set of sieves [Fritsch GmbH, D-55743 Idar-oberstein, Germany] of 100µm, 200µm, 350µm, digital weighing machine (figure 2 and table 1).



Figure 2. Photo of the ingredients

Table1: Elemental Composition analysis of periwinkle shell particles.

S/No	Element	Level Detected (Periwinkle Shell)	Unit
1	SO ₃	0.30	Wt%
2	CaO	96.09	Wt%
3	Fe ₂ O ₃	0.79	Wt%
4	K ₂ O	0.52	Wt%
5	MgO	1.54	Wt%
6	Na ₂ O	0.10	Wt%
7	SiO ₂	0.09	Wt%
8	MnO	0.06	Wt%
9	Cr ₂ O ₃	0.003	Wt%

Process Technology

The periwinkle was purchased in a market in IkotEkpene, Akwa Ibom State, South of Nigeria. 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 cone crusher and was reduced to between 4 and 3 mm. This was further charged into ball machine that now reduces the size of periwinkle shell to between 2 and 1 mm.

The product of roll crusher was transferred into ball milling machine and was left in the mill for two (8) hours; after which the product was transferred into a set of sieves of +350µm, +200µm and +100µm, and was sieved for 30 min using a sieve shaker machine for 30 min. While the oversize at +350µm was returned or recycled for regrinding until it passes through the sieves.

Production of brake pad consists of a series of unit operations including mixing, cold and hot pressing, cooling, post-curing and finishing (Gurunath and Bijwe, 2007). The constituent ingredients, Periwinkle shell, steel dust, graphite, silicon carbide, and resin. Different composition and sieve grades (i.e. 350mm, 200µm and 100µm) of periwinkle shell, steel dust, graphite, silicon carbide powder and resin were added together in the ratio shown in table.2.

The combination were properly dry mixed in a mixer

for 20 minutes (Model 89.2 Ridsdale& Co ltd, Middlesbrough.Eng.) until a homogenous component was formed and transferred into a mould for cold press with a Hydraulic press (Model Pi00eh-Type, 100T-Capacity, 38280-Serial No, at Federal Institute of Industrial Research, Oshodi) for 80 KN/cm² and then conveyed into electric oven (Model Memmert, Western Germany) at temperature of 150°C at 100KN/cm² pressure for 2 minutes. After removing from hot mould, the brake pad was cured in an oven at a temperature of 120°C for 8 hours [Kim, S.J. Kim, K. S. and Jang, H. (2003), Sasaki, Y, Yanagi, M., Todani, Y. and Mita, T. (2000)].

The periwinkle shell sieve sizes of 100, 200, and 350µm, were mixed with the phenolic (13%) in the proportions as stated in Table 1. 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 122 MPa (approximately 12 tons) for 5minute. The samples were post cured at 120°C for 2h.

Table 2. Formulation table for the brake pad

S/N	Ingredients	A	B	C	D	E
1	Periwinkle	40	45	50	55	60
2	Epoxy Resin	13	13	13	13	13
3	Steel slag dust	37	32	27	22	17
4	Carbon black	10	10	10	10	10

