Effect of polyester and filler on water absorption behaviour, density and porosity of kenaf (*Hibiscus canabinus)* fibre reinforced brake pads

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Abstract

Asbestos fibre has been used as traditional base material in brake pads manufacture, but due to its health hazard, non-renewability, cost, non-biodegradability and difficulty in processing, its replacement has been sought. For the purpose of improving their properties, the fibres were retted, bleached and modified using mercerization, acetylation and semi-carbonisation methods to obtain three modified fibres in addition to the crude fibre. The four fibre types (crude, mercerised, acetylated and semi-carbonised) were evaluated for moisture absorption, density and porosity. Brake pads were produced from each of the four fibre types (each 15 % by weight) using a randomised factorial experimental design (1x 3 x 3), where the concentrations of the polyester (15 %, 20 % and 25 % by weight) and filler(70 %, 65 % and 60 % by weight) were used. These materials or ingredients were mixed at 600 rpm for 5 minutes and then hot pressed at 175 ^{0}C under a moulding pressure of 32.5 MPa, which was held for 1 minute. A total of 36 samples of kenaf brake pads were produced and evaluated. Comparison among treatment means using the least significance difference (LSD) showed that at 20 % of polyester, water absorption in crude, mercerized, acetylated and semi-carbonized samples was not significantly different when 65 % weight of filler changed to 60 % by weight but the water absorption was significantly different when polyester changed from 20 % to 25 %. Also, the performance evaluation results indicated that kenaf brake pads have high percentage of water absorption capacity, porosity and density with an average value of 0.067 g, 38 % and 2.984 g/cm^3 respectively than all the samples investigated.

Keywords: water absorption; density, porosity, kenaf brake pads

1. INTRODUCTION

Brake pads for an automotive brake system are friction complex composites because they contain numerous ingredients that are diverse in physical, mechanical and chemical properties. These brake pads or friction composites comprise many disparate ingredients such as binders, fibres and fillers. Asbestos fibres which occurred naturally as mineral, have been used as traditional fibrous ingredient to reinforce the constituents in the friction material or to provide mechanical strength and also to inhibit catastrophic failure of the structure [4]. However, due to its non-biodegradability, non-renewability, difficulty in processing, high cost, high density and most especially its risk of causing cancer, asbestos – based friction materials were banned and the search for safer and cheaper alternative [5]. Several treatment techniques intended to improve the natural fibre-matrix adhesion in brake pad composites were reported in literature.

Some of the techniques considered in this paper are mercerisation, acetylation and semi-carbonisation of kenaf fibre as compared to using the crude. The objectives of this study is to compare the effects of these fibre treatment techniques on the physical properties (namely; density, water absorption and porosity) of Kenaf Brake Pad samples.

2. MATERIALS AND METHODS

2.1 Materials for kenaf brake pad production

The materials used in this study include binder or polyester, fillers (rubber crumbs, fine grain iron filings, graphites, barium sulphate), rolls of decorticated kenaf fibre, sodium hydroxide, ammonium oxalate, hydrochloric acid, hydrogen peroxide, tools and equipment include; furnace, oven, drier, hardness tester, compression moulding rig, friction test rig, impact tested, digitizer, camera fitted light microscope, microtome, stop watch, weighing balance and intsrone machine.

2.1.1 Binder

The Unsaturated Polyester Binder that was used was obtained from the Northern Scientific Laboratory Located here in Yola.Ten litres of this binder were purchased for the purpose of this research.

2.1.2 Fillers

2.1.2.1 Rubber crumbs

These are the other black particles that are seen in commercial brake pads. They are produced from scrap rubber from used tires of 50 μm and are relatively inexpensive. The low specific weight of rubber was an advantage in the final product. In this work 500 g of rubber crumbs was ground to 0.5 mm particle size and was used.

2.1.2.2 Fine grained metal iron

Fine grain metal iron or iron powder was used in this research as an abrasive. About 1.5 kg of fine-grain metal iron was sieved which was obtained as waste from Machine Shops in Jimeta, Yola. This was sieved into fine grain of 2 μm size

2.1.2.3 Graphite

Graphite occurs naturally in places like Gayama village in Taraba State, Nigeria. From this village, which is about 289 km from the state capital, Jalingo, 1.2 kg of graphite stone was mined or dug from the deposit for the purpose of this study. Thereafter, it was milled and the product, which is a fine powder, was sieved through a 200 mesh (75 um).

