
| RESEARCH ARTICLE**Eco-Friendly Brake Pad Formulation Using Agro-Waste Derived Fillers: Bush Mango Nutshell and Palm Fruit Fiber Reinforced Composites****Ifeanyichukwu Ugochukwu Onyenanu¹ ✉ Ifeanyi Ofili² and Kennedy Chinedu Owuama³**¹²³*Department of Mechanical Engineering, Chukwuemeka Odumegwu Ojukwu University, Uli – Nigeria.***Corresponding Author:** Ifeanyichukwu Ugochukwu Onyenanu, **E-mail:** iu.onyenanu@coou.edu.ng

| ABSTRACT

The increasing demand for eco-friendly automotive materials necessitates the replacement of carcinogenic asbestos in brake pads. This study investigates the development of asbestos-free brake pads using bush mango shells (*Irvingia gabonensis*) and palm fruit fibre (PFF) as sustainable fillers. Response Surface Methodology (RSM) optimized the formulation, focusing on wear rate, compressive strength, and hardness. The optimal brake pad composition, containing 30% BMS/PFF with a 300 μm particle size, exhibited competitive mechanical and wear properties: wear rate (2.97-3.96 mg/m), compressive strength (74.66-148 MPa), and hardness (94-104 HRB). Thermal stability was maintained between 200°C and 550°C. This eco-friendly formulation presents a viable alternative to asbestos, supporting sustainable automotive manufacturing.

| KEYWORDS

Asbestos-free brake pads, bush mango shell, palm fruit fibre, sustainable materials, Response Surface Methodology.

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1. Introduction

A brake is a device that inhibits motion [Onyenanu, 2021]. A friction brake is a type of automotive brake that slows or stops a vehicle by converting kinetic energy into heat energy through friction [Limpert, 2011]. Friction between brake pads and rotating discs causes the vehicle to stop. Without a brake system, the vehicle will put passengers in an unsafe position [Ukwu, 2016]. Numerous components make up the braking system, such as wheel cylinders, a hydraulic control system, a master cylinder, and brake pads on each wheel. Friction material, which is the heart of the braking device, is predicted to function dependably and effectively for an extended period under challenging operating conditions [Kumar, 2010]. Different braking systems use different kinds of brake pad material. Four common constituents are used to classify them: binders, fillers, friction modifiers, and reinforcements. In addition to a few other ingredients, asbestos fibres embedded in a polymeric matrix typically make brake pads. Beginning in the early 1900s, asbestos fibre was used as reinforcement in friction materials. Asbestos fibre satisfied the primary need for brake friction material, leading to the global popularity of asbestos-based friction composites.

When creating brake materials for the automotive industry, several factors need to be considered to meet specifications, like a stable friction coefficient and a lower wear rate at different operating speeds, pressures, temperatures, and environmental conditions [Wannik, 2012]; [Adebisi, 2024]. Because of this, it's critical to have the right mix of materials at a fair price to fulfil those specifications. The process of choosing the content is intricate and not at all simple [Maleque, 2012]; [Shaha, 2010]. Therefore, to create a new formulation for any application,

including brake pad material, the constituents are frequently based on experience or a trial-and-error process [Maleque, 2012]

The variables that are first "screened" to ascertain which are crucial to the result are handled by the design of the experiment (DOE) method of approach (excipients type, percentage, disintegration time (DT), etc.). Optimization refers to the process of identifying the optimal configurations for the significant variables. Response Surface Methodology (RSM) is used to determine the best operating conditions for a system under study or a portion of the factor field that meets the operating requirements. It also entails investigating an appropriate approximation relationship between input and output variables. The best formulations and ideal grain size for bush mango shell (BMS) and palm fruit fibre (PFF) are investigated for use in the development of asbestos-free brake pads and presented in this work.

Automobile brake lining friction materials must meet several performance requirements. In addition to being low-cost and, if feasible, lightweight, they must offer a stable, consistent level of friction, outstanding wear resistance, sufficient heat dissipation, and structural integrity. No single material can satisfy every one of these frequently incompatible performance requirements. The performance attained is largely dependent on the choice of materials, but up until now, the development of braking material compositions has primarily relied on empirical observations. [7]. One of the most important factors in achieving desirable friction and wear in automotive engineering practices, such as brake pads and discs, is selecting the appropriate formulation of friction materials [8]. Vehicle component imports are increasing significantly in Nigeria. As a result, this thesis discusses the problem and supports the local content campaign. Moreover, a new material that replaces asbestos as a friction material while preserving its mechanical qualities must be developed because asbestos is carcinogenic and cannot be used in the automotive industry or for any other purpose.

This paper introduces a new formulation for brake pads using locally sourced bush mango nutshell (BMS) and palm fruit fibre (PFF). The experiments were conducted using a Split-optimal custom design in Design Expert (13.0). Two main components—BMS/PFF and binder—were varied while others remained constant. Product attributes such as wear rate, friction coefficient, compressive strength, and hardness were measured. The optimal BMS/PFF content was determined based on these responses.