Process technology flow chart

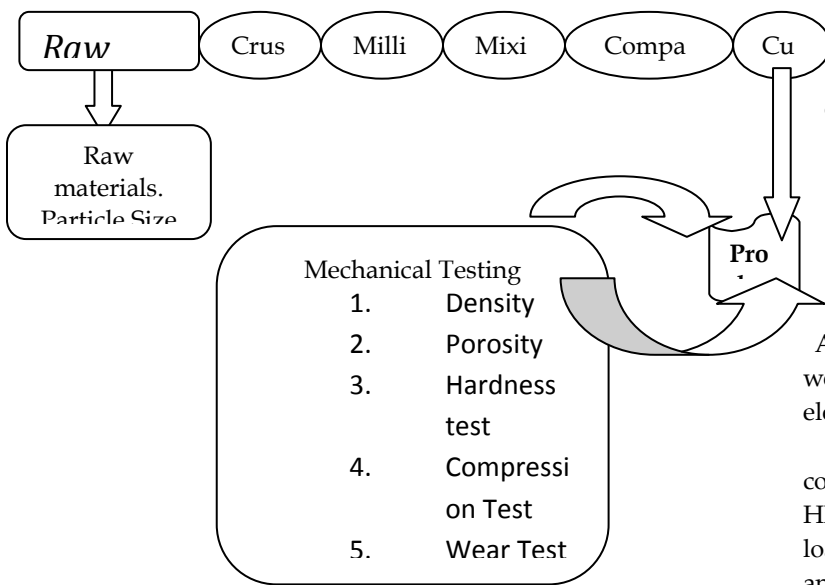


Figure 3. Process technology flow chart of the periwinkles brake pads described above



Figure 4. Photo of the produced samples (products)

**Samples Characterization
Brinell Hardness Test**

The microstructural analyses of the samples were carried out by grinding the samples using 300, 400, and 600 grit papers respectively. Dry polishing was then carried out on these samples and the internal structures were viewed under the computerized Metallurgical microscope [1].

The resistance of the composites to indentation was carried out through the Brinell hardness testing equipment to BS240, using a Tensometer (M500-25KN, Gunt Hamburg Hardness Tester and WP300) pressing hardened steel ball with diameter *D* into a test specimen. Based on ASTM specification, a 10 mm diameter steel ball was used, and the load applied *P* was kept stable at 3000 kg/f. The diameter of the indentation *d* was measured along two perpendicular directions, using an optical micrometer screw gauge. The mean value was taken and incorporated into Eqn. 1 to obtain the Brinell Hardness Number (BHN).

$$BHN = 2P \div \pi D (D - \sqrt{D^2 - d^2}) \quad \text{eqn(1)}$$

Where *P* is the load applied, *D* is the diameter of hardened steel ball into a test specimen and *d* is the diameter of indentation.

Wear Rate Test

The wear rate for the samples were measured using pin on disc machine by sliding it over a cast iron surface at a load of 10N and 20N, sliding speed of 125rev/min and 250rev/min, and sliding distance of 2000m and 4000m. All tests were conducted at room temperature. The initial weight of the samples was measured using a single pan electronic weighing machine with an accuracy of 0.01g.

During the test, the pin was pressed against the counterpart rotating against a cast iron disc (hardness 65 HRC) of counter surface roughness of 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 differences in weight measured before and after

tests give the wear of the samples.

The formula used to convert the weight loss into wear rate is below

$$\text{Wear rate} = \frac{S}{W} \dots \text{eqn(2)}$$

Where ΔW is the weight difference of the sample before and after the test in mg, S is total sliding distance in m.

Compressive Strength Test

The compressive strength test was done using the Tensometric Machine. The samples of diameter 29.40mm was subjected to compressive force, loaded continuously until failure occurred. The load at which failure occurred was then recorded.

Flame Resistance Test

Weigh about 1.20g ± 0.1g of the samples in a cooled crucible previously oven dried by heating in a furnace at 550°C for 1 hour. Then the samples were charred by heating in a hot plate thereafter the charred samples were taken into the furnace and heat at 550°C for 1 hour. Then cool in a dessicator and weigh. This processing of heating, cooling and reweigh were repeating until a constant weight is obtained.

Calculation:

$$\% \text{ ash} = \frac{W_2 - W_0}{W_1 - W_0} * 100 \dots \text{eqn (3)}$$

Where W₀=weight of empty crucible

W₁=weight of crucible + sample

W₂=weight of crucible and residue i.e. after cooling.

Porosity

A samples of diameter 29.40mm with a different height thickness of as thick as possible was used. The specimens were weight to the nearest in mg, and then soak in oil and water container at 90-100oC for 8hrs. The samples were leave for 24hrs and then taken out from the oil container. Finally, the test samples were weight to the nearest mg. this formula was noted in Eqn 4. (Malaysian standard).

$$\text{Porosity (q)} = \frac{M_2 - M_1}{D} \div V \times 100 \dots \text{eqn(4)}$$

D is the density of test oil and water M₂ is the mass of test piece after absorbing oil and water (g), M₁ is the mass of test piece (g) and V is the volume of test piece (cm³).

