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Effect of grain size on the physicomechanical properties

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Abstract ***In this study, locally available waste coconut (Cocos nucifera) shells (CSs) were investigated as possible replacement for asbestos-based brake pads. The CS-based brake pad was tested for its physicomechanical properties and compared with a commercial brake pad used as control sample. The results showed that (a) an improved interfacial bonding between the CS particles and the binder as the grain size decreases; (b) the 90 µm grain size sample had better physicomechanical properties than the control sample in all tests except the thermal conductivity and stability tests; and (c) the hardness, compressive strength, and density of the CS-based brake pad decreased with increasing grain size, whereas the absorption properties increased with increasing grain size. The study showed that further reduction of the grain size below 90 µm and matrix impregnation with metals of good thermal conductivity could provide significant improvements to properties of the CS-based brake pad.***

Keywords *brake pad, coconut shell, grain size, physicomechanical characterization*

1. Introduction

Automotive brake pads are composed of several ingredients classified as reinforcement/base materials, binders, fillers, friction modifiers, and abrasives [1]. They are essential safety components used for bringing moving vehicles to rest by converting kinetic energy to heat energy and disposing the heat to the surroundings. Besides environmental friendliness and human-healthiness compliance, this primary function of brake pads imposes some basic requirements, namely, low weight, low swelling (oil/water absorption rate), reduced porosity, good hardness, compressive strength and rigidity, good thermal conductivity and stability, good resistance to wear, good frictional properties, among others.

Asbestos has been a major reinforcement material used in the production of commercial brake pads. This is because of its wear-resistant abilities, comparatively low cost, and high thermal conductivity and stability. However, due to the carcinogenic nature of asbestos, a proposed ban was placed on its usage by the US Environmental Protection Agency in 1986. The aim of the proposed ban was to dissuade manufacturing companies from further use of asbestos in producing brake pads and other friction components and encourage the use of nonhazardous environmental-friendly materials [1]. Though the ban could not be sustained, it has paved the way for several innovative scientific researches proposing different materials that could be used as substitutes for asbestos. Among the alternative materials proposed are (a) palm kernel shell and fiber [2,3]; (b) palm ash [4]; (c) rice husk [5,6]; (d) banana peels [7,8]; (e) maize husk [9]; (f) periwinkle shell [10]; (g) bagasse [11]; (h) cow bone [12,13]; (i) fly ash [14]; (j) cocoa bean shells [15]; (k) lemon peel powder [16]; and (l) seashell [17]. Similar efforts were made with the use of coconut (*Cocos nucifera*) shells (CSs).

CSs (*C. nucifera*) are cheap renewable agro wastes commonly used as fuels. They are also useful in the production of activated carbon. Unfortunately, these practices involve the release of gaseous substances into the environment, thereby posing serious health challenges [18]. A previous study by Maleque et al. [19] showed that CSs have good mechanical and physical properties, and hence research efforts can be directed toward alternative use of this readily available biodegradable agricultural waste. Liyanage and Pieris [20] explored the possibility of using CSs as filler materials in industries by investigating the physicochemical properties of CS powder. Also, the possibility of using CSs as substitute for asbestos in brake pad manufacture was investigated by Bashar et al. [21], who developed automotive brake pad samples from composites of CS and other ingredients. The CSs were sun-dried, hammered, pounded, ground into