2.1.2.4 Barium sulphate

Like graphite, Barium Sulphate also called barite was obtained locally from the mines at Lau village of Taraba State. This filler was ground into powder by means of a plate mill; thereafter it was sieved through a 345 mesh (5 μm). A Total of 1.5 kg of this material was used in this study.

2.1.3 Kenaf fibre

Ten rolls of decorticated Kenaf fibre were used in this study, it was procured from Jimeta Market, Yola. These fibres have been decorticated, dried and packed in form of rolls. The material represents a matured fibre and the best of its type available in the locality. These fibres were purified and transformed into better fibre through some innovative chemical treatments discussed previously. Using a plate mill, I kg each of the four fibre Treatments was ground and sieved into an average length of 2 - 4 mm and an average diameter of 12 цm. According to Jang *et al* [3]; Marthur *et al* [4], 10 % - 20% fibre reinforcement was used in brake padding (Pre

trials to determine the fibre volume fraction were also carried out). Thereafter, 15% by weight of kenaf fibre was considered adequate for reinforcement in this study.

2.2 Methods

2.2.1 Acetylation of kenaf fibre.

To 1 g of mercerized fibre, 5.5 cm^3 of acetic acid; 2.5 cm^3 acetic anhydride 0.65 cm^3 conc. H_2SO_4 were added. The content was warmed gently to 45 ^{0}C for 1 h. 0.65 cm^3 of H_2SO_4 was again added and stirred using a magnetic stirrer at 43-45 ^{0}C for another 1 h. The dark colored solution was poured into 50 cm^3 of water and allowed to stand for 48 h.

The precipitate was dried and the yield determined. The water absorptions characteristics was conducted on the acetylated fibre samples.

2.2.2 Surface modification by mercerization.

About five batches of 1.0 g each of 10% treated ammonium oxalate and 10% hydrogen peroxide and bleached samples of cellulose fibre were immersed in beakers containing 10%, 15% 18%, 22% and 25% sodium hydroxide concentrations. The temperatures in these beakers were kept at 5^0C by means of ice blocks for a period of 45 mins. Thereafter mercerized samples were washed very thoroughly in 5% solution of acetic acid. The water absorptions characteristics was conducted on the mercerised fibre samples

2.2.3 Surface modification by semi – carbonization or heat treamtent

Crude fibre having been retted and bleached was semi-carbonised using a laboratory oven as shown on Figure 5a, b, and c. An equal weight of 0.300 g of the above kenaf fibre sample was weighed. And using a heating rate of 2.21 ^{0}C/min, each batch was heated to temperatures of 100^0C 150^0C, 200^0C, 250^0c and 300^0C respectively. It was then soaked for 5 mins at each temperature threshold [6].

2.2.4 Production of kenaf brake pads

2.2.5 Experimental design

The experiments were of the randomised 1 x 3 x 3 factorial design for four different types of kenaf fibre at 15 % weight each.. Table 1 shows the outline of the experimental design for the four types of kenaf fibers with three levels of polyester binder and filler.

Main treatment: $\mathbf{T_1}$ (Untreated fibre); $\mathbf{T_1}$ (Mercerized fibre); $\mathbf{T_1}$ (Acetylated fibre); T_1 (Semi – Carbonized fibre) at 15 % weight each. **Sub-treatmen**ts: $\mathbf{S_1}$ (15 % polyester (Binder)); $\mathbf{S_2}$ (20% polyester); $\mathbf{S_3}$(25% polyester), then filler at $\mathbf{R_1}$ (60 %), $\mathbf{R_2}$ (65 %), and $\mathbf{R_3}$ (70 %) by weght

Treatment combination = for the four types of fiber treatments = 1 Level of fibre × three levels of binder (Polyester) x three levels of Filler = 1 x 3 x 3 = 9 (T1S1R1, T1S1R2, T1S1R3, T1S2R1, T1S2R2, T1S2R3, T1S3R1, T1S3R2 and T1S3R3)

Total experiments conducted = 9 treatments x 4 Fibre Types × 5 replications = 108 experiments.

