

## Effect of Process Parameters on the Physical Properties of Chemically Activated Carbon Composites as Organic Friction Lining Materials

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### ABSTRACT

Epoxy/activated carbon composites made of 0%CSAC:100%PKSAC, 50%CSAC:50%PKSAC and 100%CSAC:0%PKSAC with particle sizes of 60, 105 and 150 $\mu$ m and percent weight of 4, 6 and 8 has been fabricated. The composites were experimentally evaluated for physical properties performance such as thickness swelling (cold-water, hot-water and oil), water absorption test (cold-water, hot-water), and oil absorption test using box-behnken design and compared with two procured commercial brakepads (CB1 and CB2). The influence of particle size (XPS), reinforcement weight (XRW) and activated carbon type (XCSAC) on the physical properties was approximated by second-order multivariate polynomial models and response surfaces plotted using sigma plot software. The property response surface plots at mid value of reinforcement concentration (XRW) were used to study the process parameters effects on the swelling and absorption behaviours of the chemically activated carbon composites. Both cold-water and hot-water thickness swell surface plots exhibited convex shaped surfaces and operated within the boundaries of  $0.158 \leq TSCW \leq 0.778\%$ , and  $-0.252 \leq TSHW \leq 0.58\%$ , whereas oil thickness swell surface plot exhibited a convex shaped surface and operated within the boundaries of  $-0.190 \leq TSOIL \leq 0.385\%$ , but lower than TSCW. CB1 showed TSCW = 2.0%, TSHW = 0.44% and TSOIL = 0.17%, CB2 showed TSCW = 1.84%, TSHW = 0.895%, TSOIL = 0.607%. Both cold-water and hot-water absorption surface plots exhibited concave shaped surfaces and operated within the boundaries of  $4.34 \leq AbCW \leq 12.62\%$  and  $2.75 \leq AbHW \leq 10.96\%$ , whereas oil absorption surface plot exhibited a convex shaped surface and operated within the boundaries of  $3.08 \leq AbOIL \leq 9.49\%$ , with intersection at some points. CB1 showed AbCW = 1.81%, AbHW = 2.41%, AbOIL = 7.913%, CB2 showed AbCW = 1.12%, AbHW = 1.52%, AbOIL = 3.535%. This new formulations showed that within the boundary of reinforcement particle sizes of  $60 \leq XPS \leq 150\mu$ m and coconut shell activated carbon (CSAC) concentration of  $0 \leq XCS \leq 100\%$ , TSCW > TSHW > TSOIL. This showed that the reinforcement has high resistance to oil and hot water but less resistance to cold water in relation to swelling. In terms of absorption and swelling capabilities, AbCW > TSCW, AbHW > TSHW and AbOIL > TSOIL. It can be recommended that the process parameters are important inputs for the fabrication of friction lining composite samples using chemically activated carbon from agro-wastes as they greatly influenced their physical properties.

**Keywords:** Process Parameters, Physical Properties, Agro waste, Box-Behnken design, Activated Carbon, Composites

#### AIMS Research Journal Reference Format:

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## 1. INTRODUCTION

One of the concerns of polymer composite is its sustainability in moisture and oil related applications. Composite materials have been employed to produce new materials which exhibit excellent physical properties suitable for various applications such as in moist, water and oil operating conditions. Researches have been conducted for the purpose of developing diverse composite materials in order to evaluate its industrial applications. According to Daryoush and Musbah, (2011), latest research reports of polymer based composite materials have established various approaches for polymer formulations and have permitted the production of innovative products with ideal properties for distinctive application. During the past decade, natural fiber reinforced polymer composites are attaining most convenient applications in sea vehicles, various parts of automotive, medical devices, sporting goods and aerospace industry, due to their classical merits like lesser weight, minimum cost, resistance to corrosion and wear and elevated specific strength etc (Piyush 2015).

