Characterization of Powdered Pentaclethra Macrophylla Pod as a Friction Material

[1]2Iloabachie, I. C. C., [2]Nwankwo, A. M., [3]Nzoiwu, C. F., [4]Aneke V. I.
[1]Department of Mechanical Engineering, University of Environmental and Agricultural Science, Umuagwo, Imo State.
[2]Division of Works and Engineering Services. Federal Polytechnic, Oko, Anambra State
[3]Department of Mechanical Engineering, Federal Polytechnic, Oko, Anambra State
[4]Department of Agricultural and Bio-Environmental Engineering, Federal Polytechnic, Oko, Anambra state

Abstract - This study characterized the particle density and particle size distribution (PSD) of powdered Pentaclethra macrophylla pod in un-carbonized and carbonized states, with a view to assessing its suitability as reinforcement in composite and friction materials. Dry pods were cleaned, washed, oven-dried, milled and sieved in accordance with BS 1377:1990. Particle density was determined by psychometry, while PSD was evaluated by standard sieve analysis and gradation parameters were derived from semi-logarithmic plots. The particle density of the un-carbonized powder was 2.95 g/cm³, reducing to 2.65 g/cm³ after carbonization, which is attributed to the removal of volatile constituents and bio-impurities. The carbonized powder exhibited a well-graded distribution with D_{10} = 176.86 µm, D_{30} = 491.70 µm, D_{60} = 1287.84 µm, Cu = 7.28 and Cc = 1.06, whereas the un-carbonized powder was poorly graded with $D_{10} \approx$ 160 µm, $D_{30} \approx$ 260 µm, $D_{60} \approx$ 510 µm, Cu = 3.19 and Cc = 0.83. The well-graded nature of the carbonized powder is expected to enhance packing efficiency, reduce voids and improve matrix–filler interfacial bonding, which are critical for brake pad and polymer composite applications. Overall, the results indicated that carbonization improves the gradation characteristics of Pentaclethra macrophylla pod powder and supports its use as a sustainable reinforcement in engineering composites.

Keywords: Density, particle size, carbonized, un-carbonized, distribution.

1. INTRODUCTION

Automotive brake pads must deliver stable friction, high wear resistance, and adequate thermal stability while remaining lightweight and cost-effective [1]. Historically, asbestos-based friction materials fulfilled many of these requirements but are now restricted due to their carcinogenicity and environmental toxicity, prompting a global shift toward asbestos-free, eco-friendly friction materials [2]; [3].
Natural fibers and agro-waste fillers (e.g., coconut shell, cocoa bean shell, maize husk, palm kernel shell, bush mango nutshell, palm fruit fiber) have been widely investigated as sustainable alternatives in brake pad formulations, often achieving properties comparable to or better than commercial pads [4], [5], [6], [7], [8], [9]. Within this context, lingo-cellulosic biomass from Pentaclethra macrophylla (African oil bean) pods has emerged as a promising resource. Studies on this pod biomass have reported high cellulose content, significant levels of silica and alumina, good thermal stability, and favorable morphology and porosity, all of which are desirable for polymer reinforcement and potential tribological applications [10], [11], [12]. Heat-treated and carbonized P. macrophylla pod powders contain hard oxides (SiO_2, Al_2O_3, Fe_2O_3) that can act as abrasives and friction modifiers in brake pads [13] and have already been suggested as suitable particulate reinforcements in resin-based composites [14], [15]. Optimization work on brake pads reinforced with powdered P. macrophylla pod has shown promising hardness and mechanical behavior at specific particle sizes and weight fractions [15], [8].

However, existing studies largely focus on chemical composition, cellulose extraction, nano-cellulose production, or single-property optimization, rather than a systematic tribological characterization of powdered P. macrophylla pod explicitly as a friction material. This, therefore, has created avoidable knowledge gaps which include:

- Comprehensive evaluation of friction coefficient, wear behavior, and thermal response of P. macrophylla pod–based friction composites over relevant operating ranges;
- Correlation between particle size, carbonization state, and frictional performance; and
- Benchmarking against standard and other agro-waste-based friction materials [4], [5], [2], [6], [7], [15], [9].