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powder, and sieved into a grain size of 710 μm. The sieved CS powder was homogeneously mixed with cast iron filings, silica, epoxy, catalyst, and accelerator, and then molded into shape at varying compositions of the constituents. The mechanical properties of the developed sample were tested and compared with a commercial Honda brake pad. The results showed that coconut reinforcements can be used as possible substitute for asbestos in brake pad manufacture. In another study, Egeonu et al. [22] employed palm kernel shell powder and CS powder, which were combined at varying mass compositions as reinforcement materials. The study showed that samples with a higher percentage weight of CS to palm kernel shell had better mechanical properties when compared with a commercial brake pad. Also, Daud et al. [23] investigated the use of CS powder as base material for producing brake pads, and the physicomechanical properties of the brake pad samples developed were analyzed. The study concluded that CS is an effective substitute for asbestos in producing brake pads. Abutu et al. [24] developed CS reinforced automotive brake pads using the gray relational analysis and the central composite experimental design approach. The developed samples produced less noise and vibration during application when compared to the control sample. In a more recent study, Juan et al. [25] used a composite of candlenut shell and CS as reinforcement materials for brake pad production. The study showed that the sample with a higher percentage of candlenut shell to CS (25 wt%:15 wt%) possessed better mechanical and tribological properties, with a wear rate of 3.67×10^{-5} g/mm^{2}s. From the foregoing discussion, it is evident that several efforts have been made to study the effect of the weight composition of the base-matrix and reinforcement material on the desirable properties of green automotive brake pads. However, evidence concerning the effect of base-material particulate grain size on the physicomechanical properties of green brake pads seems to be scarce. In this study, the effect of particulate grain size on the physicomechanical properties of CS-based brake pads was investigated with the aim of producing brake pads that have better desirable properties than their commercial equivalent. The study was conducted experimentally and the results of the CS-based brake pad were analyzed and compared with the corresponding results of a commercial brake pad.

Table 1. Chemical composition of CS [26]

	Component	Amount (%)
1	Lignin	30.1
2	Pentosans	28.2
3	Cellulose	27.7
4	Moisture	6
5	Solvent extractives	5.3
6	Uronic anhydrides	2.3
7	Ash	0.5

CS, coconut shell.

2. Materials and Methods

2.1. Materials

The materials used for the sample preparation include (a) waste CSs (Figure 1) obtained from Swali market, Yenegoa, Nigeria, (b) calcium carbonate ($CaCO_3$) fillers, (c) diethylenetriamine hardener, (d) colorless methyl-ethyl ketone peroxide (MEKP) catalyst (99.9% purity), (e) phenol formaldehyde resin, and (f) carbon black friction modifier. The chemical composition of a typical CS is shown in Table 1.

Laboratory equipment used for sample preparation include sieves of various sizes (BS 410 Standard sieves), electric oven (model: GE30), electronic weighing balances (model: ECB600, Germany), mild steel molds, and SAE 40 lubricating

Figure 1. Waste coconut (Cocos nucifera) shell.

Table 2. Material formulation for the CS-based brake pads

S/N	Material	Composition (wt%)
1.	Phenol formaldehyde (binder/resin)	25
2.	$CaCO_3$ (filler)	9.8
3.	CS powder	58.9
4.	MEKP catalyst	2.8
5.	Hardener	3.0
6.	Carbon black (friction modifier)	0.5

CS, coconut shell; MEKP, Methyl-ethyl ketone peroxide.

oil (Oando). The equipment used to test the sample are specified in the respective sections that discussed the various test conducted on the samples.

2.2. Sample preparation

The CS brake pad samples were produced by following standard procedures published in the literature [21,27]. Phenol formaldehyde resin was initially poured into the mold cavity to determine the quantity of mixture that would take up the volume of the mold. The CS was sun-dried for 5 days to completely remove all moisture content, and the shells were first reduced to small fragments by pounding in a mortar. The reduced fragments were grounded into powder using a grinding machine and sieved into different grain sizes of 90 μm, 500 μm, 600 μm, and 850 μm, which were then used to produce different samples based on grain size. For each grain size, specific quantities of the different constituents were measured and mixed as shown in Table 2. Calcium carbonate ($CaCO_3$) filler, carbon black friction modifier, and sieved CS powder were measured and mixed in a vessel to obtain a homogeneous composite. Also, phenol formaldehyde resin, MEKP catalyst, and diethylenetriamine hardener were thoroughly mixed in a separate container before adding and mixing them with the homogeneous composite mixture. The final homogeneous mixture was transferred to the mold cavity for compression molding and allowed to cure for 45–120 min. The samples were further cured by heating in an electric oven at 140°C for 60 min to improve hardness. The post-cured samples were kept in a ventilated environment for 7 days, which is the recommended minimum strengthening duration before any mechanical tests could be carried out on such samples [28,29].

