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Optimization of NaOH Treatment Conditions of Baobab Pod Fibres Using Box-Behnken Method

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Optimization of NaOH Treatment Conditions of Baobab Pod Fibres Using Box-Behnken Method

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Abstract. Natural fibres are suitable materials that represent an opportunity to partially minimize on the environmental impacts by integrating them in place of synthetic fibres because of their low cost, biodegradability, low density and renewability. This work is aimed at studying the effect of chemical treatment conditions on the Mechanical properties of baobab fibres. The tensile strength and young modulus of baobab fibres were investigated. Design expert V10 software was used to analyze the experimental data. Results were processed using analysis of variance (ANOVA) technique, namely the Box-Behnken method. The effect of NaOH treatment on functional group, surface morphology and crystallinity of fibres were also investigated. The results indicated that within the limits of treatment conditions used in this study, the proposed models adequately predicted the fibre Tensile Strength and Young Modulus.

Key Words: Baobab fibres, mechanical properties, NaOH treatment, ANOVA.

1. Introduction

Natural fibre`s availability, renewability, low density, and price as well as satisfactory mechanical properties make them an attractive ecological alternative to man-made fibres, they are more environmentally friendly, and are used in transportation, military applications, building and construction industries, packaging, consumer products, etc. Natural fibres are composed mainly of cellulose, hemicellulose, lignin, wax, pectin, and other materials. Among these, cellulose, hemicellulose and lignin are the basic components of natural fibres accounting for mechanical properties [1]. Although composites reinforced with synthetic fibres possess superior mechanical properties, but they have some severe drawbacks that include high cost, poor recyclability and non-biodegradability [2]

Studies have been carried out using different natural fibres reinforcement for polymers, little attention had been given to the potential use of baobab pod fibre as reinforcement for polymer matrix. The Baobab, popularly known as “Kuka” among the dominant Hausa speaking populace of northern Nigeria is widely distributed and is found in all parts of the region. Baobab fibres, even though available locally, are presently underutilized in Nigeria due to lack of good marketing system and poor processing [3]. Baobab is a tree predominantly found in Africa, pods from the tree consist of fibres that contain high percentage of cellulose and covered with pectin and waxy substances which can hinder reaction with most materials, this render the fiber least attractive for reinforcement. The plant is a very massive tree with a very large trunk (up to 10 meter diameter) which can grow up to 25 m in height and may live for hundreds of years. Figure 1a shows the image of a baobab tree. Figure 1b shows images of baobab pod which is also called baobab fruit and Figure 1c shows baobab pod fibres which is mechanically extracted from the pod. The plant is widespread throughout the hot and drier regions of tropical Africa. Like other natural fibres, baobab fibres are lignocellulosic fibres with high percentage extension at break and low density compared to other natural fibres [4]

The reinforcing ability of natural fibres is usually enhanced by fibre treatment, which leads to improvement in the properties of fibre-reinforced resulting polymer composites [5].


(a) (b) (c)

Figure 1: (a) baobab tree (b) baobab pod (c) baobab pod fibres

Response Surface Methodology is an important branch of experimental design. It's a critical technology in developing new processes and optimizing their performance. The objectives of quality improvement, including reduction of variability and improved process and product performance, can often be accomplished directly using RSM. This study is concentrated mostly on building and optimizing the empirical models and consideration is given to the problems of experimental design. Chemical treatments are considered in modifying the fibre surface properties. It's aimed at improving the adhesion between the fibre surface and the polymer matrix may not only modify the fibre surface but also increase fibre strength.

2. Materials and Methods

2.1 Materials

The raw baobab pod fibres (BPFs) used in this study were collected from Zaria city market in Northern Nigeria, Sodium hydroxide (NaOH) pellets (98% purity, analytical grade), distilled water (100% purity) were supplied by a vendor and V10 design expert software.

