Development and characterization of green automotive brake pads from waste shells of giant African snail (*Achatina achatina* L.)

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Abstract

In this study, waste shells of African giant snail (*Achatina achatina* L.) were explored as candidates for asbestos-free non-carcinogenic brake pads. The results obtained showed that the density, Brinell hardness, and compressive strength of the snail shell (SS) brake pads were superior to the commercial sample used for comparison. These properties were found to decrease with increase in particle size, following a negative index power law model after the order of the Hall-Petch equation. However, the liquid absorption characteristics increased with increase in particle size and its model followed a positive index power law due to the pores in the matrix. On the other hand, the thermal conductivity showed no significant change with variation in particle size. The SS-based brake pad exhibited better frictional grip at the rubbing interfaces compared to the commercial brake pad sample. From the frictional results obtained, the commercial brake pad can be rated as Edge-Code-D whereas the frictional ratings for the SS-based brake pad with different particle sizes are Edge-Code-E (500 μm and 250 μm), Edge-Code-F (375 μm), Edge-Code-G (125 μm), and Edge-Code-H (90 μm). The wear rates and wear areas of the developed SS-based brake pads were inferior to the commercial sample but can be improved by impregnating the matrix with more iron fillings to enhance the poor thermal conductivity and hence wear characteristics.

Keywords Green brake pads · Giant African snail shells · Hall-Petch equation · Grain size · Power law model

1 Introduction

The phasing out of asbestos-based materials for engineering applications due to their carcinogenic impact on human and animal health has prompted researches for “greener” alternatives. In tribological parlance, engineering materials that have negligible negative impacts on human and animal health, as well as the environment, are considered to be “green” materials, and hence the need to explore biodegradable materials. Locally available biodegradable materials, such as rice husk and rice straw [1], palm kernel shells [2], coconut shells [3], periwinkle shells [4, 5], and sawdust [6], have been explored with varying performance levels.

Mutuk and Gurbuz [7] investigated pure titanium samples of particle sizes ≤ 30 μm, ≤ 43 μm, and ≤ 150 μm sintered at 1100°C for 120 min with a view to ascertain the role of particle size on the density, hardness, wear resistance, and microstructural properties. In the study, the sample with lowest particle size ≤ 30 μm showed the best mechanical properties. Their wear rate and SEM results indicated that the superior mechanical properties was attributable to good bonding and strong neck formation between the particles with smallest size. These results were corroborated by Ossia et al. [3] showing that particulate grain size affected the physico-mechanical properties of organic brake pads.

In the search for green biodegradable alternatives to the carcinogenic asbestos-based commercial brake pads, waste shells of African giant snail (*Achatina achatina* L.) are yet to be explored. Besides the opportunity to mop up this waste organic material, its conversion to biodegradable brake pad composites will offer good economic and environmental competitiveness. It is this potential that the present study seeks to explore.

2 Materials and Methods

2.1 Materials

The base material for the green brake pad is waste shells of the African giant snail obtained from refuse dumps at
local market in Port Harcourt, Nigeria. Typical waste shells are shown in Fig. 1.

The chemical reagents applied in formulating the SS-based brake pads matrix are the following: (a) milky phenol formaldehyde (99% purity) (Dachy polymer, Taiwan) used as resin; (b) whitish calcium carbonate, CaCO₃ (99.5% purity) (Skyline Chemical, USA) used as filler; (c) colorless methyl ethyl ketone peroxide (MEKP) (99% purity) (Akzonobel, China) used as accelerator; (d) purple cobalt naphthenate (99% purity) (AkzoNobel, China) used as catalyst; (e) carbon black (99.9% purity) (Loba Chemie, India) used as friction modifier; and (f) iron fillings used to boost thermal conductivity.

Others include the following: (a) distilled water, (b) engine oil (SAE 40 Oil), brake pad mold fabricated from carbon steel plate, Vernier caliper, weighing balance, hardness tester, crushing machine, sieves of different sizes, milling machine, and electric oven.