2.3. Brake pad development process

The formulation of the CS-based brake pad used in this study was based on the material composition in Table 2, whereas a flowchart of the actual processes and procedures applied to develop the brake pad is shown in Figure 2.

2.4. Brake pad testing

The brake pad samples were subjected to the following physicomechanical tests as recommended in the literature [27,30–32]: water and oil absorption, density, hardness, compressive strength, and thermal conductivity test.

2.4.1. Oil and water absorption test

The dry weight of each CS-based brake pad sample was measured and recorded as W_0 before immersing in different absorbent media (water and Oando SAE 40 oil) for 24 h. At the expiration of that time, the samples were brought out of the absorbent medium and thoroughly cleaned with ethanol to remove surface-adhered absorbents. The new weight of the sample was then recorded as W_1. The percentage of absorption of the different samples was obtained using the following Eq. (1) [28,32].

$$\text{Absorption}(\%) = \frac{W_1 - W_o}{W_o} * 100 \quad (1)$$

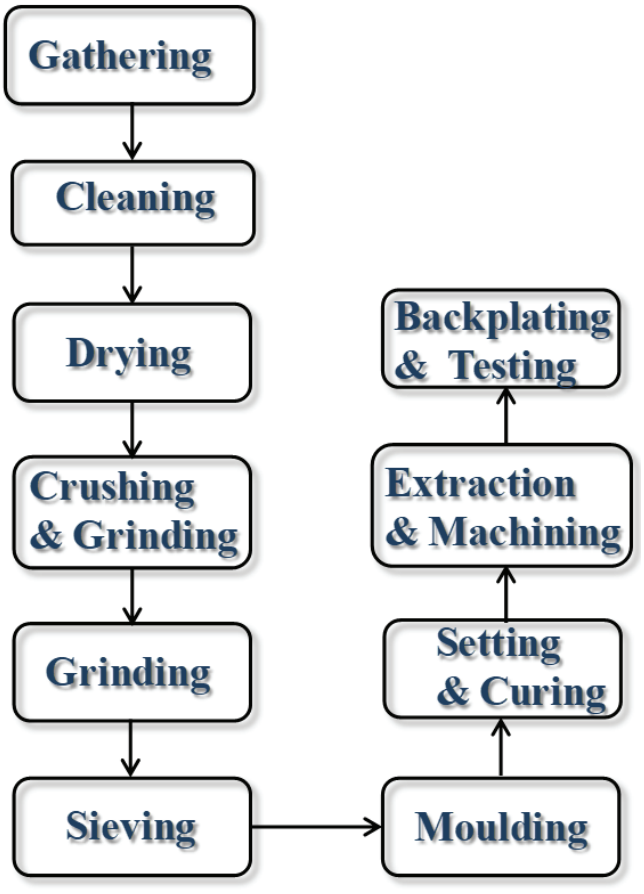

Figure 2. Procedure used to produce CS-based brake pad.

2.4.2. Density test

The density of the brake pad sample was obtained by applying Archimedes' principle of displaced volume [27]. Hence, the density (ρ) of the brake pad sample was simply calculated as:

$$\rho\left[\frac{\mathrm{g}}{\mathrm{cm}^3}\right]=\frac{m}{V}=\frac{m_a}{m_a-m_w} \quad (2)$$

where m_a is mass in air and m_w is mass in water.

2.4.3. Hardness test

Hardness test was conducted on each grain size of brake pad sample using a Brinell hardness testing machine (model: B3000(H), London). A force of 3,000 kgf was applied on the samples using a 10.00 mm steel ball indenter. The recorded hardness number was based on an average reading of three replications of each brake pad formulation, that is, grain size.

2.4.4. Compression test

A Universal Testing Machine (model: UTM-3000, Germany) was used to determine the compressive strength of the samples. A sample was firmly held on the compression plates between the adjustable and bottom cross-heads. When load is applied, the output display unit shows the corresponding strain value, which was taken at regular intervals.