Athijayamani et al., (2010) stated that in order to predict the response parameters, an empirical, statistical method and theoretical or analytical methods must be followed in general. Maniya and Bhatt, (2016) conducted a study to develop a statistical method for evaluating the value of cotton using high volume instrument (HVI) to calculate cotton fibre properties. Prediction of surface roughness in drilling of glass fiber reinforced polymer (GFRP) composite materials has been performed using fuzzy logic rule-based modeling and ANOVA (Latha and Senthilkumar, 2010). Manickam et al., (2015) studied experimentally the mechanical performance of roselle fiber-Reinforced vinyl ester (RFRVE) composites and thereafter, studied the optimization of process variables in accordance to mechanical properties of RFRVE composite using the grey based-Taguchi method. In the study of Huseyin and Mustafa, (2006) where they used the multiple linear regression analysis, the yarn parameters of ring spun cotton yarns were predicted and confirmed that yarn properties were affected by fiber properties, number of yarns, twist and roving properties. Zykova et al.; (2015) studied the influence of particle size on water absorption capacity and mechanical properties of polyethylene-wood flour composites and concluded that an increase of the filler particle size decreases mechanical parameters and increases water absorption.

The capacity of water absorption and swelling thickness is directly related to the density, the presence of voids and the bond between the fiber and the matrix. This factor causes water to be trapped in the void and increase composite weight (Nunna et al.; 2012). The effect of different fibres wt. % on the water absorption characteristics of unidirectional sisal fibre reinforced epoxy composite was evaluated and stated that the water uptake percentage was determined to rise as an increase in fibres content (Gupta and Srivastava, 2016). Similarly, Zhong et al.; (2007) also evaluated the water absorption behaviour of sisal/urea-formaldehyde composites with differing fibres wt. % (10, 20, 30, 40, 50, 60 and 70%). The lowest water uptake for composite with 30 wt% fibres was only 0.98 wt%, credited to strong bonding between the fibres and matrix. Water uptake was found directly proportional to fibres concentrations when water absorption behaviour of unidirectional jute/epoxy composites was studied (Gupta and Srivastava, 2017).

Sapaun et al.; (2018) found that unidirectional sugar palm fibre/vinyl ester resin composite demonstrated the lowest value of water absorption as compared to bidirectional fibre composites. However, all the composites showed high water absorption compared to neat vinyl ester, which might be attributed to incompatibility between fibre and matrix that led to micro-bubble and voids. Masoodi and Pillai (2012) developed jute bio epoxy composites for water absorption measurement and stated that pristine epoxy absorbs less water as compared to jute bio epoxy composites. The reason was that jute fibre is polar thereby they attract more water. The impact of fibre's lengths on water absorption properties of pine needles fibres/phenol-formaldehyde composites was investigated and higher water absorption was seen in the case of long fibres composite compared to the short one (Thakur and Singha, 2010).

Daramola et al.; (2017) studied the effect of variation in weight percentages of banana fibre on the water absorption properties of banana/polyester composites and a linear relationship of water uptake with fibres concentrations was observed. Water absorption of hemp fibre/unsaturated polyester was found to increase with an increase in weight percentages of hemp fibres (Rouison et al.; 2005). According to Singh and Tiwari (2018), the luffa cylindrica fibre was used as a reinforcing to fabricate the composite with epoxy resin. It was found that the values of moisture uptake increase with an increase in fibres loading. Further, a higher moisture uptake in the saline environment over distilled water was obtained. Santos et al.; (2020) studied the effect of ageing of autoclaved on water absorption, porosity and flexural behaviour of epoxy/flax composites, and concluded that water absorption levels increased with the ageing time.

The effect of charcoal particles incorporation on the mechanical and water absorption behaviour of sisal composites was studied, and it was displayed that mechanical and water resistant performance was significantly improved due to charcoal particles loading up to 4 wt. % and then decreased (Binayaka and Gupta, 2019). The aim of this study was to examine the influence of reinforcement particle size, (XPS), reinforcement weight (XRW) and activated carbon type (XCSAC) on the thickness swelling and absorption behaviour of the chemically activated carbon composites as friction lining materials.

## **2. MATERIALS AND METHODS**

### **2.1. Materials and Equipment**

Materials used in the production of the activated carbon includes palm kernel shell (PKS), coconut shell (CS), calcium chloride, distilled water, volumetric flask, measuring scale, heat source, aluminium pot with lid, ramming mass (castable), small ceramic pot with laboratory mortar, ball milling machine and a timer. The basic material types used in the production of the composites include: activated carbon from palm kernel shells and coconut shells to produce activated carbon type; 0%CSAC:100%PKSAC, 50%CSAC:50%PKSAC and 100%CSAC:0%PKSAC, epoxy resin (Epochem 105), epoxy hardner (Epochem 205) and the mould made from beewax.