T, S and **R** were mere letters chosen to represent fibre treatment, binder (polyester) and Filler, respectively The numbers 1, 2, 3 were used to denote the levels of the factors. This exercise was done this way for convenience only.

Table 1 Experimental design for kenaf brake pad samples

		Treatment			
S/N	Sample code	Kenaf Fibre Type (1 level)	Polyester (3 levels)	Filler (3 levels)	Replications
1.	T1S1R1	1	1	1	5
2.	T1S1R2	1	1	2	5
3.	T1S1R3	1	1	3	5
4.	T1S2R1	1	2	1	5
5.	T1S2R2	1	2	2	5
6.	T1S2R3	1	2	3	5
7.	T1S3R1	1	3	1	5
8.	T1S3R2	1	3	2	5
9.	T1S3R3	1	3	3	5
10	Control (commercial sample)	NA	NA	NA	NA

NA = Not Available

1 Kg of rubber crumbs, 1 Kg of iron filings, 1 Kg of barium sulphate and 1 Kg graphite to give 4 Kg of Fillers.

Total Raw Materials used in this study = 100g x 1 treatment x 5 replications = 500g

Where the treatment combination does not give exactly 500g, calcium carbonate was used as a space filler to adjust the remaining percentage during replications as was done by Marthur *et al* [4]. Then the materials used in this study is approximately, 500g x 9 Treatments x 4 Fibre Types = 18,000g

2.2.6 Mixing of raw materials

In order to assist fiber dispersion, kenaf fibres were fiberized in a double-bladed kitchen blender for 8 mins. The fiberized kenaf fibers were dried at 60^0C for 24 h and stored in a desiccator, prior to compounding. A total of 9 samples with five replicates for each formulation of the four fibre treatments were mixed batch by batch.

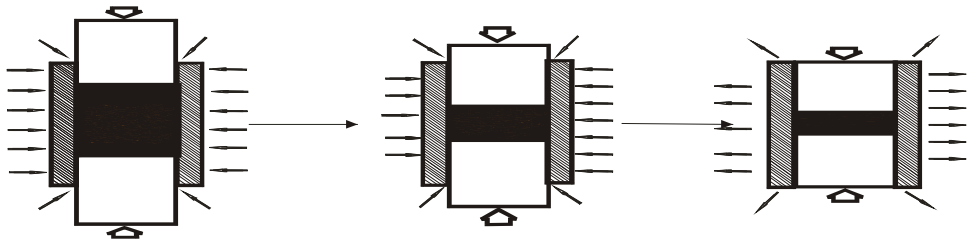
The mixing was done at a temperature of 38^0C, mixing time of 5 mins, and rotor speed of 600 rpm [3]. The first treatment mixing was carried out in this manner: the binder resin (unsaturated polyester (15 % of the Total weight) was first added inside the mixing chamber then another 15 % by weight of the fibre type was added, and finally 70 % by weight of the last component (filler) was also added. The mixer containing 100 g of kenaf fibre, polyester and filler was started and run for 5 mins until the three components were mixed. Thereafter the mixed material was removed for the next process.

2.2.7 Compression moulding

The mixed material was molded on a Hydraulic press into pads with dimensions of 6 cm x 4 cm x 2 cm. The mold was first preheated to raise the temperature to175 ^{0}C. As shown in Figure 1 and Figure 2, the mixed material was then placed in the mould and heated for 1 min under a constant pressure of 32.5 MPa; thereafter the moulded material was removed and cooled at room temperature for 8 mins as outlined on Figure 2a-d.

Figure 1 Compression moulding process using the CMR Equipment

The pressed samples were then cured in an Oven for 8h, therafter it was finished and prepared for performance properties [2]



(a) Charging and heating b) Heating and moulding (c) Cooling

(d) Demoulding

Figure.2 a, b, c and d. Compression Molding process for kenaf brake pad

Figure 3 Kenaf brake pad samples

2.2.2 Test for Density, water absorption and Porosity

2.2.2.1 Testing for Density of kenaf brake pads

In this method, each sample of the kenaf brake pads measuring 2.4 cm x 2.2 cm x 1cm were prepare [7]. The specimens were weighed on a top pan balance and their weights ($W_{1)}$ recorded, while theie volumes were calculated from their dimensions. The results generated using the following formular were statistically analyzed.