2.2 Methods

2.2.1 Development process

The procedure and processes used in the development of the sample brake pads are similar to that used by Ossia et al [3] in the production of brake pad samples from waste coconut (Cocos nucifera L.) shells as in Fig. 2.

(a) Gathering/washing/cleaning: The waste snail shells were gathered from Choba Market, Port Harcourt, Nigeria, to get the required quantity. The snail shells was thoroughly washed and cleaned to remove dirt and bad smell.

(b) Drying/crushing/grinding: The snail shells were dried in sunlight for 3 days to reduce their moisture content; then, the snails were crushed into smaller pieces and grinded in the laboratory using a grinding machine.

Fig. 1 Waste shells of African Giant Snail in a gathered, b crushed, and c grinded conditions
Sieving: The grinded snail shells were separated into different grain sizes (90 μm, 125 μm, 250 μm, 300 μm, and 500 μm) and separated into different bags with their grain size labels. This process involved arranging the sieves in descending order, applying a quantity of the grinded snail shell in the largest sieve size at the top. The top sieve is covered with the sieve pan cover and shaking vigorously for 10 min to separate the grinded snail shells at the top to different particle sizes; the quantity left in every sieve size at the end of 10 min is put in a pan with its label.

Molding/mixing: The mold was designed with SolidWorks software and fabricated at the University Engineering Workshop. The process involves cutting into shape and welding. The formulation from Table 1 was used; a clean bowl was used in mixing the formulation which was stirred thoroughly to have a homogeneous mixture. The binder was added last to avoid the mixture getting hard before leaving the bowl. The mixture was poured into the mold and rammed so the mixture fully occupies the mold.

Setting/curing: The laboratory brake pad was cured in an electric oven (model: GE30) at a temperature of 120 °C for 2 h.

Extraction: Extraction was done after leaving the newly made brake pad for 1 day to cool and become very hard. The grease applied before the mixture was poured into the mold helps in separating the sample from the mold.

Machining (milling): The extracted brake pad sample is then machined to shape using a carrot stone and a cutting disk with a spindle speed of 288 rpm. The SS-based brake pad samples thus developed were then shaped to size using the milling machine (model: HURE SA-PU771, France).

2.2.2 Evaluation tests

The physico-mechanical tests involving oil (SAE 40) absorption, water absorption, density, Brinell hardness, compressive strength, and thermal conductivity properties were performed on the SS-based brake pad samples following procedures adopted from Ossia et al [3]. Friction and wear tests were performed using Anton Paar GmbH TRB3 Tribometer (version 6.1.19) with ø6mm stainless steel ball-on-brake pad disc samples in dry sliding contact. All the tribological tests were performed at ambient temperature and humidity conditions of 29 °C and 55%, respectively. The brake pad samples were turned into a rotating disc sliding against a stationary ball loaded with 8N at 10 cm/s sliding speed in accordance with ASTM procedure [8]. The friction histories of the interface were recorded, as well as the wear scar area and wear rates.

3 Results and discussion

3.1 Absorption test

From Fig. 3, it was shown that oil absorption of the SS-based brake pad sample increased with increasing grain size. This can be attributed to better bonding between the smaller grain sizes and the binder. When compared with the commercial brake pad, all the developed samples did better. The sample brake pad will do better than the commercial brake pad when there is hydraulic oil leakage.

Also, the water absorption of the sample brake pad increased with increasing grain size. This can be attributed to

| s/N | Materials                                     | Composition (wt %) |
|-----|-----------------------------------------------|--------------------|
| 1   | Snail shell                                   | 53.9               |
| 2   | Phenol formaldehyde (resin)                   | 25                 |
| 3   | Calcium carbonate (CaCO₃) (filler)            | 11.7               |
| 4   | Carbon black (friction modifier)              | 0.5                |
| 5   | Methyl ethyl ketone (accelerator)             | 2.8                |
| 6   | Cobalt nephthenate (catalyst)                 | 2.1                |
| 7   | Iron fillings (abrasive)                      | 5.0                |
better bonding between the smaller grain sizes and the binder. When compared with the commercial brake pad, the 90μm and 125μm sample did better than the commercial brake pad (with 1.2% absorption in water and 6.1% absorption in SAE40 oil). The sample brake pad will do better than the commercial brake pad if used in a wet environment, for instance, when an automobile goes through a flooded road.