2.2 Characterization of Baobab Pod Fibres (BPFs)

In order to fully comprehend the behavior of baobab pod fibres and to have a good understanding of its properties, different physical, thermal, chemical and mechanical tests were carried out. These include diameter measurement, density measurement, proximate analysis, elemental analysis, chemical analysis, tensile test, moisture adsorption and water absorption measurements, Fourier Transform Infra-red (FTIR) analysis, Scanning Electron Microscopy (SEM/EDS) and X-ray Diffraction (XRD) analysis.

2.3 *Experimental Design*

The effect of the alkaline treatment conditions on the mechanical properties of fibres is of paramount importance in natural fibre composites production. The effect of treatment parameters such as NaOH concentration, soaking time, and treatment temperature on fibre tensile strength and Young's Modulus was investigated. Design-Expert software was used to analyze the experimental data and to establish the design matrix. Numerical and graphical optimization techniques were used. In this work, the responses are single fibre Tensile Strength and Young Modulus. NaOH concentration (A), soaking time (B), and soaking temperature (℃) are selected (based on pilot experiments) to describe this system with reasonable ranges. The test was based on a three-factor three-level Box–Behnken design, as presented in Table 1. Response surface methodology (RSM) was applied to the experimental data using Design-Expert software V10. The statistical significance of the terms in the equations was examined using the sequential F-test, lack-of-fit test, and other adequacy measures using the same software to obtain the best fit.

2.4 *Chemical Treatments of BPFs*

Mercerization method of fibre treatment was adopted for the purpose of this study; analytical grade NaOH was used. Different concentrations of aqueous NaOH were prepared (2.5%, 5%, 7.5% and 10% by weight) by dissolving NaOH pellets in distilled water. Ground BPFs were then immersed into the solution and heated at 60℃ for 2 hours with a solution to fibre ratio of 40ml to 1g. After the immersion, the fibres were washed several times with laboratory water and finally with distilled water to get a neutral pH and to ensure no NaOH was left in the fibres. Subsequently, the fibres were sun dried for 24 h.

3. Results and Discussion

3.1 Physical behavior of raw BPFs

Average length of the fibre obtained to be 50.42mm indicates it's a short fibre when compared to that of other plant fibres like, Flax fibres length of range of up to 90cm, Ramie fibres with length range of up to 90cm and Sisal fibres with average length of up to 1m. BPFs has a very low density of 0.65g/cm^3 when compared to that of Ramie, Flax and Hemp fibres, all with densities of 1.5g/cm^3 [9]. However, it has a very high water absorption capacity of 226% which is due to the hydrophilic nature of the fibre. Water molecules attract the hydrophilic groups (-OH) of the cellulose molecules to form hydrogen bonds [6].

Table 1: Physical properties of BPFs

Fibre	Length (mm)	Diameter (mm)	Density (g/cm3)	Water Absorption (%)
BPFs	50.42	0.28	0.65	226

3.2 Proximate Analysis of raw BPFs

The proximate analysis of the BPFs reveals that, the fixed carbon which is 71.9%, is the major component and the remaining composition is volatile content and ash amounting to about 18.4%. Amount of ash content have direct correlation with the flexural properties fixed carbon and volatile matter, which indicates the amount of inorganic substituent in the carbon. High ash content is bad because adsorption capacity will be reduced. The low moisture content and low ash content of the BPFs makes it an excellent candidate for polymer reinforcement.

Table 2: Proximate analysis of BPFs

Fibre	Moisture (%)	Ash (%)	Volatile Content (%)	Fixed Carbon (%)
BPFs	9.4	8.3	9.1	71.9

3.3 Ultimate analysis of raw BPFs

Table 5 summarizes the results for the ultimate analysis, it can be seen that the sample consist of carbon greater than 60% and about 19% of hydrogen, 16% nitrogen and less than 1% of oxygen. The value of elemental carbon was consistent with that of the fixed carbon in the proximate analysis