Density = Mass/Volume (g/cm^3) 1

2.2.2.2 Apparent porosity

The apparent porosity of kenaf brake pads was determined and the results obtained were analyzed. The specimen measuring 2.4 x 2.2 cm x 1 cm was cut from a kenaf brake pad sample and was thoroughly cleaned. The specimen was dried in an Oven at 110 ^{0}C to a constant weight with an accuracy of 0.1 g. The dried specimen was immersed in distilled water such that the specimen does not touch the bottom or sides of the container. It was boiled for two hours while still immersed in water and afterwards it was cooled to room temperature and its weight noted. The specimen was removed from water and excess water was wiped off from its surface by lightly blotting with a wet towel and weighed in air (W). The apparent porosity (P) is then calculated by equation 2.

$$p = \frac{v1}{v2} x100 \quad \text{..... 2}$$

$$where$$

$$v1 = actual\ volume\ of\ open\ pores\ of\ the\ specimen(w_a|D_a)$$

$$v2 = external\ volume\ of\ the\ specimen(w_s|D_a)$$

$$since\ density = \frac{mass}{volume}$$

$$where$$

$$w_a = weight\ of\ the\ specimen\ in\ air(g)$$

$$w_s = weight\ of\ soaked\ specimen(g)$$

$$D_s = density\ of\ specimen(g|cm^3)$$

$$D_a = density\ of\ specimen(g|cm^3)$$

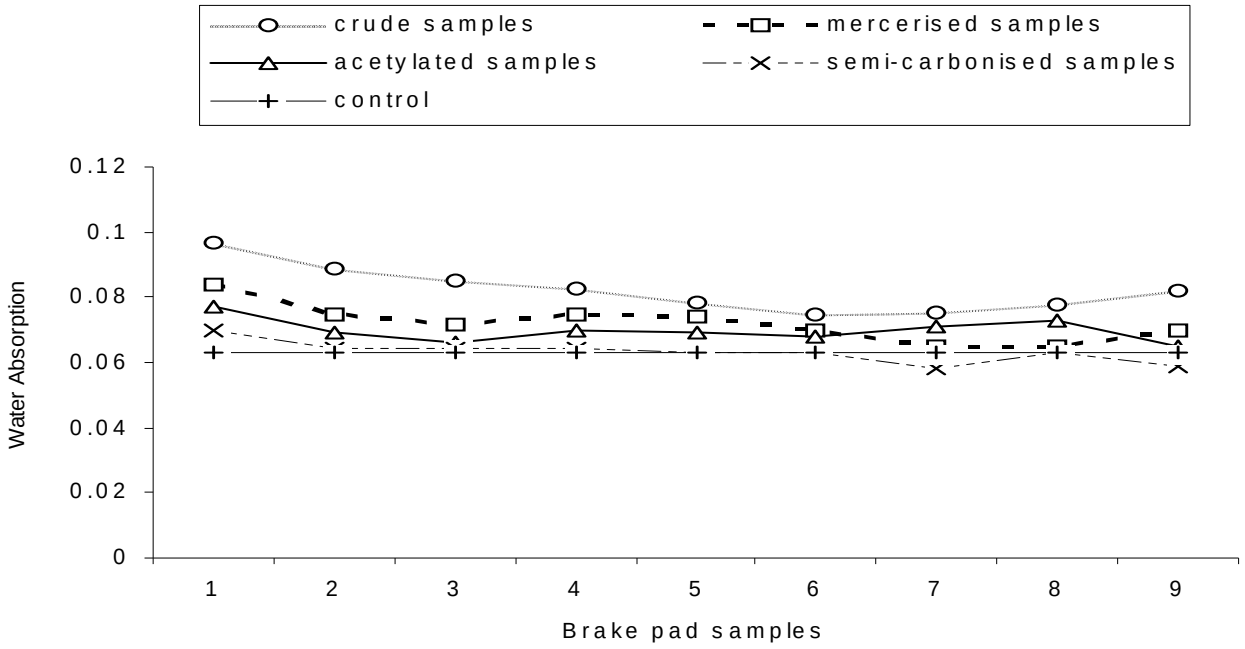
2.2.2.3 Water absorption of kenaf brake pads