Addressing these gaps is essential to establish powdered P. macrophylla pod as a technically viable and locally available friction material for asbestos-free brake pad applications.

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2. MATERIALS AND METHODS

2.1 Materials

The materials used in this research work were dry *Pentaclethra Macrophylla* pods and distilled water.

2.2 Methods

2.2.1 Preparation and Sieving of dry *Pentaclethra Macrophylla* pods

The dry *Pentaclethra Macrophylla* pods were manually cleaned by removing dirt particles on the surface, washed with distilled water, and sun-dried for about six hours. This was followed by oven drying at a temperature of about 110 °C for three hours to achieve a constant weight, and allowed to cool. The dried *Pentaclethra Macrophylla* pods were pulverized to a powdered form using a locally fabricated crushing and milling machine and later sieved. The sieving was carried out using a set of sieves arranged in descending order of fineness in accordance with BS1377:1990 standard, as was reported by [1] at the Civil Engineering Department soil laboratory of the Institute of Management and Technology, Enugu.

2.2.2 Particle Density Determination Using Pycnometry

The density of the powdered *Pentaclethra Macrophylla* pods was determined using a pycnometry bottle. The weight of the empty pyconometer with known volume was measured using a digital scale, Precisa XB6200D weighing balance, and recorded as w_0. The pycnometer was filled with quantities of powdered *Pentaclethra Macrophylla* pod, and the weight of the pycnometer with the powdered *Pentaclethra Macrophylla* pods was determined using a digital weighing balance and recorded as w_1. The difference in weight w_1-w_0 was used to calculate the weight of the powdered *Pentaclethra Macrophylla* pods. Density calculation of the powdered *Pentaclethra Macrophylla* pod was determined using the formula:

$$Density = \frac{Mass}{Volume\ of\ Sample} \quad (1)$$

3. RESULTS AND DISCUSSION

3.1 Particle Density Measurement:

The density of the particles of the *Pentaclethra Macrophylla* pod is as presented in Table 1. The particle density of un-carbonized and carbonized *Pentaclethra Macrophylla* pod was found to be 2.95 g/cm^3 and 2.65 g/cm^{3,} respectively. The result showed a reduction in the particle density of the carbonized *Pentaclethra Macrophylla* pod compared to the un-carbonized one. This may be attributed to the heat treatment of the *Pentaclethra Macrophylla* pod by carbonization, which resulted in the release of volatile matter and other bio impurities [13]. This result agreed with the work of [16], where a similar trend was established via chemical treatment of egg shell and limestone powders using stearic acid.

Table 1: Particle Size Density of Powdered *Pentaclethra Macrophylla* Pod

Reinforcement Particle	Density g/cm^3
Un-carbonized	2.95
Carbonized	2.65

3.2 Particle Size Distribution Analysis

Mechanical analysis is the determination of the size range of particles present in a powdered material expressed as a percentage of the total dry weight. Figures 1 and 2 represent the particle size distribution curves of the powdered un-carbonized *Pentaclethra Macrophylla* Pod and the powdered carbonized *Pentaclethra Macrophylla* Pod respectively. From the graphs, the powdered un-carbonized *Pentaclethra Macrophylla* Pod showed a broader particle size distribution range than that of the powdered carbonized *Pentaclethra Macrophylla* Pod. The observed narrower particle size distribution range in powdered carbonized *Pentaclethra Macrophylla* Pod may be attributed to carbonization which significantly reduced the particle size of the *Pentaclethra Macrophylla* Pod. This suggests that the carbonization process may have removed bio-organic impurities in the *Pentaclethra Macrophylla* Pod and also, possibly reduced agglomerated particles of the *Pentaclethra Macrophylla* Pod into smaller particle sizes. This is in line with the works of [16] and [6] where treatment of egg shell and limestone shell using stearic acid established similar trend.