2.4.5. Thermal conductivity test

A simple thermal conductivity test was conducted using the following equipment: 240 W hot plate, cotton wool, mercury-in-glass thermometer, coconut oil, commercial (control) brake pad, and the CS-based brake pad samples. A Vernier caliper was used to measure the thickness of the brake pad samples. After the hot plate was turned on, the flat surface of the brake pad sample was placed on the hot plate until the temperature of the other surface started increasing. Then, the temperatures of both sides of the sample were taken using a mercury-in-glass thermometer. The thermal conductivity (λ) of the brake pad sample was estimated using the following Eq. (3).

$$\lambda=\frac{Q*d}{a*(\Delta t)} \quad (3)$$

where Q is the power of the hot plate (W), d is the mean thickness of the sample brake pad (m), a is the surface area of the brake pad (m2), and Δt is the temperature gradient between the hot and cold surface of brake pad sample (K).

3. Results and Discussion

A commercial brake pad (model: ICER 140403-700) was used as a control sample for comparison with the CS-based brake pad samples having different grain sizes. The results of the comparisons are discussed in this section with the aim of evaluating the effect of grain size on the physicomechanical properties.

3.1. Density

Figure 3 shows a decrease in the density of CS-based samples as the particle grain size increases, with 90 µm particle size having the highest density of 1.702 g/cm^3. This is attributable

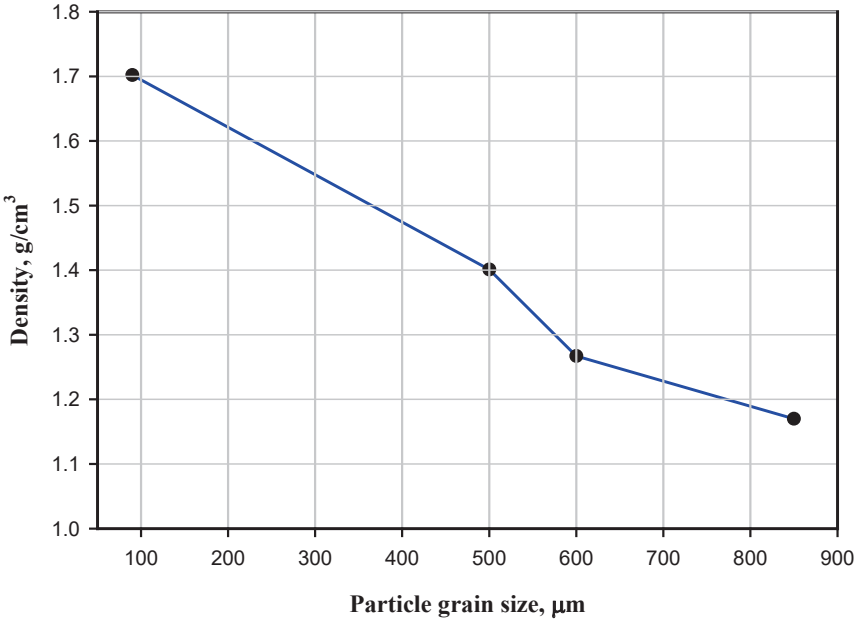

Figure 3. Density variation with particle grain sizes for CS-based brake pad samples.

to the fact that increased grain size leads to reduced interfacial bonding (between the binder and base material particles) and increased porosity. When compared with the commercial (control) sample with density of 3.227 g/cm^3, the CS-based samples showed much lower densities. This implies that the CS-based samples are lighter than the control sample and hence would give an overall weight reduction in the automotive braking assembly.

3.2. Liquid absorption

Figure 4 shows liquid absorption results of the CS-based brake pad samples, indicating an increase in both oil and water absorption as the grain size increases. The increase in porosity with grain size causes the water and oil absorption to increase. This absorption trend is corroborated by the results of Ibhadode and Dagwa [33], although their work was based on palm kernel shell. Their study showed that the increase in absorption (referred to as swelling) was due to (a) bulging of hygroscopic elements and (b) the release of compression stresses imparted to the brake pad during pressing (spring-back phenomenon).