Table 3: Ultimate analysis of BPFs

Fibre	Carbon (%)	Hydrogen (%)	Nitrogen (%)	Oxygen (%)
BPFs	63.6	19.6	16.3	0.51

3.4 FTIR for Raw BPFs

The peak at 891 cm-^{1} in Figure 2(a) correspond to v(C−H) stretching, which indicates the presence of hemi-cellulose for untreated fibres. [7] But the peak disappeared in Figure 2(b) because hemicellulose was reduced drastically after treatment. The absorption peak at 1733 cm^{-1} in Figure 2(a) is attributed to v(C=O) stretching of methyl ester and carboxylic acid in pectin [8]. The presence of this band at 1733 cm^{-1} indicates the presence of pectin; this peak seems to flatten in Figure 2(b) because pectin was removed after treatment. The absorbance peak of Natural fibres in the region of 1610 cm^{-1} indicates the presence of fatty acids [9] and is attributed to v(C=C) stretching confirming the of any traces of oils. The untreated fiber spectra also exhibit weak absorption peak at 1378 cm^{-1}, which indicates the presence of lignin and is attributed to v(C=C) stretching [10].

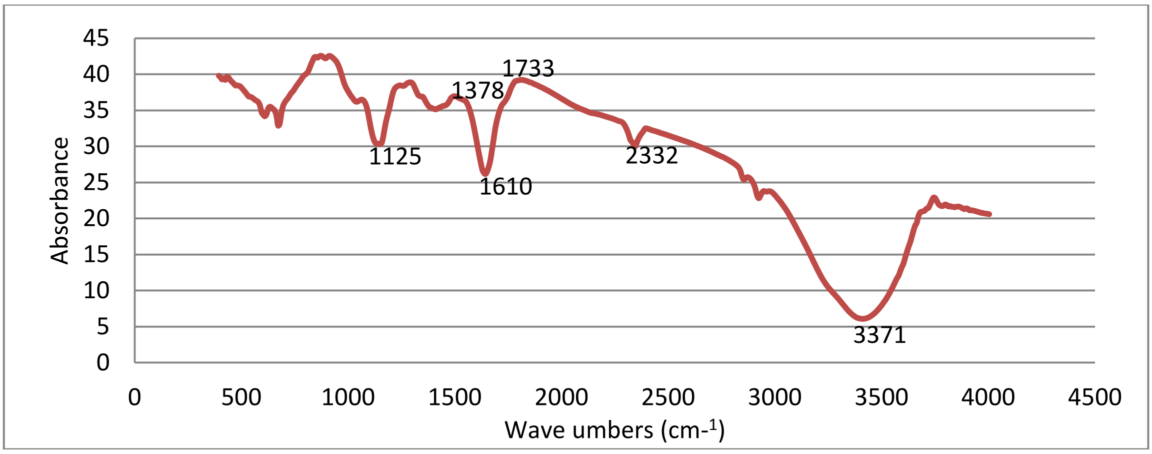


Figure 2a: FTIR for Raw Baobab pod fibres

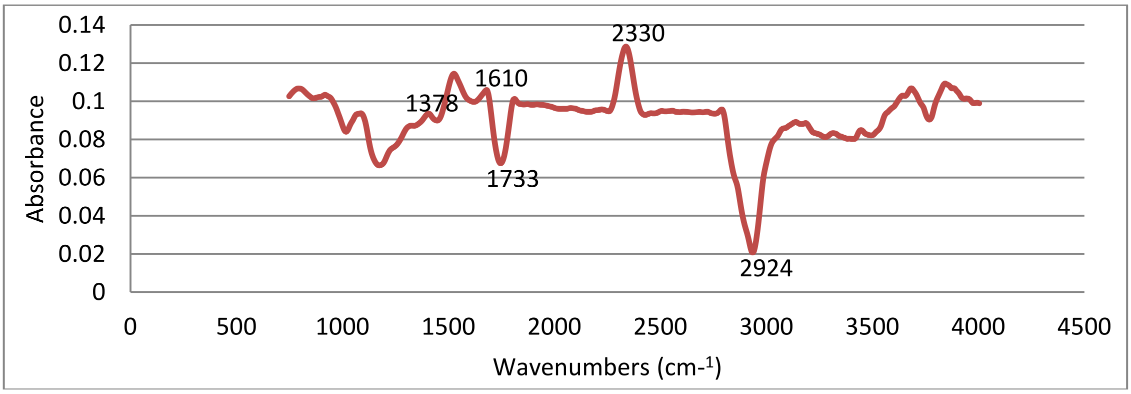
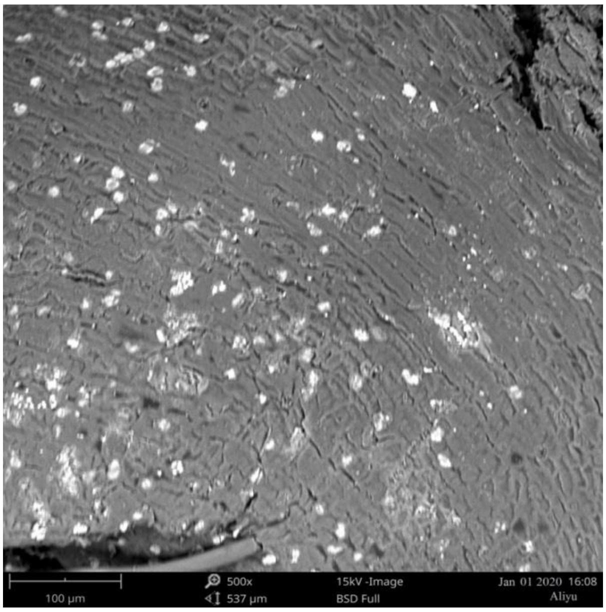


Figure 2b: FTIR for treated BPFs

3.5 SEM/EDS for raw BPFs

The micrograph indicates three distinct regions: an inner region (lumen), a middle region (cortex) and an outer surface (epidermis). Between the outer surface and the inner core, there are radial pathways, which probably serve as conduits for water/moisture exchange