1.1 Aim and Objectives of the Study

The study aims to formulate an eco-friendly brake pad using agro-waste derived fillers: bush mango nutshell and palm fruit fibre reinforced composites. To achieve this, there are specific objectives to pursue, which are;

1. To develop an experimental design for brake pad formulation and prepare the experimental samples.
2. To investigate the mechanical properties of the produced BMS/PFF brake pad samples.
3. To optimize the experimental design table and compare the actual and predicted values using the coded equations, evaluate interactions between the factors and the responses, and validate the experiment using ANOVA
4. To investigate the pad material's thermal decomposition, microstructure, and elemental composition.
5. To study the correlation between the newly formulated and commercial brake pads.

2. Methodology

2.1 Materials

The material and equipment used during this work are epoxy resin, palm fruit fibre (PFF), bush mango nutshell (BMS), aluminium oxides, iron oxide, silica, graphite, and metal chips (Iron filings). Table 3.1 shows the categories of the brake pad material. Furthermore, the material descriptions used to obtain the characteristics are explained below;

Table 3.1: The categories of brake pad material and material choice.

S/N	Brake pad material	Material choice
1	Binder Matrix	Epoxy Resin
2	Reinforcement	Aluminium oxide and iron filings
3	Filler	Palm Fruit Fiber/ Bush Mango Nutshell
4	Abrasive	Iron Oxides and Silica
5	Friction modifiers	Graphite

2.2 Equipment

The equipment that was used for this study includes a set of sieves, a steel spatula, a stirrer, bowls, hand gloves, metal files, a mobile hardness tester, a furnace, a milling machine, an inertial dynamometer, an inclined plane, calipers, water, and engine oil. Hydraulic press, Brake pad mould, Heater, Scanning Electron Microscope (SEM), XRF, TGA, FTIR, and Digital Weighing Machine.

3. Methods

The methodology of this study, which will cover the overall process discussed in the chapter, is summarized in Figure 3.1. The process can be classified into three (3) sections, i.e., characterization of the raw materials, preparation of the samples, and characterization of the products.

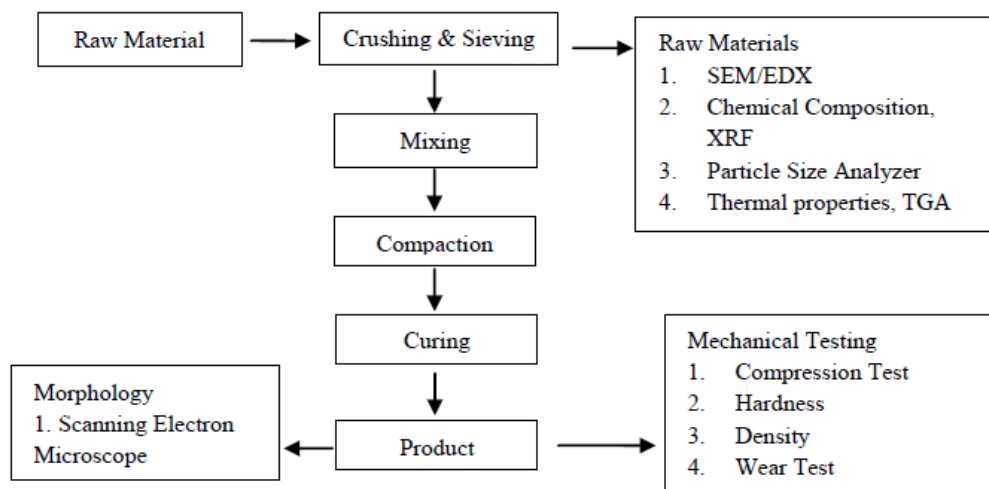


Figure 3.1: Schematic diagram for the development of the BMS/PFF brake pad

3.1 Design of Experiment

To determine the optimum fibre content of BMS/PFF in the brake pad, a split-plot optimal (custom) design was employed using Design Expert Software 13.0. The split-plot optimal (custom) design is a specialized form of response surface method. The runs are determined by a selection criterion chosen during the build.

Table 3.2: Showing the Design of the Experiment setup

	Name	Units	Change	Type	Levels	L[1]	L[2]	L[3]
c [Numeric]	BMS/PFF	wt%	Hard	Discrete	3	30	35	40
d [Numeric]	Binder (Epoxy)	wt%	Hard	Discrete	3	10	15	20
e [Categorical]	Grain Sizes of	Micrometer	Hard	Nominal	2	300	600	

This means that the weight percent of Epoxy must fall in the range of 10% to 20% while that of BMS/PFF must fall in the range of 30% to 40%. These components represent 50% of the total weight percent of the total formulation. Table 3.3 shows the formulation according to the Software.

NB: It may be noted that the composition of BMS/PFF is in the ratio of 1:1.