The CS-based samples were found to compare favorably with the control sample, with an absorption value of 1.2% in the water absorption test. This means that the CS-based samples will produce similar braking performance as the control sample in wet rainfall conditions of operation. On the other hand, the CS-based brake pad samples showed oil absorption values (1.2–2.3%) which were much lower compared with the control sample absorption value (5.9%). In the event of oil leakage that comes in contact with the brake pad, the CS-based brake pad will obviously do better than the commercial brake pad since it absorbs less oil.

3.3. Hardness and compressive strength

Figure 5 shows the variation of brake pad hardness with the grain size for the CS-based samples. The brake pad hardness decreased with increased grain size because the CS particles bonded less compactly as the grain size increased. The sample with a grain size of 90 μm had a Brinell hardness value of 42BHN compared with control sample, which had a Brinell hardness value of 39BHN. Based on Holm's wear theory, which states that wear rate decreases with increase in hardness of the softer surface of a mating pair [34], the CS-based sample with a grain size of 90 μm has a potential to exhibit better wear rate properties than the control sample. However, more experimental study is required for better understanding of the wear properties of the CS-based brake pad.

From Figure 6, the compressive strength of the CS-based samples decreased with increase in grain size, a trend similar to the dependence of hardness on grain size. As the grain size increases, the strength of the interfacial bonding decreases and the nucleation sites for mechanical failure multiply. Hence, the samples with larger grain sizes fail at lower compressive loads due to the combined effect of weaker bonding and more crack initiation and propagation sites. The 90 μm grain size sample gave the highest compressive strength of 3.1 MPa, which was observed to be higher than the compressive strength (2.85 MPa) of the control sample.

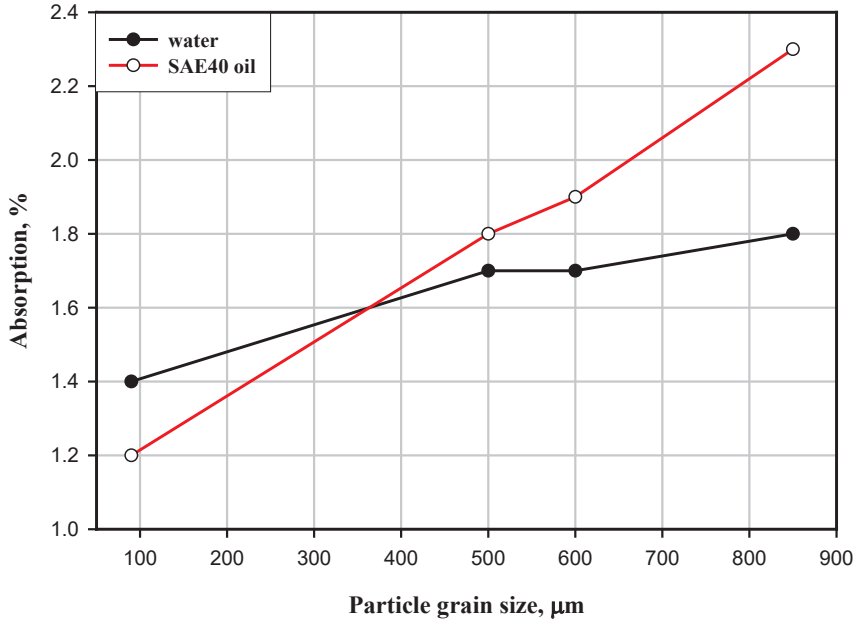

Figure 4. Water and oil absorption of CS-based brake pad samples with different grain sizes.