The particle size distribution of the carbonized *Pentaclethra macrophylla* pod powder was evaluated using standard sieve analysis, and the resulting gradation curve is as presented in Fig. 3.1. The distribution curve, plotted on a semi-logarithmic scale, exhibits a smooth and continuous profile, indicating a well-graded particulate system.

The characteristic particle sizes obtained from the curve included D_{10} = 176.86 µm, D_{30} = 491.70 µm, and D_{60} = 1287.84 µm. These values were used to compute the gradation coefficients: the coefficient of uniformity (Cu) and the coefficient of curvature (Cc),

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which were found to be 7.28 and 1.06, respectively. The Cu value greater than 4 suggests a wide range of particle sizes, while the Cc value between 1 and 3 confirms a well-graded distribution.

The implication of this gradation is significant for composite fabrication. A well-graded particle system enhances packing efficiency, reduces void content, and improves interfacial bonding between the reinforcement and matrix. This contributes positively to load transfer efficiency and mechanical integrity of the composite material. Furthermore, the presence of both coarse and fine particles promotes better surface contact and frictional characteristics, which are critical for brake pad applications.

The relatively high D_{60} value indicates the presence of coarser particles, which can improve thermal stability and wear resistance, while the finer fraction (D_{10}) contributes to matrix filling and cohesion. Overall, the particle size distribution of the carbonized *Pentaclethra macrophylla* pod demonstrates suitability as a reinforcement material in polymer-based brake pad composites, particularly in achieving balanced mechanical and tribological performance.

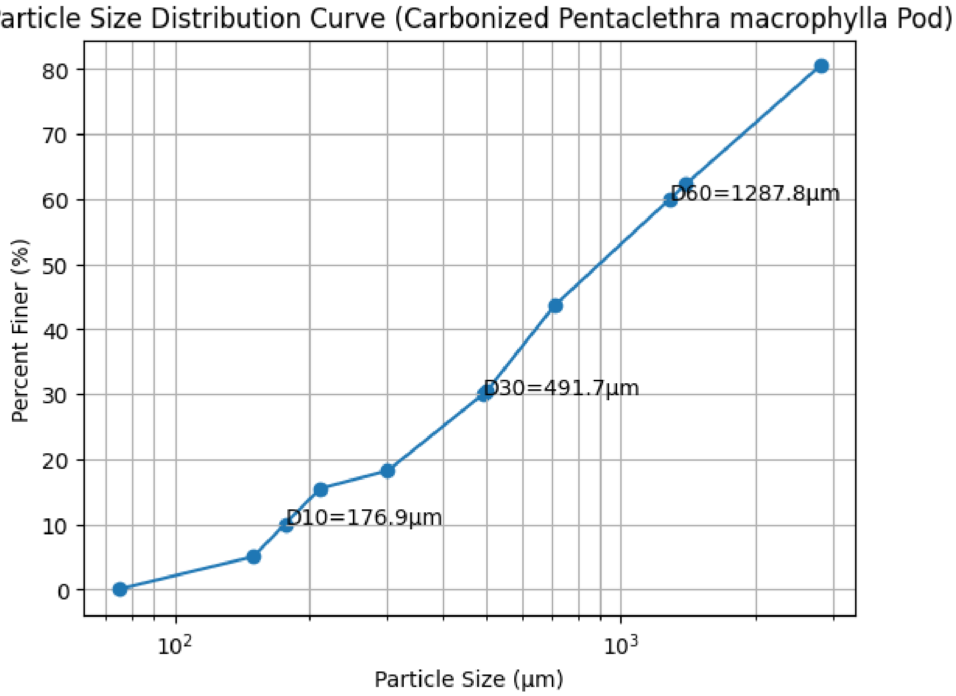

Figure 1: Particle Size Distribution Curve of Carbonized Powdered Pentaclethra Macrophylla Pod.