Density Test

The true density of the samples was determined by weighing the samples mass on a digital weighing machine and divided by measuring their volume by liquid displacement method. The formula is show in Eqn. 5 below.

$$\text{Density (q)} = \frac{M}{V} \times 10 \dots \text{eqn(5)}$$

Where M is the mass of test piece (g) and V is the measuring

volume of test piece (cm³) by liquid displacement method.

Table 3. The products are in compliance with National and International Standards:

S/N	Test procedure
1	Specific Gravity
2	Rockwell Hardness Test
3	Compressive Strain Test Method
4	Internal Shear Strength of Lining Material
5	Shear Test Procedure
6	Resistance to Water, Saline Solution, Oil & Brake Fluid
7	Assessment of Friction Materials and Wear/ Microstructure
8	Determination of Thermal Conductivity
9	Porosity Measurement
10	Swell, Growth and Dimensional Stability
11	Dynamometer Noise Matrix Test
12	Dynamometer Brake Effectiveness Test
13	Vehicle Braking Test
14	Replacement Brake Lining Assemblies
15	Field Test on LRT Test Track
16	Ash test

Results and Discussion

Microstructure

Microstructural 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 5).



100µm A 100µmB 100µmC 100µmD 100µmE



200µm A 200µmB 200µmC 200µmD 200µmE



350µm A 350µmB 350µmC 350µmD 350µmE

Fig. 5 Microstructural studies

During the production of the formulation of the brake pad it was observed that proper bonding was achieved when decreasing the sieve size from 350µm to 100µm.

HARDNESS TEST RESULT

The hardness values of the samples are shown in **Fig. 6** below.

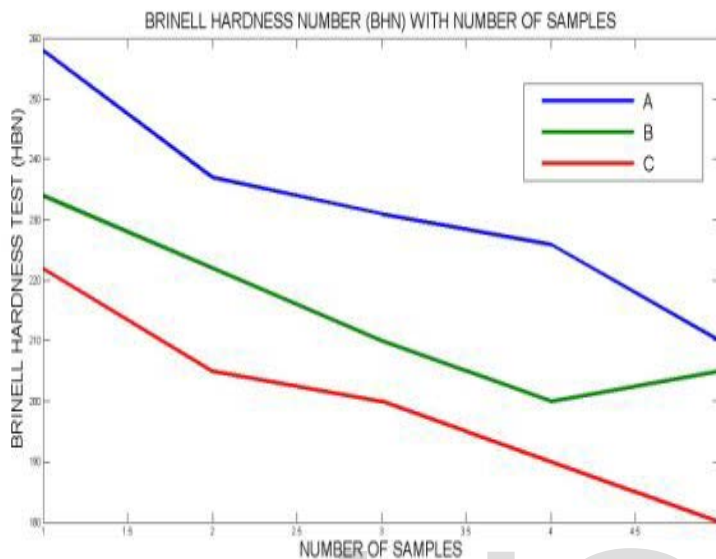


Fig. 6 Show the variation of Brinell Hardness of the developed brake pad samples

From the results it is clear that the hardness increased with decreasing the particles size of periwinkle shell. The sample with 100µm sieve grade has the highest hardness values of 258 HBN. A decrease in hardness values was observed in the samples with higher sieve grades (200 µm and 350 µm).

The high hardness values for the 100µm sieve grade 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 100µm size [2]. This is probably due to the presence of Fe₂O₃, CaO and SiO₂ of the chemical made up of periwinkle shell particles [11].