The absorption variation with respect to particle grain size corroborates conclusions of previous studies [2–4, 9, 10]. The present results show that the absorption property of the control (commercial) brake pad in SAE40 oil was poor compared to those of the SS-based brake pad.

3.2 Density

Figure 4 shows a variation of the density of the SS-based brake pad with particle grain size. The decrease in density can be attributed to the increase in pore size derived from increased aggregate particle size. The 90μm brake pad sample has the highest density (1.74g/cm³) which is due to closer packing of snail shell aggregate creating more homogeneity in the entire phase of the composite body. Similar decrease in brake pad density with increase in particle grain size had been reported by Yawas et al. [6] who used periwinkle shells in their study.

From Fig. 4, the densities of all SS-based brake pad samples were lower than the control brake pad density (2.18g/cm³). This makes the SS-based brake pad lighter and brings about a reduction in the mass of the automotive braking assembly.

3.3 Hardness (Brinell)

Figure 5 shows that the hardness of sample brake pad varies with increase in grain size. The 90μm sample has the highest hardness value of 49 BHN. A sharp drop in hardness was observed in the samples with higher grain sizes (125μm, 250μm, 375μm, and 500μm). The high hardness for the 90μm particle size sample is attributable to the increase in particle surface area which resulted to increased bonding with the polyester resin. This corroborates the results of Yawas et al. [4] who observed a similar trend with periwinkle shells. It can be observed from Fig. 4 that the hardness of the samples developed from 90μm and 125μm particle grain sizes (49 BHN and 41 BHN, respectively) was greater than the hardness of the control brake pad (39 BHN).

3.4 Compressive strength

Figure 6 shows the variation of compressive strength with grain size of the sample brake pad. From the results obtained, it was observed that the compressive strength increases with decrease in grain size of the specimens. The 90μm sample had the highest compressive strength (3.77MPa), which was greater than that of the control brake pad (2.85MPa). The gradual decrease in compressive strength as the aggregate particle...
grain size increases can be attributed to the decreasing surface area and pore packaging capability of the snail particles in the phenol formaldehyde resin.

Hence, compressive strength increases as aggregate size of the snail shell decreases. During braking, the brakes are exposed to continuous compressive force and the result show that the 90μm sample will do well under such conditions. The observed trend in the relationship between compressive strength and grain size is corroborated by the conclusions of Yawas et al. [4] and Jaya et al. [6].

### 3.5 Thermal conductivity test

Thermal conductivity is an important consideration in the design of a brake pad. Figure 7 shows how the sample brake pad thermal conductivity (2.02Wm/K) superior to the sample brake pad (1.81–1.84Wm/K). The thermal conductivity appears not to be significantly affected by the increase in the grain size. The thermal conductivity of the sample brake pad can be improved by introducing particles of strong (hard) metallic conductors in its formulation.

#### 3.6 Modeling green brake pads’ mechanical property variation with SS particle size

To model the influence of snail shell particle size on the mechanical properties of the developed green brake pad, a modified form of the classic Hall-Petch equation was adopted. Morris [11] proposed an inverse proportionality relationship with respect to the square root of the grain size diameter (d) and material constants (K_p and σ_o) to explain the influence of grain size on the mechanical properties (σ_y) of metals. Morris’ model is a form of the classic Hall-Petch equation and is shown in Eq. (1).

\[
σ_y = σ_o + \frac{k_y}{d^{1/2}}
\]  

(1)

Morris [12] reported that in nanometals, the increase of mechanical properties with respect to decrease in grain size (d) continues to a peak at about 20nm grain size (d) beyond which a fall in properties prevails. Hence, two regimes of property variation were reported, namely, regime I for 0 ≤ d ≤ 20nm (inverse Hall-Petch effect regime where the property decreases with decreasing grain size) and regime II for d ≥ 20nm (Hall-Petch effect regime where the property decreases with increasing grain size).