## 2.2 Methods

### 2.2.1 Production of Activated Carbon

The methods and processes involved in the production of the activated carbons from palm kernel shells (PKS) and coconut shells (CS) shown in Figure 1 had been reported by Akuwueke et.al., (2022).

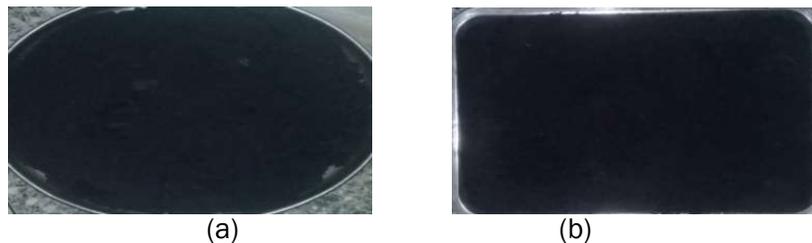


Figure 1: Activated Carbon produced from (a) palm kernel shells (PKSAC) and (b) coconut shells (CSAC).

### 2.2.2 Production of epoxy/activated carbon composites

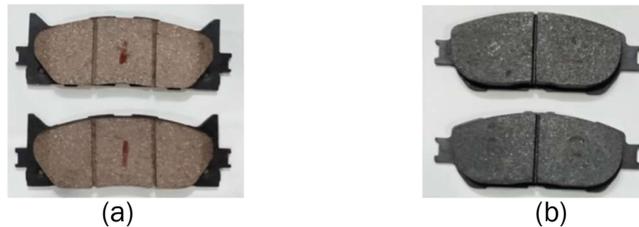
The methods and processes involved in the production of the chemically activated carbons composites from palm kernel shells (PKS) and coconut shells (CS) shown in Figure 2 had been reported by Akuwueke et.al., (2022).



Figure 2: Epoxy/activated carbon composite samples.

### 2.2.3 The control samples

Two (2) different commercial brake pads bought from a local automobile market in Port Harcourt, Nigeria were used as control samples. Figure 3 shows the brake pads designated as CB1 (TOYOTA with OEM No/Model: 04465-33450) and CB2 (SUPERFIT with Model No D2223) manufactured in Japan and China respectively. The physical properties of the commercial brake pads samples were evaluated and compared with the developed epoxy/activated carbon composites of three different particle sizes and weight percentages.



**Figure 3: Control samples: (a) Commercial Brake pads 1 (CB1) (manufactured by TOYOTA with OEM No/Model: 04465-33450), (b) Commercial Brake pads 2 (CB2) (manufactured by SUPERFIT with Model No D2223).**

### 2.2.4 Physical properties test of epoxy/activated carbon composites

In this study, the physical properties of the epoxy/activated carbon composites (0%CSAC:100%PKSAC), (100%CSAC:0%PKSAC) and (50%CSAC:50%PKSAC) evaluated were thickness swelling (cold-water, hot-water and oil), water absorption test (cold-water, hot-water), and oil absorption tests.

#### 2.2.4.1 Thickness swelling test

To study the thickness swelling behaviour of the various compositions of the epoxy/ activated carbon composites according to their particle sizes and weight percentages after a six (6) hour cold distilled water, hot distilled water, and oil immersion. The samples dimension of 10 mm x 10 mm of various epoxy/activated carbon compositions were prepared according to ASTM D570 for percentage cold water, oil and hot water thickness swelling examinations (Figure 4 and Figure 5).