Wear Rate

The wear rate values 1kg at 125rev/min of the samples are shown in **Fig 7**. below

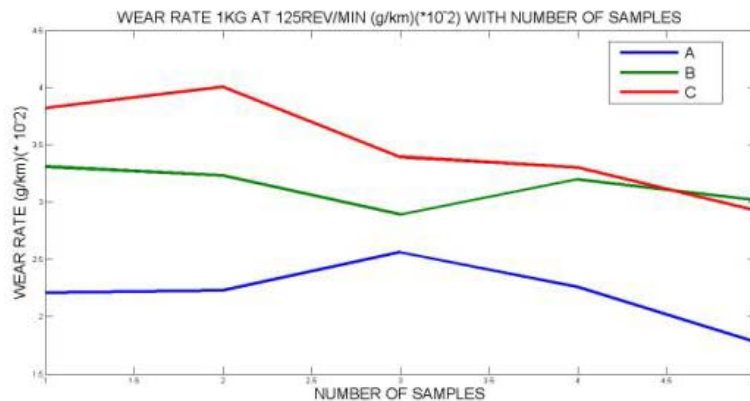


Fig. 7 Shows the variation of wear rate 1kg at 125rev/min (g/km) (10²) of the developed brake pad samples

Fig. 8 Below, shows the wear rate values 1kg at 250rev/min of the samples.

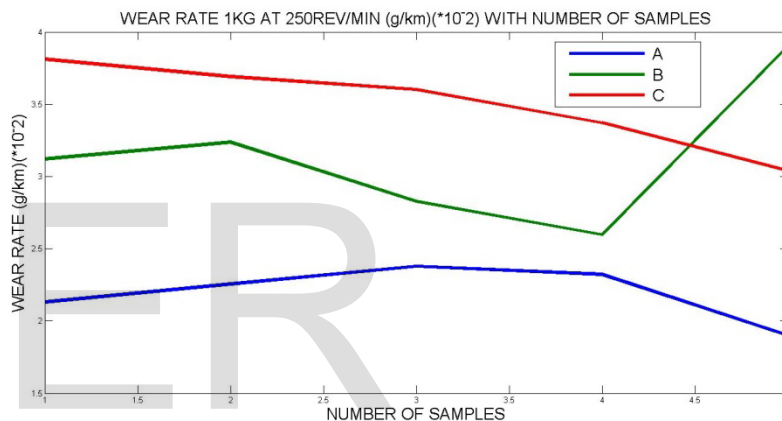


Fig 8. Show the variation of wear rate 1kg at 250rev/min (g/km) (10²) of the developed brake pad samples.

Fig 9. Below, shows the wear rate values 2kg at 125rev/min of the samples.

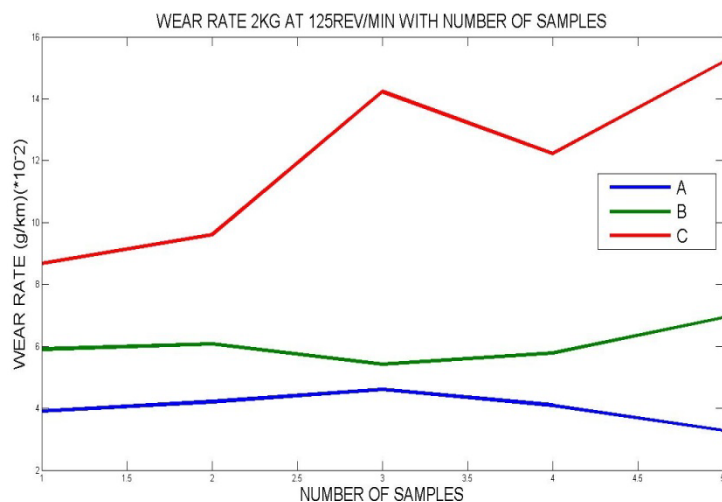


Fig. 9 Shows the variation of wear rate 2kg at 125rev/min (g/km) (10*2) of the developed brake pad samples.

Fig 10. Below, shows the wear rate values 2kg at 250rev/min of the samples.