Other exponents apart from \(x = \frac{1}{2}\) have been reported for the Hall-Petch model. Different x-exponents in the range 0 < x ≤ 1 have been reported based on experiments. Dunstan and Bushby [13] obtained \(x = 1\) for FCC and BCC metals and ceramics in the compression testing of micropillars by using grain size as bulk micropillar diameters (d) which was corroborated by Li et al. [14]. Agraie-Khafri et al. [15] reported \(x = 0.66\) for hot rolled AISI 300 stainless steel in uniaxial tensile tests at 0.2% strain.

In this study, the mechanical properties, \(y_p\), of the sample brake pad were modeled by power law relationship after the order of modified Hall-Petch Eq. (2).

\[
y_p = \frac{K_p}{X^a}
\]  

(2)

where:

- \(Y\) particle size value (μm) equivalent to Hall-Petch grain size (d);
- \(K_p\) particle size constant;
- \(a\) particle size index equivalent to Hall-Petch grain exponent (1/2).
The mechanical property tests results obtained in Figs. 2, 3, 4, and 5 were modeled by power law for best fit after 100 iterations using SigmaPlot 8 software. The models obtained for the mechanical properties, their $R^2$ value, and error estimate are summarized in Table 2.

The nonlinear regression of thermal conductivity with particle grain size is rather weak because of the lower coefficient of determination $R^2 = 0.7668$.

It is instructive to observe that all mechanical property models in Table 2 are similar, following a power law with a positive or negative particle size index, indicating similar generating mechanism. It showed positive particle size indices for oil absorption, water absorption, and thermal conductivity (flow processes) and negative particle size indices for density, hardness, and compressive strength (non-flow processes). These properties are attributable to the roles of pores in the composite matrix. The higher the matrix particle size, the higher the pore size and distribution that enhance absorption as the pores are being filled in the soaking (absorption) medium. But the corollary is true for other mechanical properties, since the higher pores associated with higher matrix particle sizes become crack nucleation sites in the course of loading before failure in hardness or compression test. Hence, higher particle sizes become associated with lower hardness and compressive strength. Obviously, for a fixed volume of brake pad matrix, the sample with higher particle size, hence higher pore sizes and distribution, will exhibit less mass, and less density which corroborates the model result. This explains the results earlier obtained by Dagwa et al. [2], Yawas et al. [4], Zykova et al. [9], Ameh et al. [10], and Jaya et al. [6].

### 3.7 Tribological performance characterization

#### 3.7.1 Friction characterization of the ball-on-disc sliding contact

(a) Friction history

The friction history, which is a measure of kinetic friction trace of all the developed brake pad samples, showed 2 friction regimes, namely, the transient and steady state regimes. The transient regime is characterized by friction rise from zero (or minimum value) to the onset of steady state value. From Figs. 8, 9, 10, 11, 12, and 13, when the steady state value is attained, the kinetic friction remains at this value until the end of the sliding contact. So, the friction signatures in Figs. 8, 9, 10, 11, 12, and 13 can be observed to follow through a minimum, average, and maximum friction values for every

![Fig. 8](image_url) Friction history of SS-based brake pad sample with 90μm particle size