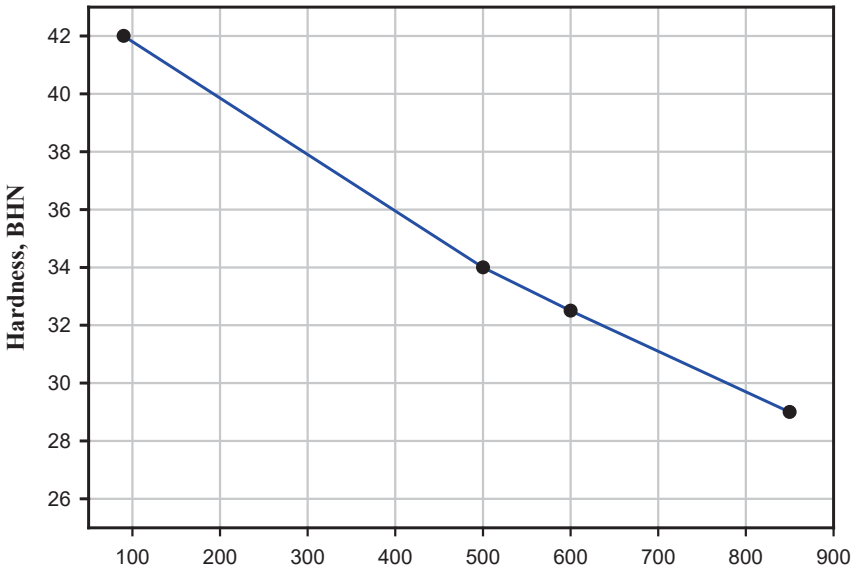

Figure 5. Brinell hardness (BHN) of CS-based brake pad samples with different grain sizes.

3.4. Brake pad thermal conductivity test

As shown in Figure 7, the thermal conductivity of the brake pad samples increased with increase in grain size, (from 1.58 Wm/k for 90 μm to 1.70 Wm/k for 850 μm particle sizes). When compared with the control sample with 2.33 Wm/k thermal conductivity, the CS-based brake pad samples exhibited lower thermal conductivity. This implies that the CS-based brake pad would have a higher heat resistance than the commercial brake pad. Low thermal conductivity increases the tendency of raising the brake pad temperature because increased thermal resistance builds up temperature at the tribological contact surface. This trend was also observed by Ibhadode and Dagwa [33] for palm kernel-based brake pad. A good way to improve the thermal conductivity of the CS-based brake pad is to impregnate the matrix with highly conductive metal particles such as iron filings. Commercial

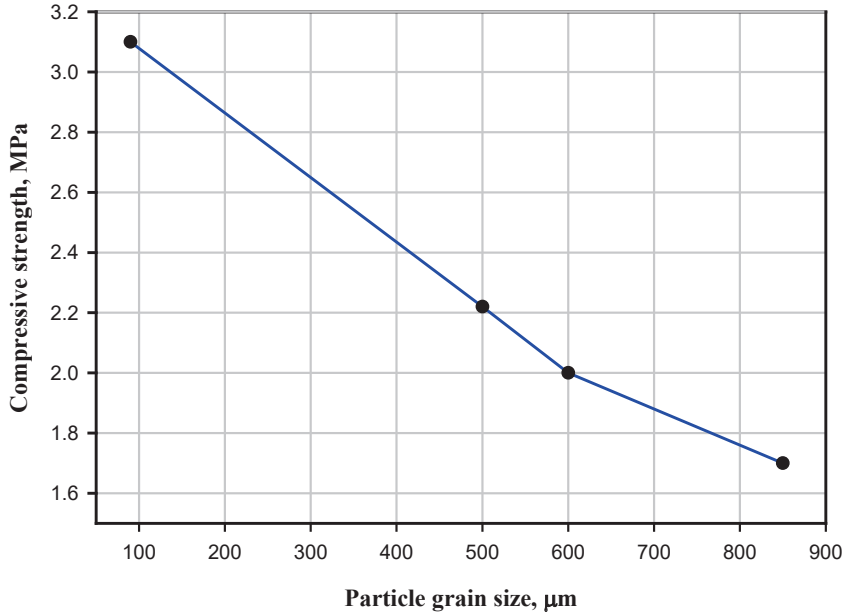

Figure 6. Compressive strength of CS-based brake pad samples with different grain sizes.