The particle size distribution of the un-carbonized *Pentaclethra macrophylla* pod powder was evaluated using standard sieve analysis, and the resulting gradation curve is presented in Fig. 2.

The PSD curve exhibits a characteristic sigmoidal (S-shaped) profile, typical of lingo-cellulosic agro-waste particulates. A significant proportion of the particles lies within the intermediate size range of 212–710 µm, as evidenced by the steep slope of the curve within this region. This indicates that the powder contains a dominant fraction of medium-sized particles, with relatively fewer coarse and fine particles.

The characteristic particle diameters corresponding to 10%, 30%, and 60% finer fractions were determined from the PSD curve as:

- D10 ≈ 160 µm
- D30 ≈ 260 µm
- D60 ≈ 510 µm

These values were further used to evaluate the gradation characteristics of the powder. The coefficient of uniformity (Cu) and coefficient of curvature (Cc) were calculated as:

$$Cu = \frac{D60}{D10} = 3.19 \qquad (2)$$

$$Cc = \frac{D30XD30}{D10XD60} = 0.83 \qquad (3)$$

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The obtained values indicate that the un-carbonized powder is poorly graded (uniformly graded), since the coefficient of uniformity is less than 4 (Cu < 4) and the coefficient of curvature lies outside the recommended range of $1 \le Cc \le 3$. Poor gradation suggests a relatively narrow particle size distribution, which can influence packing density and inter-particle bonding in composite formulations.

From a materials engineering perspective, the dominance of medium-sized particles enhances mechanical interlocking but may limit void filling due to the insufficient presence of finer particles. Consequently, this gradation may affect the densification, porosity, and tribological performance of the resulting composite, particularly in applications such as brake pad development where particle packing and surface interactions are critical.

Furthermore, the relatively low percentage of fines (<150 μm) suggests reduced surface area, which may influence resin–filler adhesion and overall composite strength. However, the presence of coarse fractions contributes to load-bearing capacity and wear resistance.

Overall, the PSD characteristics of the un-carbonized *Pentaclethra macrophylla* pod powder indicate its suitability as reinforcing filler in polymer composites, although optimization through particle size blending or controlled milling may be required to achieve improved gradation and enhanced performance properties.