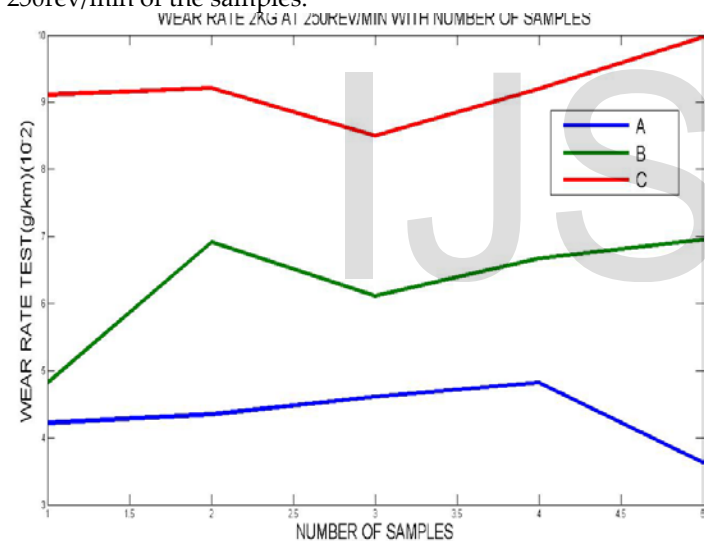


Fig. 10. Shows the variation of wear rate 2kg at 250rev/min (g/km) (10*2) of the developed brake pad samples

It can be seen that as the load increases, the wear of developed brake pad also increased (see Fig. 7, 8, 9, and 10). The wear of the 350µm particle size is more than that of the other samples. However, the wear rate decreases with decreasing the periwinkle shell particle content. From Fig. 7, 8, 9, and 10, 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.

Compressive Strength Test

Fig. 11 below, is showing the compressive properties of the samples.

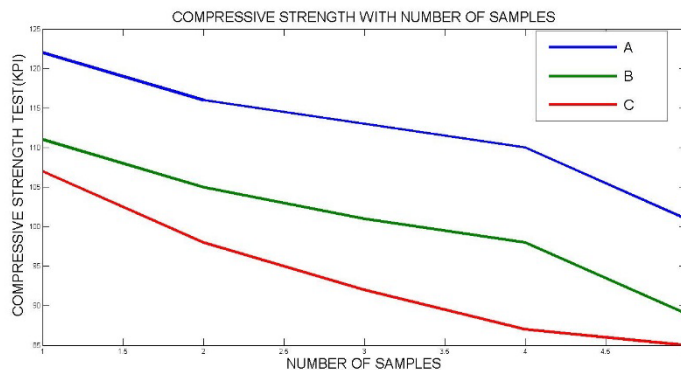


Fig. 11 Show the variation of compressive strength of the developed brake pad samples

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 [12].

Brake pad formulation with 100µm periwinkle shell particles showed the highest compression strength compared to other sieve size used. The less pores and the more compact mixture in 100µm 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.

Ash Content Test

Fig. 12 below has shown the flame resistance of the produced samples.

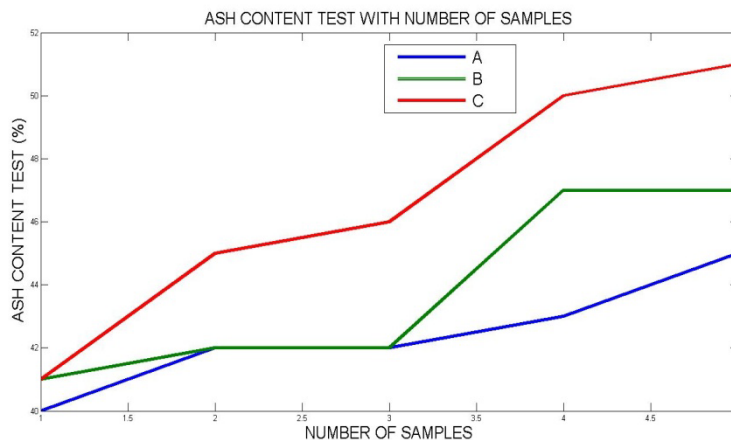


Figure 12 Shows the result of the ash content samples with sieve size.