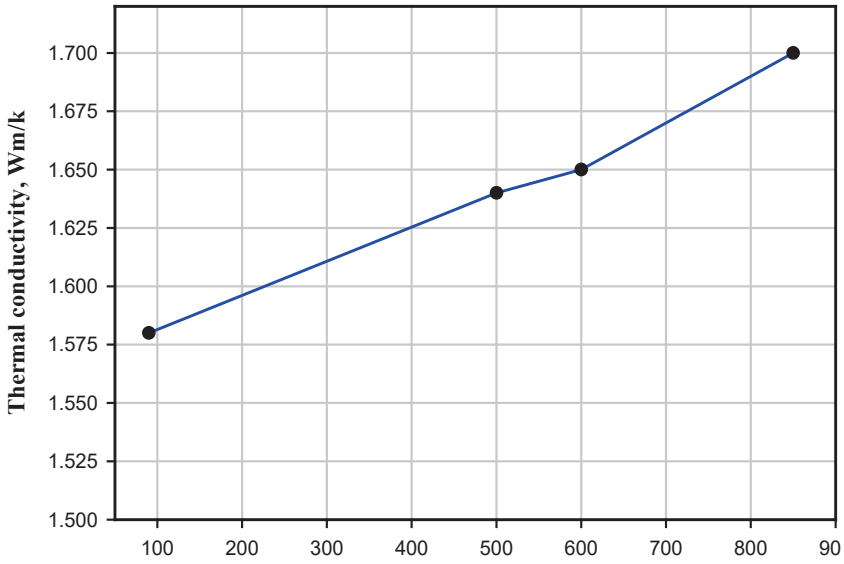

Figure 7. Thermal conductivity of CS-based brake pad samples with different grain sizes.

brake pads, such as the control sample used in this study, already contain iron filings as a means to improve the thermal characteristics of the brake pad.

3.5. Thermal stability characterization

The thermal stability of the CS-based samples were tested and compared with that of the control sample in order to ascertain the extent of charring under intense frictional heating arising from spontaneous emergency brake application. These thermal stability tests were performed by thermogravimetric analyses (TGAs) (ASTM E1131) in an inert Nitrogen gas environment at heat ramp of 20°C/min for 25 min duration. The results of the TGA are shown in Figure 8 for the CS-based brake pad samples and the control sample. It can be observed from Figure 8 that only about 20% (approximately 1.15 g) of the 90 μm grain sized CS-based sample was left at

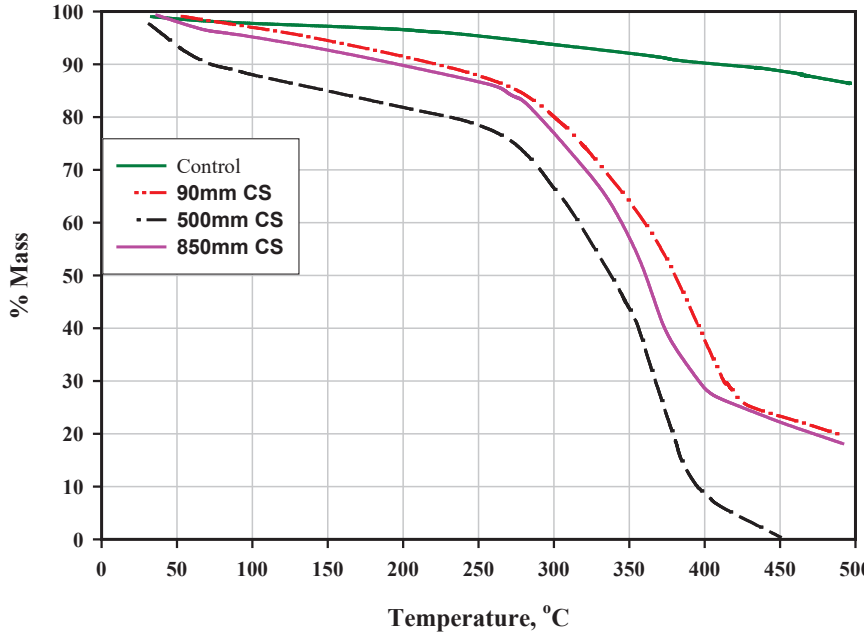

Figure 8. TGA results for the control and CS-brake pad samples of different grain sizes.