Water absorption characteristics of Brake Pads were determined. Rectangular specimens were cut from each sample with dimensions of 15.4 mm x 36.2 mm. The samples were dried in an oven at 50 ^{0}C for 24 h, cooled in a desiccator, and immediately weighed to the nearest 0.001 g. In order to measure the water absorption of the Brake Pads, all samples were immersed in water for about 24 h at room temperature as described in ASTM procedure D570-99 [1]. Excess water on the surface of the samples was removed before weighing.

3. RESULTS AND DISCUSSIONS

3.1 Water absorption of kenaf brake pad samples

From figure 4, Kenaf brake pads produced from crude fibre with sample code T1S3R1 absorbs as much water as 0.0670g more than semi-carbonised sample with code T1S1R1 that absorbed the least of water (0.0607g) after the same period of test, suggesting that, changes in surface chemistry resulting from semi-carbonization have reduced the affinity of fibers to moisture. Strong intermolecular fiber-matrix bonding decreased the rate of moisture absorption. For automotive brake pads, the least water a brake pad material absorbs the better.



(Serial number as in Table 1)

Figure 4 Water Absorption of Kenaf Brake pad samples

3.1.1 Effect of polyester and filler on water absorption behavior of kenaf brake pads

Figure 5 shows the Effect of the Interaction of Polyester and Filler on water absorption of kenaf brake pads, the levels of polyester in combination with the levels of filler gave a significant decrease in the water absorption of semi-carbonized samples as can be seen on Figure 5. Comparison among treatment means using the least significance difference (LSD) showed that at 20 % of polyester, water absorption in crude, mercerized, acetylated and semi-carbonized samples was not significantly

different when 65 % weight of filler changed to 60 % by weight but the water absorption was significantly different when polyester changed from 20 % to 25 %.

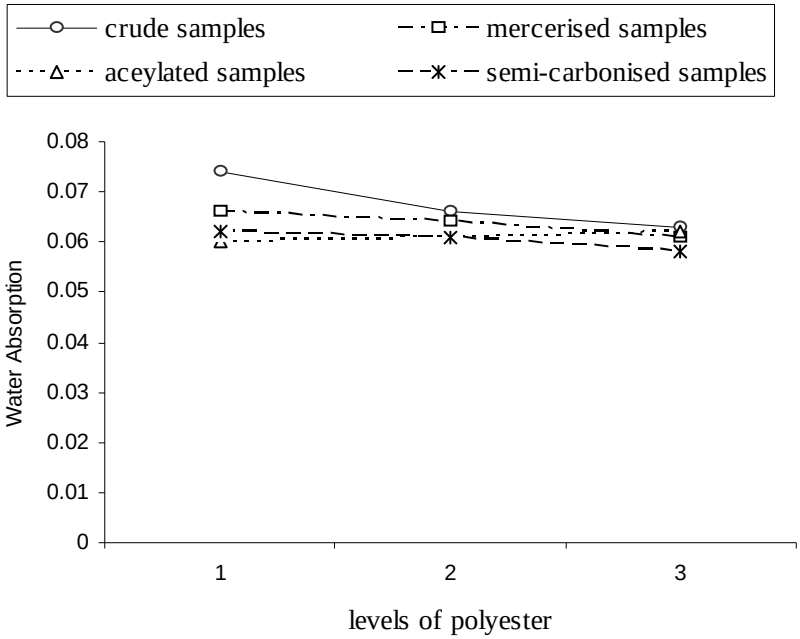
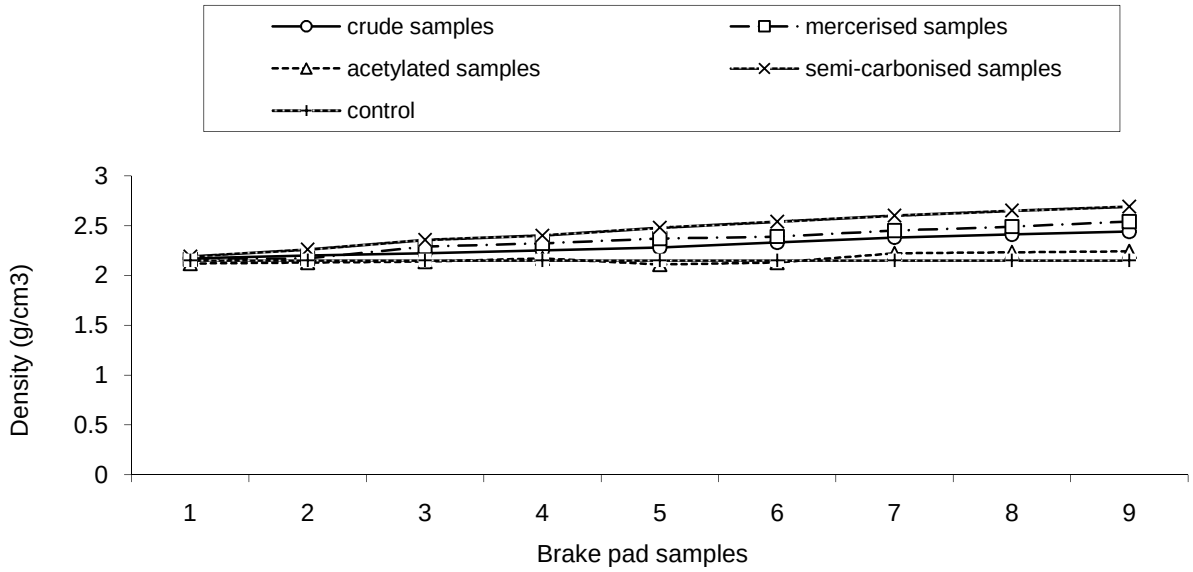