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### Table 2 Models of mechanical properties of SS brake pad samples as functions of particle size

| s/N | Mechanical property          | Mathematical model                      | Coefficient of determination $R^2$ | Standard error of estimation $\varepsilon$ | $p$-value |
|-----|-------------------------------|-----------------------------------------|------------------------------------|------------------------------------------|-----------|
| 1   | Oil absorption, $A_o$        | $A_o = 0.03077X^{0.6284}$               | 0.9939                             | 0.0383                                   | 0.0002    |
| 2   | Water absorption, $A_w$      | $A_w = 0.06516X^{0.5718}$               | 0.9094                             | 0.2248                                   | 0.0119    |
| 3   | Density, $\rho$              | $\rho = \frac{3.596}{X^{0.0005}}$      | 0.9889                             | 0.0217                                   | 0.0005    |
| 4   | Harness, BHN                 | $BHN = \frac{191.6881}{X^{0.00005}}$   | 0.9778                             | 1.4335                                   | 0.0041    |
| 5   | Compressive strength, $\sigma$ | $\sigma = \frac{10.715}{X^{0.008156}}$ | 0.9709                             | 0.1039                                   | 0.0021    |
| 6   | Thermal conductivity, $k$    | $k = 1.7416X^{0.0000156}$              | 0.7668                             | 0.0068                                   | 0.0516    |
friction trace. While the friction mechanism in the transient regime could be explained by the surface roughness (asperity interlock) friction theory [16], that of the steady state regime can be explained by the simple adhesion friction theory due to the filling of the matrix pores close to the interface by initial wear metal transfer.

(b) Average friction coefficient

Figure 14 shows the average friction coefficient for the sliding contact of a steel ball sliding on the brake pad disc in a 1010-s test. The results showed increase in friction coefficient with increase in particle grain size. The friction coefficient of the commercial sample was less than that of the highest grain size sample, that is, the 500μm grain size sample. This result is in contrast with Yawas et al. [4] result based on periwinkle shells, but corroborated that of Amaren et al. [5] who studied the effect of periwinkle particle size on the wear of brake pad using full factorial experimental design. Amaren et al. [5] obtained a negative main effect (−0.025) of periwinkle particle size on friction coefficient models for brake pads. Of all the 4 independent variables (load, speed, temperature, and particle size) used to model friction and wear in the study, particle size was the most significant (p-value 0.02777). This friction coefficient variation trend with respect to particle size was also observed by Sasaki [17] and Aigbodion et al. [18].

From the friction values in Fig. 14 and based on the work of Blau [19] and SAE J866a [20] standards for friction identification of brake linings and brake blocks, the commercial brake pad can be rated as Edge Code-D whereas the 500μm sample is Edge-Coded-E, 375μm
sample is Edge-Coded-F, 250μm sample is Edge-Coded-E, 125μm sample is Edge-Coded-G, and 90μm sample is Edge-Coded-H. Finally, the frictional responses in Fig. 14 suggest that the green brake pads offer better or more effective grip at the rubbing interface relative to the commercial brake pads. This frictional property improved with decreasing particle size.

The average friction coefficient $\mu_{\text{ave}}$ was modeled as Eq. (3).

$$
\mu_{\text{ave}} = \frac{5.01326}{x^{0.4716}}
$$

where coefficient of determination $R^2 = 0.9533$, standard error of estimate = 0.0358, and $p$-value = 0.0043. It is obvious that as the particle size increases, the average friction coefficient decreases, hence corroborating Amaren et al. [5] negative effect.

### 3.7.2 Wear characterization of green brake pads using ball-on-disc sliding contacts

The wear rates and wear areas of the worn brake pads were observed to decrease with the increase in snail-shell granular particle size as in Fig. 15. All SS-based brake pad samples exhibited higher wear rates and wear surface areas compared to the control brake pad. This is attributable to the poor thermal conductivity of the sample brake pads which made them thermally unstable during the friction heating of the rubbing contacts. This poor wear behavior can be compensated by impregnating the composite matrix of the sample brake pads with higher percentage of iron fillings to obtain better frictional grip with reduced wear rates and wear surface.
The wear area was modeled as Eq. (4).

\[ W_{\text{area}} = 55.8769 - 0.01977x^{1.1469} \]  

where coefficient of determination \( R^2 = 0.9828 \), standard error of estimate = 1.6631, and \( p \)-value = 0.0172. Also, the wear rate model was obtained as Eq. (5).

\[ W_{\text{rate}} = 250.717 - 1.859x^{0.6934} \]  

where coefficient of determination \( R^2 = 0.9384 \), standard error of estimate = 14.7525, and \( p \)-value = 0.0616. Comparing Eq. (4) and Eq. (5) with Eq. (1), it can be observed that wear rate and wear area models followed the classical Hall-Petch equation with negative particle constants \( k_y \) and positive particle (grain) size indices since wear particle generating mechanism was by plastic flow due to crack propagation.