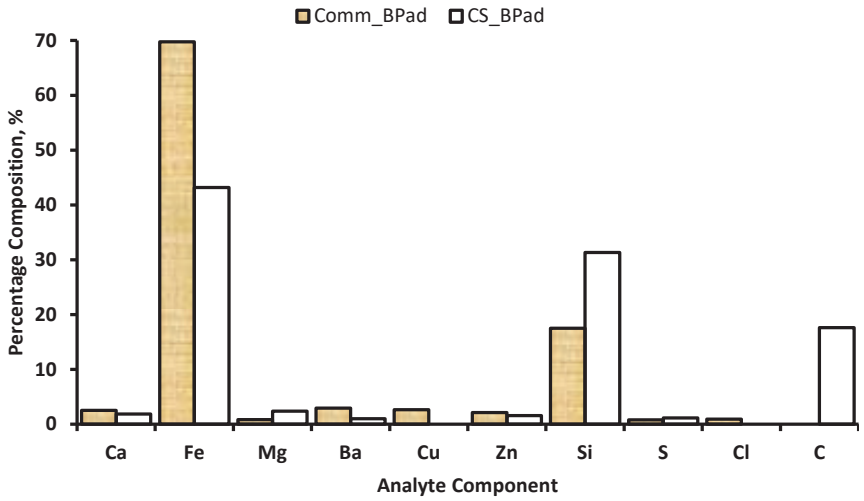

Figure 9. XRF analysis of control sample and CS-based samples with different grain size.

475°C, 0.4% (approximately 0.03 g) of the 500 μm grain size CS-based sample was left at 450°C, and 20% (approximately 1.2 g) of the 850 μm grain size CS-based sample was left at 475°C (Figure 8) compared to 86% (approximately 11.4 g) at 475°C for the control sample. Frictional heating derived from braking is stored in the CS-based brake pads rather than being conducted or diffused from the interface, thus causing the brake pad material to be significantly charred. Hence, we can say that the control brake pad sample produced a thermal stability characteristics better than the CS-based brake pad samples. This corroborates the thermal conductivity results in Figure 7 when comparing the CS-brake pad samples to the control sample.

3.8. Chemical composition by X-ray fluorescence spectroscopy

Two X-ray fluorescence (XRF) spectroscopy tests were carried out in vacuum environment for each sample in order to confirm the results obtained. A comparison of the major elements of the CS-based brake pad and that of the control brake pad is shown in Figure 9. The results indicated higher concentration of iron filings and lower Si impurities in the control brake pad compared to the CS-based brake pad. The higher Si impurity in CS-based brake pad can be attributed to handling and storage in the brake pad development process. The carbon component in the CS-based brake pad was obviously derived from the carbon occurring naturally in CS particles and the carbon friction modifier added during formulation. Figure 9 shows that the thermal stability and conductivity of the CS-based brake pad can be further enhanced by increasing the percentage of Fe particles in the matrix.

3.9. Finished CS-based brake pad

The finished CS-based brake pad with a back plate in place is shown in Figure 10. It has a groove in the middle to enhance the dissipation of heat from the interface and release pressure between the pad and the disk under emergency severe braking. It also serves as drainage for fluid (oil, water, and debris) transported away from the interface.

Figure 10. Finished CS-based brake pad sample.

4. Conclusion

- There was better interfacial bonding between the CS-based (*C. Nucifera*) particles and the binder as the particulate grain size decreased.
- The CS-based brake pad with 90 μm grain size performed better than the control (commercial) brake pad in all physicomechanical tests except for thermal conductivity and thermal stability tests.
- The hardness, compressive strength, and density test values decreased with increase in grain size.
- The absorption (water and oil) properties of the CS-based brake pad samples increased with increase in grain size due to enhanced porosity.
- Further reduction of the particle grain size below 90 μm and impregnation of the matrix with metals of good thermal conductivity (such as iron filings) will improve the physicomechanical properties of the CS-based brake pad.

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Conflict of interest

None declared.

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