Figure 5 Effect of Polyester on the Water absorption behaviour of kenaf brake pads

From the graph, the main effects of polyester, there appears to be a systematic drop in the water absorption of all the brake pad samples, the drop was shown to be highly significant from the results . Also, the response showed a significant linear effect of the polyester on all the samples investigated. The main effects of filler , shows an apparent increase in water absorption of mercerized, acetylated and semi-carbonised samples as the dosage increased from 65 % to 70 % a drop was noticed thereafter. The drop was highly significant (P = 0.01). This is not trend with the crude sample which drops steadily in terms of water absorption as the dosage increases. However, further increase in the percentage by weight of filler resulted in a non-significant increase in the water absorption of the semi-carbonized and acetylated samples.

3.2 Densities of kenaf brake pad samples

From Figure 6, the densities of all the kenaf brake pad samples seem to have close density values having a steady increase with increase in combination of the levels of both polyester and filler. With axetylated sample having the lowest average of 2.03 g/cm^3 to as high as 2.42 g/cm^3 in semi-carbonized (T1S2R3) brake pad samples.



(Serial number as in Table 1)

Figure 6 Effect of the Interaction of Polyester and Filler on the Densities (g/cm^3) of Kenaf Brake pads

The densities of semi-carbonized brake pad samples are so because of the improved ability of the fibres to absorb much polyester resin together with filler. Again from the Figure 10, a particular sample with code T1S3R3 (acetylated) having a value of 2.04 g/cm3, this value fall within the range reported for brake pads which is between 1.8 g/cm^3 to 2.5 g/cm^3 (Nicholson, 1995)

3.2.1 Effect of polyester and filler on the densities of kenaf brake pads

Figure 7 shows the main effects of polyester, again there appears to be an increase in the density of crude, mercerized, acetylated and semi-carbonized samples as the dose was increased from 20 to 30 %, the increase was shown to be highly non significant from the results .