It can be seen from the graph as the properties increased so also the sieve grade increases which can eventually be attributed to the increases pores as sieve size

increases. It can also be observed from the result that samples with 100µm gave the best properties as a result of a very good dispersion of decreased in size particles.

Porosity Test Result

Fig. 13 below has shown the porosity of the produced samples.

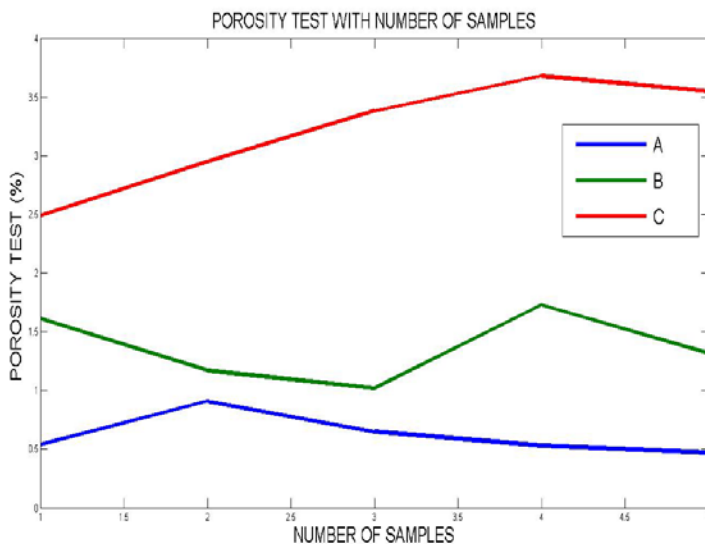


Figure 13 shows the result of the porosity with sieve size.

Those properties increased as the sieve grade increases which can eventually be attributed to the increases pores as sieve size increases. These results are in par with the earlier observation of [1-5]. It can be seen from the result that sample with 100 µm gave the best properties as a result of a very good dispersion of periwinkle particles as shown by the white region and dark region resin (see Fig. 5) which led to a better interfacial bonding of the resin and the periwinkle particles as seen in subsequent samples.

Density Test Result

Fig. 14 below shows the results of density of the samples

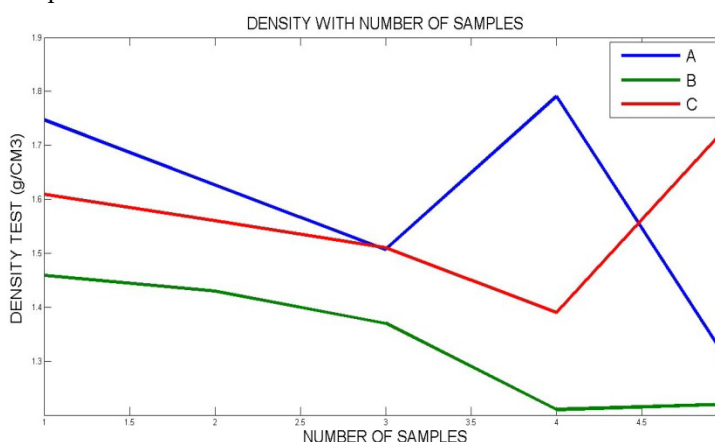


Fig. 14 shows the result of the density with sieve size

The density of the samples increased as the sieve size decreased from 350 to 100µm. This can be attributed to the increases in bonding achieved i.e. increased packing of the particles. The 100µm 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 [12]. 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 [1].

CONCLUSION

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 350 µm to 100µm
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 350 to 100µm, while the oil soak, water soak and wear rate decreased with decreasing the particle size of periwinkle shell.
4. The sample containing 100µm of periwinkle shell particles gave the best properties in all.
5. The results obtained at 100µm of periwinkle shell particles compared favorably with that of commercial brake pad.
6. The results of this research indicate that periwinkle shell particles can be effectively used as a replacement for asbestos in the production of brake pads.

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