Table 3.3: Weight percent of BMS/PFF and Epoxy according to Design Expert Software

	Component 1	Component 2	Factor 3	Factor 4	Factor 5	Response 1	Response 2	Response 3
Run	A: A	B: B	c: BMS/PFF	d: Binder (Epoxy and Resin)	e: Grain Sizes of BMS/PFF	Wear Rate	Compressive Strength	Hardness
	wt%	wt%	wt%	wt%	Micrometer	mm ³ /m	N/mm ²	HRB
1	25	25	30	20	600			
2	50	0	30	20	600			
3	0	50	30	20	600			
4	0	50	30	10	300			
5	50	0	30	10	300			
6	25	25	30	10	300			
7	0	50	40	20	300			
8	25	25	40	20	300			
9	50	0	40	20	300			
10	25	25	40	10	600			
11	0	50	40	10	600			
12	50	0	40	10	600			

NB: The other materials (Alumina, Iron filings, silica, Iron oxide, and graphite) are held constant and complete the remaining 50% of the formulation as shown in Table 3.5. For this experiment, they are ignored.

3.2 Sample Preparation

Palm Fruit Fiber (PFFs): About 10 kg of PFFs were collected from a local mill in Enugu. The fibres were soaked in a sodium hydroxide solution for 24 hours to remove any remaining oil, washed with water, and sun-dried for one week. The dried fibres were ground into powder using a grinding mill.

Bush Mango Nut Shell (BMS): 10 kg of BMS was collected from various sources in Enugu, sun-dried, and crushed using a jaw crusher. The crushed material was then further ground into smaller sizes using a high-speed grinder.

Sieving: Both BMS and PFF were sieved using a UTS Sieve shaker to obtain uniform particle sizes. The particles were separated into two sizes: less than 300 µm and 600 µm.

3.3 Sample Formulation

Each powdered constituent was weighed and mixed thoroughly for 15–20 minutes to achieve homogeneity. Epoxy resin and hardener, in a 2:1 ratio, were combined and stirred for 5 minutes to form the matrix. This mixture was then added to the powdered friction material and stirred into a paste. The paste was poured into mould cavities pre-coated with powdered talc and pressed using a hydraulic press at 100kN for 2 minutes at room temperature. The samples were cured for 90 minutes and hardened in an oven at 150°C for 3 hours to ensure complete curing.

3.4 Optimization (Response Surface Methodology - RSM)

Response Surface Methodology (RSM) can be of first-order and second-order equations as shown below:

$$\eta = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \dots + \beta_k x_k ; \quad \dots \text{Eqn. (3.1)}$$

$$\eta = \beta_0 + \sum_{j=1}^k \beta_{1j} x_1 + \sum_{j=1}^k \beta_{jj} x_j^2 + \sum_{i < j} \sum_{j=2}^k \beta_{ij} x_i x_j \quad \dots \text{Eqn. (3.2)}$$

The relationship between the response variables and the independent variables (factors) was presented in the form of an equation below.

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{12} x_1 x_2 + \varepsilon ; \quad \dots \text{Eqn. (3.3)}$$

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{12} x_1 x_2 + \varepsilon \quad \dots \text{Eqn. (3.4)}$$

3.5 Brake Pad Manufacturing Process

After various tests were conducted on the various samples of the brake pad material, using the analysis of the results obtained from the test, the best three compositions were used to produce three brake pads.

Mould: A mould made of sheet metal to be used in the moulding process was fabricated. This was made in a shape identical to that of a brake pad already existing in the market. It was made by folding a sheet metal of height 3mm to the required shape and the joint was arc welded.

Moulding Process: Using the best compositions and the same method of producing the samples, new brake pads were produced and were cured at 250°C for 90 min after removal from the hydraulic press. The produced samples were finished by polishing them using a polisher-grinder with various grinding papers of various sizes to obtain the final products.

Finishing Process: The heat groove was cut by use of a grinding machine. The cut was done to a depth of about 3mm on the lining. The brake pad was finally painted to look presentable. The resulting pads are shown below.

4. Results and Discussion

4.1 Results of the Physical Properties of the Brake Pad Material

4.1.1 Density

Fig 4.1 shows the result of the densities of the new brake pad samples developed. From the result, sample B shows the highest density of 2.42g/cm³, followed by sample I, while sample G has the lowest density of 0.99g/cm³.