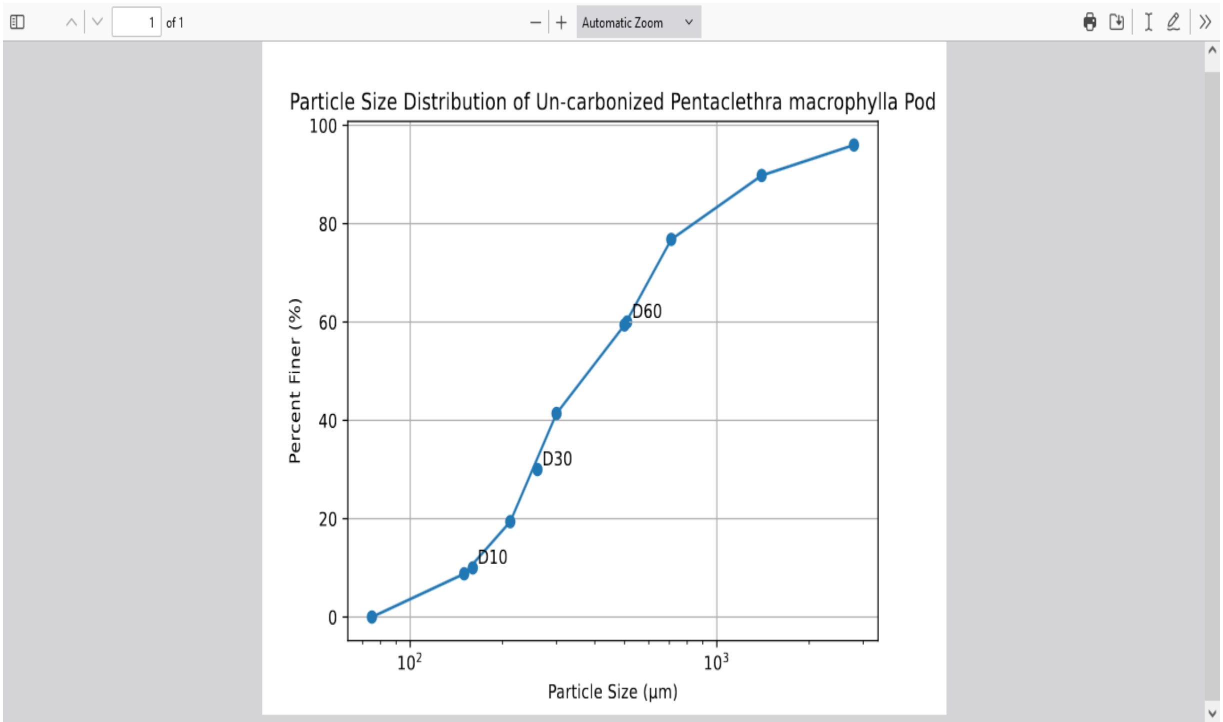

Figure 2: Particle Size Distribution Curve of un-carbonized Powdered Pentaclethra Macrophylla Pod.

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Figure 3: Pentaclethra Macrophylla pod

Figure 4: Un-carbonized Powdered Pentaclethra Macrophylla **pod**

Figure 5: Carbonized Powdered Pentaclethra Macrophylla **pod**

Figures 3 -5 shows the Pentaclethra macrophylla pod and the uncarbonized pod and carbonized pod of Pentaclethra macrophylla.

4. CONCLUSION

The physical characterization of Pentaclethra macrophylla pod powder demonstrated that carbonization and controlled milling and sieving significantly modified its density and particle size distribution, with important implications for composite applications. The reduction in particle density from 2.95 g/cm³ for the un-carbonized powder to 2.65 g/cm³ for the carbonized powder is consistent with the loss of volatile matter and organic impurities during heat treatment. Sieve analysis revealed that the carbonized powder

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possesses a well-graded PSD (Cu = 7.28, Cc = 1.06), in contrast to the poorly graded un-carbonized powder (Cu = 3.19, Cc = 0.83). This improved gradation is expected to enhance particle packing, minimize void content, and strengthen matrix–filler interactions, thereby improving load transfer, wear resistance, and frictional stability in brake pad and related composite systems. Consequently, carbonized Pentaclethra macrophylla pod powder emerges as a promising, sustainable reinforcement for polymer-based friction materials and other engineering composites, although further mechanical and tribological testing in full composite formulations is recommended to optimize its utilization.

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Authors' contributions

Conceptualization, I. Iloabachie, and A. Nwankwo; **data curation**, A. Nwankwo; **formal analysis**, A. Nwankwo, **investigation**, I. Iloabachie, **methodology**, I. Iloabachie. **project administration**, A. Nwankwo, **resources**, I. Iloabachie; **software**, I. Iloabachie, **supervision**, I. Iloabachie; **validation,** A. Nwankwo, and I. Iloabachie; **visualization**, A. Nwankwo; **writing—original draft preparation**, I. Iloabachie, A. Nwankwo, **writing, reviewing, and editing**, I. Iloabachie and A Nwankwo,

- All authors have read and agreed to the published version of the manuscript.

Authors' information

- **AMN** is currently a Director at the Directorate of Works and Engineering Services, Federal Polytechnic Oko, Anambra State. **ICI** is a Senior lecturer, Department of Mechanical and Production Engineering, University of Environmental and Agricultural Science, Umuagwo, Imo state.

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