Different scholars attempting to relate wear rates with particle size of specimens have obtained variant results in the recent past. Sevim and Eryurek [21] obtained results showing that wear resistance of non-heat treated steel was inversely proportional to the square root of the abrasive particle size by gravimetric measurement. This is at variance with the results of the present study due to the fact that Sevim and Eryurek [21] considered the particle size of the harder (abrasive) material while the present study focused on the particle size of the softer (abraded) material. Arora et al. [22] investigated the influence of particle size and temperature on the wear properties of rutile-reinforced aluminum metal matrix composites. They obtained results for a 49N load on a pin-on-disc tribometer which indicated that finer particles (50–75\( \mu \)m) exhibited wear resistances that are two orders greater than the corresponding results of composites with coarse particle sizes (106–125\( \mu \)m). Santos et al. [23] used dimensional change measurement of AISI 1020 steels with Alumina Al2O3 coatings of different particles sizes (92nm–76.79\( \mu \)m) at 10.2N...
load in the adhesive wear and 130N in abrasive wear mode. Their results showed a logarithmic rise of wear volume with respect to particle size which was more pronounced at nanoscale (<100nm); but for particle size > 10μm, there was little or no particle size effect on wear in both adhesive and abrasive modes.

A typical finished SS-based brake pad with 125μm particle size mounted on the back-plate ready for installation on a brake-disc is shown in Fig. 16.

4 Conclusion

Green automotive brake pads from non-carcinogenic waste shells of African giant snail (Achatina achatina L.) base materials have been developed by compression molding and curing at 120°C for 2 h. These brake pads with different particle grain sizes were subjected to several physico-mechanical and tribological performance tests and the results compared with those of a control commercial asbestos-based brake pad.

Experimental evidence in the present study showed that the application of waste African snail shell as base material for brake pads has the potentials of a good replacement for the carcinogenic asbestos-based brake pads. The mechanical properties of the SS-based brake pads, such as density, Brinell hardness, and compressive strength, decreased with increase in particle size, following a power law model with negative power exponent after the order of the Hall-Petch equation. However, the liquid absorption and thermal conductivity properties (flow processes) exhibited models with positive particle size indices. Whereas the absorption of the SS-based brake pads increased with increase in particle size due to the pores in the matrix which increased with particle size, the thermal conductivity showed lower variation with particle size. The SS-based brake pads exhibited better frictional grip at the rubbing interfaces compared to the commercial brake pad. However, the wear behavior was poor compared to the commercial sample. This poor wear behavior can be compensated by impregnating the composite matrix with higher percentage of iron fillings to improve its thermal conductivity and stability.

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Authors’ contribution The research contained herein describes a green, non-carcinogenic, asbestos-free automotive brake pad derived from biodegradable organic composite of waste snail shells as an alternative to the asbestos-based brake pads. The lead/correspondence author conceptualized, supervised, and implemented the research while the second (contributing) author performed the editing.

Data availability All data generated or analyzed during this study are included in this manuscript and its supplementary files.

Declarations

Ethics approval and consent to participate Ethical approvals are not required for this research work. Consent is hereby given by authors for the Journal Editorial/Review Team to participate in the Manuscript Blind-Review Process prior to publication

Consent for publication Consent is hereby granted by the authors for the Editorial Team and Publishers to proceed with the publication of the research manuscript after acceptance

Competing interests The authors declare no competing interests.

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