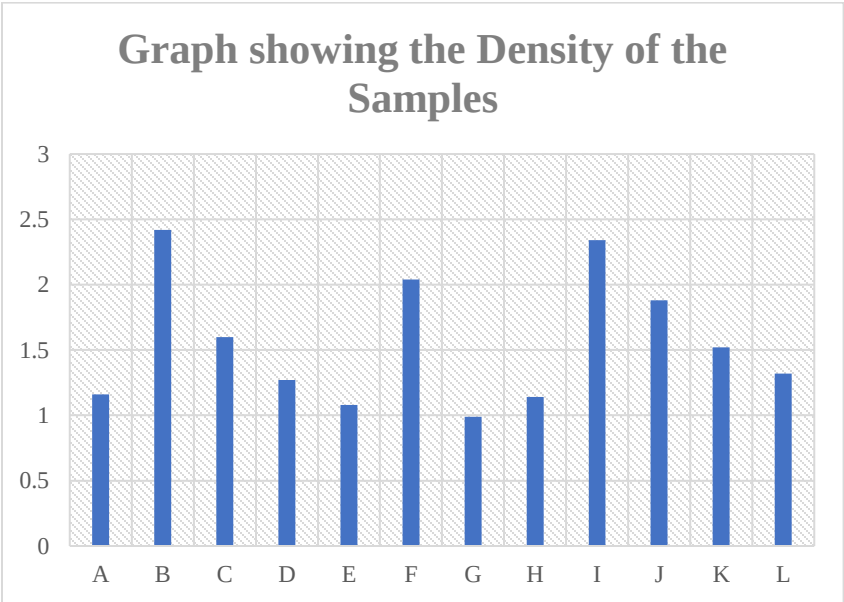


Fig 4.1: Plot of the Density values of the various BMS/PFF brake pad samples

4.1.2 Water and Oil Absorption Test

The water absorbent property of samples decreased sharply from sample A to B and even more to C before it started increasing again. Sample L shows the highest thickness swelling in water and oil at the rate of 13.2% and 2.24%, respectively, while sample C has the lowest thickness of swell at the rate of 1.86% and 0.89%, respectively.

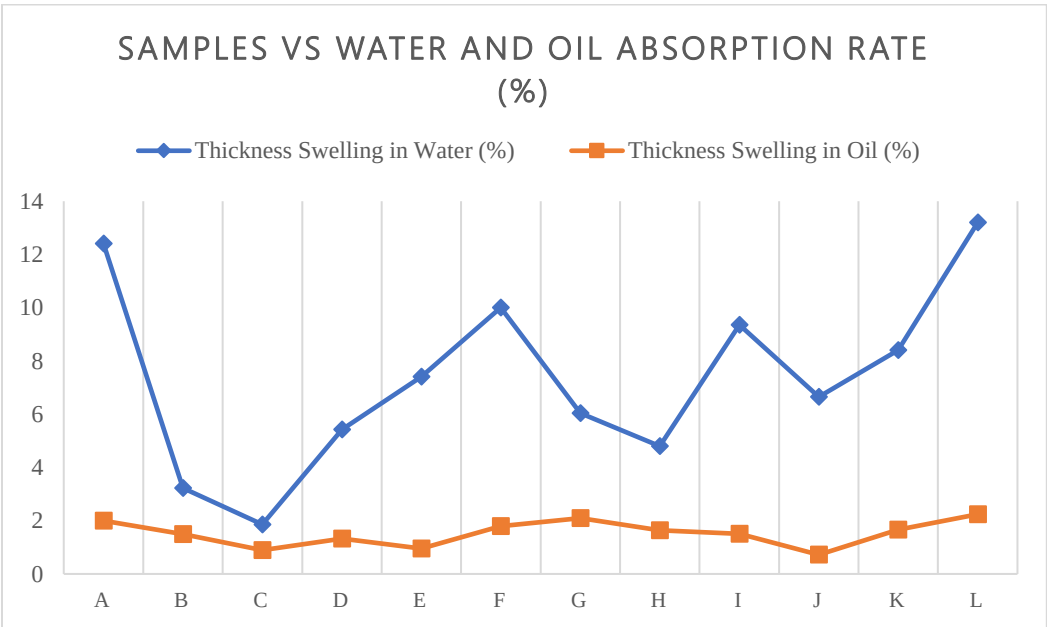


Fig 4.2: Plot of the water and oil adsorption rate of the BMS/PFF brake pad samples

4.2 Results of the Chemical and Morphological Properties of the Brake Pad Material

4.2.1 Chemical Composition

Chemical compositions and the concentration of elements in bush mango shells and palm fruit fibre are presented in Table 4.1. The data obtained from the XRF analyses of these two bios- materials also were compared with the properties of asbestos that were obtained from the literature.

Table 4.1: Concentration of compounds in different fillers (*determined by an XRF spectrometer*)

Compounds	Concentration (%)		
	Bush mango Shell	Palm fruit fiber	Asbestos (Dellisanti <i>et al.</i> , 2002)
SiO ₂	10.970	12.845	43.74
K ₂ O	12.969	13.456	0.43
CaO	23.501	28.451	16.97
MgO	13.258	15.245	6.13
Fe ₂ O ₃	8.544	5.789	14.87
Al ₂ O ₃	15.798	17.543	2.16
TiO ₂	1.467	1.345	0.23
NiO	0.015	0.012	-
CuO	0.823	0.982	-
P ₂ O ₅	1.533	2.544	-
ZnO	0.269	1.234	-
Cr ₂ O ₃	0.025	0.0245	-
MnO ₂	0.430	0.543	1.31
SO ₃	4.430	5.431	-
Cl	5.386	9.854	-

4.2.2 Scanning Electron Microscopy Results

The micrographs shown in Figure 4.3 are that of the Optimum brake pad at different magnifications. The micrograph depicts the microstructural distribution of the binder resin with bush mango nutshell (BMS)/palm fruit fibre (PFF) and other additive and their bonding effect. The result showed that the fibre particles were not evenly distributed due to the nature of its sieve size, which resulted in a coarse texture of the brake pad.