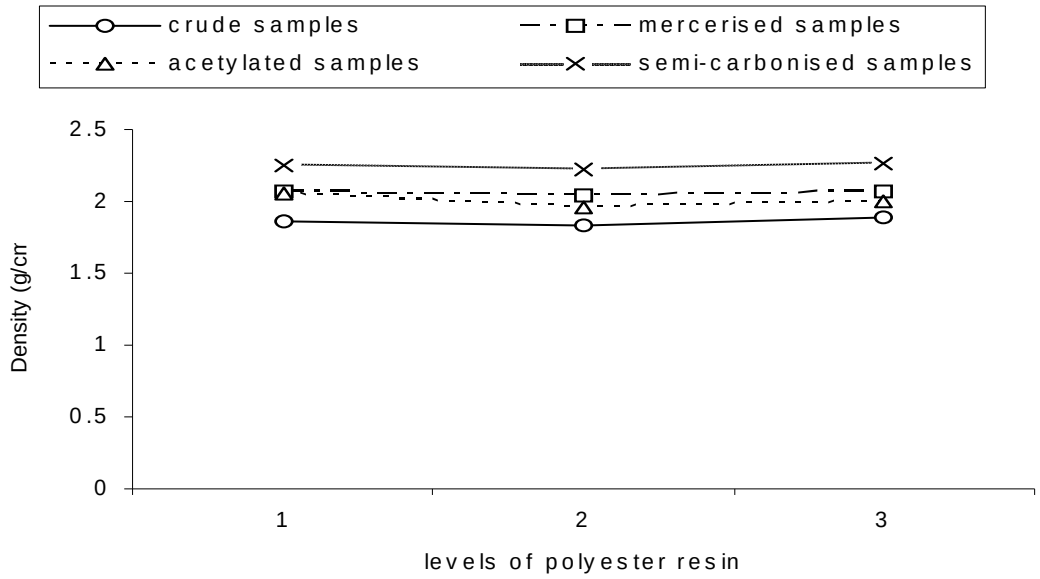


Figure 7 Effect of Polyester on the Water absorption behaviour of kenaf brake pads

The main effects of filler , shows an increase in the density of semi-carbonized samples as the dosage increased from 65 % to 70 % a drop was noticed in acetylated and crude samples. The drop was non significant. However, further increase in the percentage by weight of filler resulted in a non significant increase in the density of the crude, mercerized and acetylated samples

3.3 Porosities of kenaf brake pad samples

From figure 8, the range of porosity of the brake pads indicates that samples produced from crude fibre are more porous with an average value of 38.34%. However, the porosity is much lower for the semi-carbonized samples with the least being 13.03% (T1S1R1). Why the crude samples are more porous as can be seen on Figure 7 could suggest that polyester resin did not flow well during hot molding. The Porosity range reported for brake pads is between 7 –21% [4]. It is important to note here that, the more porous a brake pad is, the more noise it will generate during brake applications.