##### (i) Cold-water thickness swelling

All samples including the control samples (commercial brake pad 1 (CB1) and commercial brake pad 2 (CB2)) were cut to length and width of 10 mm x 10 mm in dimensions. The samples were cleaned to remove dirt and initial thickness (T<sub>1</sub>) readings taken. The samples were immersed separately in distilled water at room temperature (25 °C) for six (6) hours and then removed, cleansed and the final thickness (T<sub>2</sub>) at six (6) hours taken. All measurements were taken using a digital venire caliper. The cold-water thickness swelling was calculated using Equation 1.

$$\% T.S(CW) = \frac{T_2 - T_1}{T_1} \times 100 \quad (1)$$

##### (ii) Hot water thickness swelling

All samples including the control samples (commercial brake pad 1 (CB1) and commercial brake pad 2 (CB2)) were cut to length and width of 10 mm x 10 mm in dimensions. The samples were cleaned to remove dirt and initial thickness (T<sub>1</sub>) readings taken. The samples were immersed separately in hot distilled water (75 °C) for six (6) hours and then removed, cleansed and the final thickness (T<sub>2</sub>) at six (6) hours taken. All measurements were taken using a digital venire caliper.

The hot-water thickness swelling was calculated using Equation 2.

$$\% T.S(HW) = \frac{T_2 - T_1}{T_1} \times 100 \quad (2)$$

**(iii) Oil thickness swelling**

Similarly, all samples including the control samples (commercial brake pad 1 (CB1) and commercial brake pad 2 (CB2)) were cut to length and width of 10 mm x 10 mm in dimensions. The samples were cleaned to remove dirt and initial thickness (T<sub>1</sub>) readings taken. The samples were immersed separately in a commercial motor oil (Mobil 1 OW-40) at room temperature (25 °C) for six (6) hours and then removed, cleansed and the final thickness (T<sub>2</sub>) at six (6) hours taken.

All measurements were taken using a digital venire caliper. The oil thickness swelling was calculated using Equation 3.

$$\% T.S(Oil) = \frac{T_2 - T_1}{T_1} \times 100 \quad (3)$$

Where, T<sub>1</sub> is the initial thickness before immersion (mm), T<sub>2</sub> is the final thickness after 6 hours immersion (mm), T<sub>SCW</sub> is the thickness swelling (cold water) (%), T<sub>SHW</sub> is the thickness swelling (hot water) (%), T<sub>SOIL</sub> is the thickness swelling (oil) (%).

**2.2.4.2 Water absorption test by thermogravimetric analysis**

To study the water absorption behaviour, samples of the various epoxy/activated carbon composites used for cold and hot water thickness swell for six (6) hours were used for cold (25°C) and hot (75°C) water absorption test using thermogravimetric analysis (TGA) following ASTM D570 standard.

**(i) Cold-water absorption test**

All samples including the control samples (commercial brake pad 1 (CB1) and commercial brake pad 2 (CB2)) were prepared to a dimension 10 mm x 10 mm for cold water thickness swelling test (6 hours immersion in distilled water) were used to determine the cold water absorption (%) behavior of the samples using thermogravimetric analysis. After a 6 hour sample immersion in cold water (25°C), known weights of all samples were taken and subjected to thermogravimetric measurements performed on a TA Analyser (TGA 2950, TA Instruments, USA) under the heating rate 20 °C min<sup>-1</sup>, a dynamic atmosphere of nitrogen (50 mL min<sup>-1</sup>) in the temperature range of 30 –500 °C. The processes were repeated for all samples. *The percentage cold water absorption for all samples was determined from the respective thermogravimetry (TG) thermograms of the individual samples as percentage weight loss at end of first step decomposition at temperature, T, approximately 110.33°C as specified by ASTM D570 standard depending on the thermogram shown by each sample.*

However, in other to validate the result from the TGA thermogram, the percentage cold water absorption was theoretically calculated for 0%CSAC:100%PKSAC, 100%CSAC:0%PKSAC and 50%CSAC:50%PKSAC samples using data from the TGA thermogram using Equation 4.

$$\% \text{ Cold water absorption} = \frac{W_1 - W_2}{W_1} \times 100 \quad (4)$$

Where, W1 (mg) is the initial TGA sample weight, W2 (mg) is the sample remaining weight at 1<sup>st</sup> step decomposition (at  $\approx 110.33^\circ\text{C}$ ). Note: W2 is calculated as (Initial TGA sample weight (W1) – Weight loss at 1<sup>st</sup> step decomposition). Alternatively, the percentage cold water (CW) absorption can easily be calculated using the short form of Equation 3.13 as expressed in Equation 5.