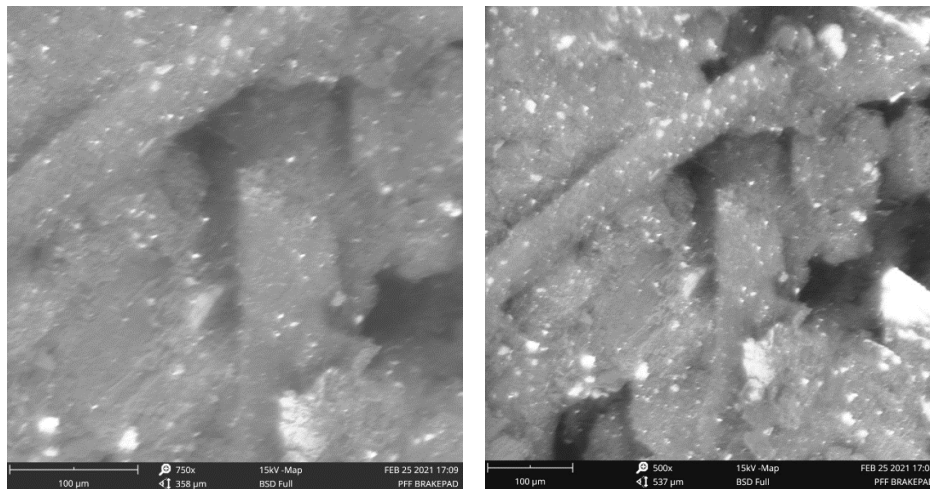


Figure 4.3: Micrographs of BMS/PFF brake pad sample

4.3 Results of the Mechanical properties of the Brake Pad material

4.3.1 Hardness

The hardness values can be seen in Figure 4.4. It can be observed that the hardness values conform with SAE standards (SAE J2654_2015 08 - Hardness of Break lining stabilized August 2015). The sample with a sieve particle size of 300 – 315µm gave higher hardness values than their counterparts (i.e., those from 600 – 630µm sieve). The sample with the highest hardness number is sample F, with a BMS/PFF composition of 30% and 300–315µm sieve size, whereas the sample with the lowest hardness value is sample D, with a BMS/PFF composition of 30% and 300 – 3150µm but having just only PFF.

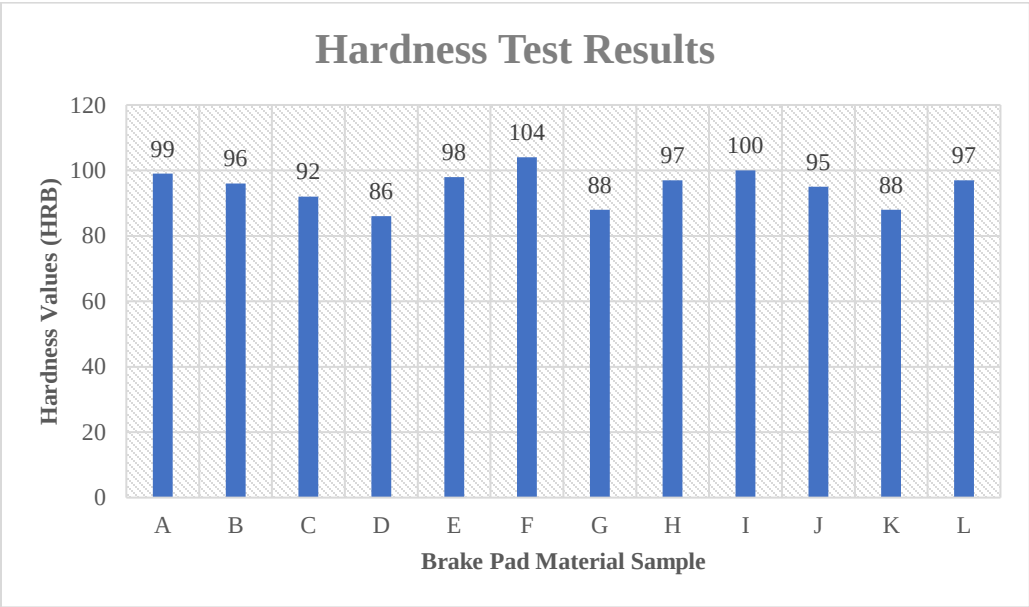


Figure 4.4: Hardness Test Result

4.3.2 Compressive Strength

The compressive properties of the samples are presented in Figure 4.5. it can be observed that the compressive strength of the samples falls almost in the same range, with Sample F being the highest with a compressive strength of 144.33 N/mm²; the difference was not that much except for Sample K which has the lowest compressive strength of 71 N/mm².