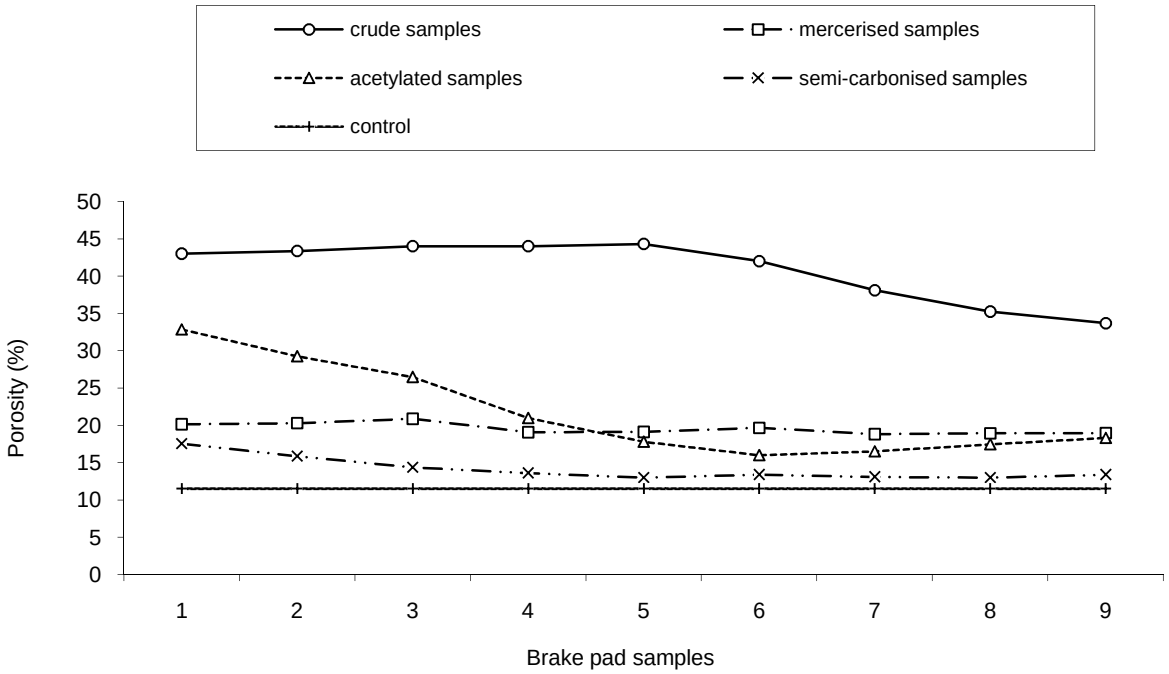


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Figure 8 Effect of the Interaction of Polyester and Filler on Porosity of kenaf brake pads

3.3.1 Effect of polyester and filler on porosities of kenaf brake pads

Figure 9 shows the main effects of polyester, again there appears to be a general drop in the porosity of mercerized, acetylated and semi-carbonized samples, the drop was shown to be highly significant from the results.

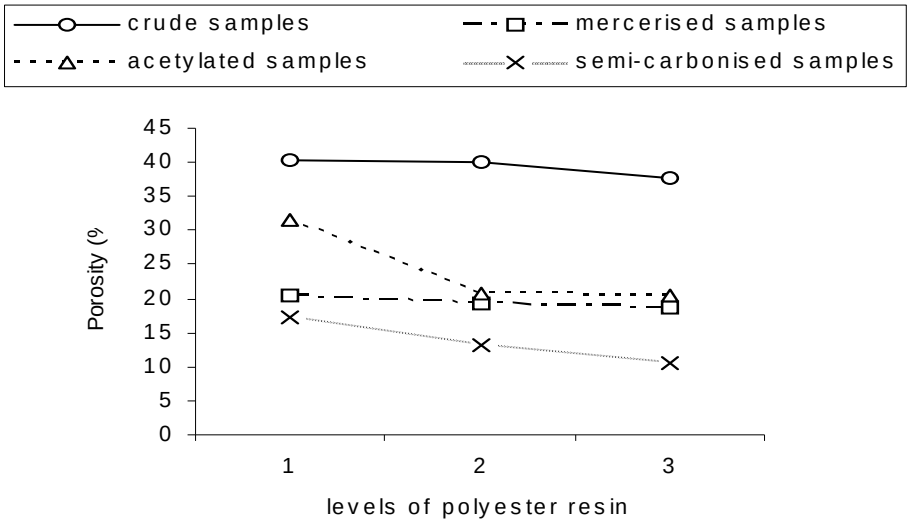

Figure 9 Effect of Polyester on the Water absorption behaviour of kenaf brake pads

The main effects of filler , shows an increase in the porosity of mercerized samples as the dosage increased from 65 % to 70 % a drop was noticed in acetylated and semi-carbonized samples. The drop was highly significant (P = 0.01). However, further increase in the percentage by weight of filler resulted in a significant increase in the porosity of the crude samples.
From the interactions graph shown on Figure 9, crude, mercerized, acetylated and semi-carbonized samples are more porous at lower levels of polyester and filler (15 %, 20 % and 60 %, 65% respectively), the reason being that at lower quantity of resin there appear to be insufficiency of the binder to bind all the ingredients in the formular, while at higher quantity, the binder appears to be more thereby appealing for more air bubbles during processing, but 20 % of polyester appears to give optimum porosity in all the treatments.

4. CONCLUSION

From the results obtained in this study, it can be concluded that the brake pad samples made from crude fibres were more porous with average porosity value of 38 % than all the samples investigated, while semi-carbonised were the least porous with an average of 13 %. The more porous a brake material is, the more noise it produces. The crude kenaf fibre based brake pads absorbed more quantity of water than all the samples after soaking in water for 24 h.

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