$$\% \text{ CW absorption} = \frac{\text{Weight loss at 1st step decomposition}}{\text{Initial TG sample weight}} \times 100 \quad (5)$$

**(ii) Hot water absorption test**

All samples including the control samples (commercial brake pad 1 (CB1) and commercial brake pad 2 (CB2)) were prepared to a dimension 10 mm x 10 mm for hot water thickness swelling test (6 hours immersion in distilled water) were used to determine the hot water absorption (%) behavior by the samples using thermogravimetric analysis. After a 6 hour sample immersion in hot water (75°C), known weights of all samples were taken and subjected to thermogravimetric measurements performed on a TA Analyser (TGA 2950, TA Instruments, USA) under the heating rate 20 °C min<sup>-1</sup>, a dynamic atmosphere of nitrogen (50 mL min<sup>-1</sup>) in the temperature range of 30-500 °C.

The processes were repeated for all samples. *The percentage hot water absorption for all samples was determined from the respective thermogravimetry (TG) thermograms of the individual samples as percentage weight loss at end of first step decomposition at temperature, T, approximately 110.33°C as specified by ASTM D570 standard depending on the thermogram shown by each sample.* However, in order to validate the result from the TGA thermogram, the percentage hot water absorption was theoretically calculated for 0%CSAC:100%PKSAC, 100%CSAC:0%PKSAC and 50%CSAC:50%PKSAC samples using data from the TGA thermogram using Equation 6.

$$\% \text{ Hot water absorption} = \frac{W_1 - W_2}{W_1} \times 100 \quad (6)$$

Where, W1 (mg) is the initial TGA sample weight, W2 (mg) is the sample remaining weight at 1<sup>st</sup> step decomposition (at  $\approx 110.33^\circ\text{C}$ ). Note: W2 is calculated as (Initial TGA sample weight (W1) – weight loss at 1<sup>st</sup> step decomposition). Alternatively, the percentage hot water (HW) absorption can easily be calculated using the short form of equation 3.15 as expressed in Equation 7.

$$\% \text{ Hot water absorption} = \frac{\text{Weight loss at 1st step decomposition}}{\text{Initial TG sample weight}} \times 100 \quad (7)$$

**(iii) Oil absorption test by thermogravimetric analysis**

All samples including the control samples (commercial brake pad 1 (CB1) and commercial brake pad 2 (CB2)) were prepared to a dimension of 10 mm x 10 mm used for oil thickness swelling test (6hours immersion in water) were used to determine the oil absorption (%) behavior by the samples using thermogravimetric analysis. After a 6 hour sample immersion in a commercial motor oil (Mobil 1 OW-40) at 25°C, known weights of all samples were taken and subjected to thermogravimetric measurements performed on a TA Analyser (TGA 2950, TA Instruments, USA) under the heating rate 20 °C min<sup>-1</sup>, a dynamic atmosphere of nitrogen (50 mL min<sup>-1</sup>) in the temperature range of 30-1000 °C. To determine the percentage oil absorbed, 4.7240mg of commercial motor oil (Mobil 1 OW-40) was subjected to TGA and used to study its optimum degradation temperature.

The thermogram of the commercial motor oil was then overlaid on the thermogram of each sample to identify the point of intersection which marked the endset temperature of 2<sup>nd</sup> step decomposition. The percentage weight loss at this point is referred to as the % oil absorbed by the samples. However, in other to validate the result from the TGA thermogram, the percentage oil absorption was theoretically calculated for 0%CSAC:100%PKSAC, 100%CSAC:0%PKSAC and 50%CSAC:50% PKSAC samples using data from the TGA thermogram using Equation 8. It should be noted that the weight loss at 2<sup>nd</sup> step decomposition was taken from the point of intersection of the two thermograms (the commercial motor oil and the samples). The processes were repeated for all epoxy/activated carbon composites samples respectively.

$$\% \text{ Oil Absorption} = \frac{\text{Weight loss at 2nd step decomposition}}{\text{Initial TG sample weight}} \times 100 \quad (8)$$



**Figure 4: Samples undergoing 6hours cold water, and oil thickness swelling tests and used for cold water and oil absorption test by TGA.**