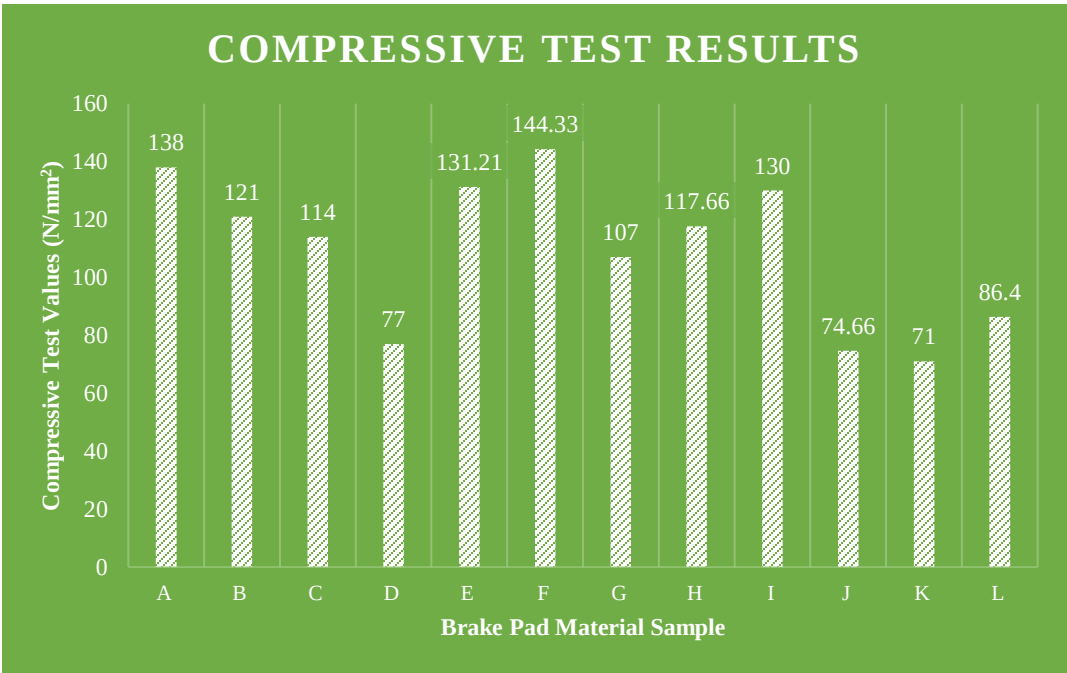


Figure 4.5: Compressive Strength Result of the samples

4.3.3 Wear Rate

The wear results of the samples are shown in Figure 4.6. The sample with the least wear rate is sample D, with a wear rate of 2.97mg/m, whereas sample B has the highest wear rate, with a wear rate of 3.96mg/m. It can be

deduced that there is no significant difference between the rate of wear of the sample materials since the difference between the highest and lowest wear rate is 0.99mg/m. However, the least wear rate value is desirable.

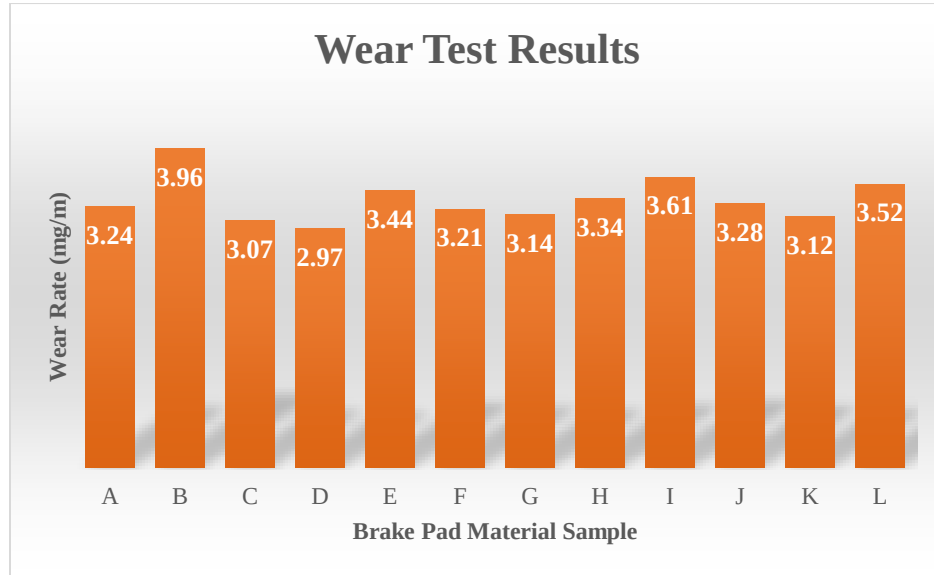


Figure 4.6: Wear Results of the samples

4.3.4 Coefficient of Friction

The coefficient of Friction of the brake pad material, as shown in Figure 4.7, was found to be in the range of 0.31 – 0.36.

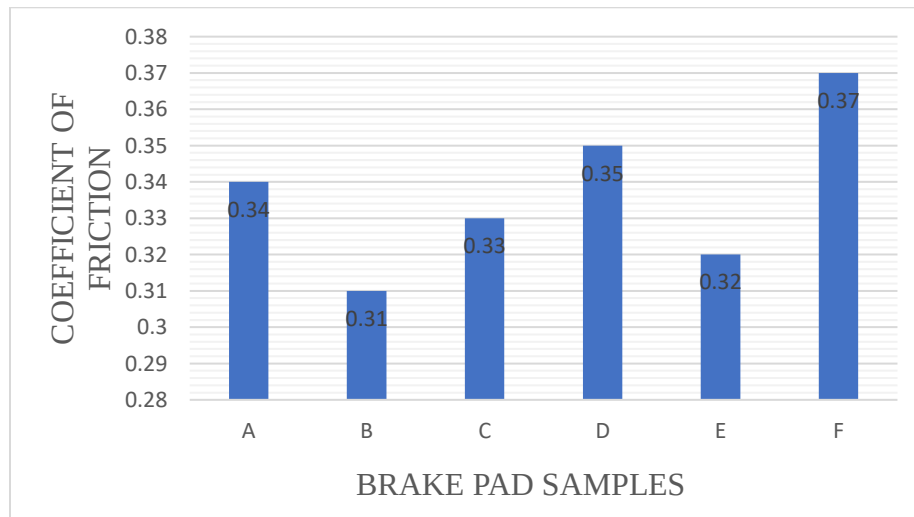


Figure 4.7: Coefficient of friction Result

4.4 Results of the Optimization Analysis using Response Surface Methodology (RSM)

A preliminary study on BMS/PFF brake pad formulation was done by investigating the components that made up the disc material. Three excipients were chosen for BMS/PFF formulation based on their function. Two of them and grain sizes were used as variables in the I-optimal mixture design as they may affect the responses. The ranges of variables were also studied by using an I-optimal mixture in design expert software. Table 4.2 shows the summary data table of the actual design after the experiment.

Table 4.2: Summary of the Actual Design table for the optimization

		Component 1	Component 2	Factor 3	Factor 4	Factor 5	Response 1	Response 2	Response 3
Group	Run	A: A	B: B	c: BMS/PFF	d: Binder (Epoxy and Hardener)	e: Grain Sizes of BMS/PFF	Wear Rate	Compressive Strength	Hardness
		wt. %	wt. %	wt. %	wt. %	Micrometer	mm ³ /m	N/mm ²	HRB
1	1	25	25	30	20	600	3.24	138	99
1	2	50	0	30	20	600	3.96	121	96
1	3	0	50	30	20	600	3.07	114	92
2	4	0	50	30	10	300	2.97	77	86
2	5	50	0	30	10	300	3.44	131.21	98
2	6	25	25	30	10	300	3.21	144.33	104
3	7	0	50	40	20	300	3.14	107	88
3	8	25	25	40	20	300	3.34	117.66	97
3	9	50	0	40	20	300	3.61	130	100
4	10	25	25	40	10	600	3.28	74.66	95
4	11	0	50	40	10	600	3.12	71	88
4	12	50	0	40	10	600	3.52	86.4	97

4.4.1 Coefficient in Terms of Coded Factors

4.4.1.1 Wear Rate

The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs. The coefficients are adjustments around that average based on the factor settings. When the factors are orthogonal, the VIFs are 1; VIFs greater than 1 indicate multi-collinearity; the higher the VIF, the more severe the correlation of factors. As a rough rule, VIFs less than 10 are tolerable.

Table 4.3: Showing the Coefficients in Terms of Coded Factors for Wear Rate

Source	Coefficient Estimate	Standard Error	VIF
A-A	3.60	0.0523	1.04
B-B	3.05	0.0523	1.04
Ad	0.1310	0.0513	1.0000

For the Final Equation in Terms of L_Pseudo Components, the model is written as;

$$\text{Wear Rate} = +3.60 A + 3.05 B + 0.1310 Ad$$

For the Final Equation in Terms of Real Components, the model is written as;

$$\text{Wear Rate} = +3.21075 A + 3.04625 B + 0.026200 A * \text{Binder (Epoxy and Hardener)}$$

Therefore, the Final Equation in Terms of Actual Components is written as;

$$\text{Wear Rate} = +0.064215 A + 0.060925 B + 0.000524 A * \text{Binder (Epoxy and Hardener)}$$

4.4.2 Analysis of Variance (ANOVA)

Analysis of variance (ANOVA) is a collection of statistical models and their associated estimation procedures (such as the "variation" among and between groups) used to analyze the differences among group means in a sample. This method was utilized to predict if there is any significant difference in the mean of the experiment;

I: For the development of the **BMS/PFF Brake Pad**, the ANOVA for the selected factorial model is seen below;

Table 4.4: Response 1: Wear Rate

Source	Term df	Error df	F-value	p-value	
Subplot	2	9.00	26.91	0.0002	significant
Linear Mixture	1	9.00	47.30	< 0.0001	
Ad	1	9.00	6.53	0.0309	

P-values less than 0.0500 indicate model terms are significant. In this case, A, B, and Ad are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

II: For the development of the **BMS/PFF Brake Pad**, the ANOVA for the selected factorial model is seen below;

Table 4.5: Response 2: Compressive Strength

Source	Term df	Error df	F-value	p-value	
Subplot	5	3.78	2.66	0.1899	not significant
Linear Mixture	1	4.00	7.04	0.0568	
AB	1	4.00	2.95	0.1610	
Ac	1	3.13	0.5865	0.4974	
Bc	1	3.13	0.0773	0.7984	
ABC	1	4.00	4.07	0.1138	

P-values less than 0.0500 indicate model terms are significant. In this case, A and B are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

III: For the development of the **BMS/PFF Brake Pad**, the ANOVA for the selected factorial model is seen below;

Table 4.6: Response 3: Hardness

Source	Term df	Error df	F-value	p-value	
Subplot	6	5.00	10.30	0.0108	significant
Linear Mixture	1	5.00	34.85	0.0020	
AB	1	5.00	17.18	0.0089	
Ac	1	5.00	0.4582	0.5285	
Ae	1	5.00	2.94	0.1469	
Bc	1	5.00	0.2037	0.6707	
ABC	1	5.00	4.49	0.0876	

P-values less than 0.0500 indicate model terms are significant. In this case, A, B, and AB are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

4.5 Comparison of the BMS/PFF with Commercial and other formulated Brake Pads.

Relating the newly formulated Bush Mango Shell/Palm Fruit Fiber (BMS/PFF) Automobile Brake pad with that of Commercial brake pads and other formulated brake pads as shown in Figure 4.23. It can be deduced that the BMS/PFF compares favourably to commercial brake pads than the palm kernel shell and periwinkle shell, respectively, as reported by [Olabisi, 2016] and [Elakhame, 2014].

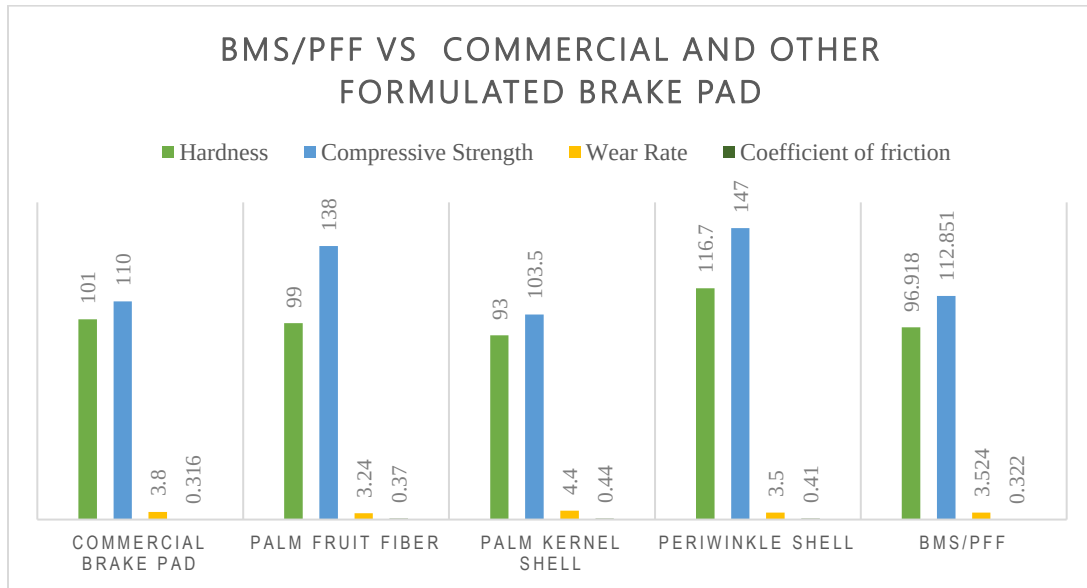


Figure 4.7: Comparison with the commercial Brake Pad

5. Conclusion and Discussion

Based on the mechanical and wear properties, this research indicated that bush mango shells can be used effectively as an alternative to other fillers in brake pad composites. Comparisons with other fillers showed that bush mango shell has significant potential for becoming an alternative material for use as the filler in brake pad composites. The densities of the bush mango shell composites also indicated that they were suitable for use in brake pads due to their lightweight, even when compared with asbestos.

The following were deduced from this study:

- The optimal sample for the brake pad material is a sample (30 wt% of BMS/PFF, 300 μ m), which is the closest to the predicted value.
- The best particle size of Bush Mango Nutshell (BMS)/Palm Fruit Fiber (PFF) for brake pad production is 300 μ m.
- The pad samples were thermally stable with no loss in weight up to 200 °C to 550 °C

5.1 Recommendation

The development and validation of a friction material involve a significant amount of testing in the laboratory and on the road. On-road brake tests with real-world conditions normally are the final tests that are performed to evaluate and validate the formulation. Thus, vehicle testing on a test track is the ultimate measure for the overall assessment of the performance of the brake pad composites.

- Further investigation of the performance and wear of the developed brake pads should be conducted for actual real-world road conditions.
- Further investigation also is needed to establish the effects of load, braking distance, and braking time on wear properties.

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