between the core of the fiber and the environment. Scanning electron micrographs of untreated BPFs shows the presence of longitudinal cracks on the surface of the fibers. Such type of cracks has been reported to affect the fracture behavior of natural fibers [11]. The cross-sectional structure of BPF is similar to the reported structure of sisal fibre [12].



Element Number	**Element Symbol**	**Element Name**	**Atomic Conc.**	**Weight Conc.**
6	C	Carbon	68.54	60.29
47	Ag	Silver	1.29	8.18
14	Si	Silicon	13.87	16.38
19	K	Potassium	2.69	6.16
17	Cl	Chlorine	2.77	5.76
39	Y	Yttrium	0.70	3.67
20	Ca	Calcium	1.07	2.52
26	Fe	Iron	0.60	1.96
15	P	Phosphorus	1.06	1.92
7	N	Nitrogen	2.15	1.77
16	S	Sulfur	0.92	1.74
13	Al	Aluminium	0.81	1.28
8	O	Oxygen	1.21	9.23
11	Na	Sodium	0.57	0.77
12	Mg	Magnesium	0.33	0.47

Figure 3: SEM/EDS for raw BPFs

3.6 XRD for raw BPFs

The XRD of raw baobab pod fibres (Figure 4) shows three intense peaks, which are peculiar to natural fibers. The presence of the diffraction peaks indicates that BPF is semi-crystalline in nature. The crystallinity index (CI) of the untreated BPF is estimated to be 71.2% which is higher than that reported by Elenga et al. of Wrighitia tinctoria seed fibers (49.2%), ramie (58%) and cotton (60%), but close to that of sisal (71%) and jute (71%), and smaller than that of flax (80%) and hemp (88%). Low crystallinity means that the fibres will have relatively high amorphous regions. These amorphous regions increases the amount of moisture absorbed in natural fibres [13].

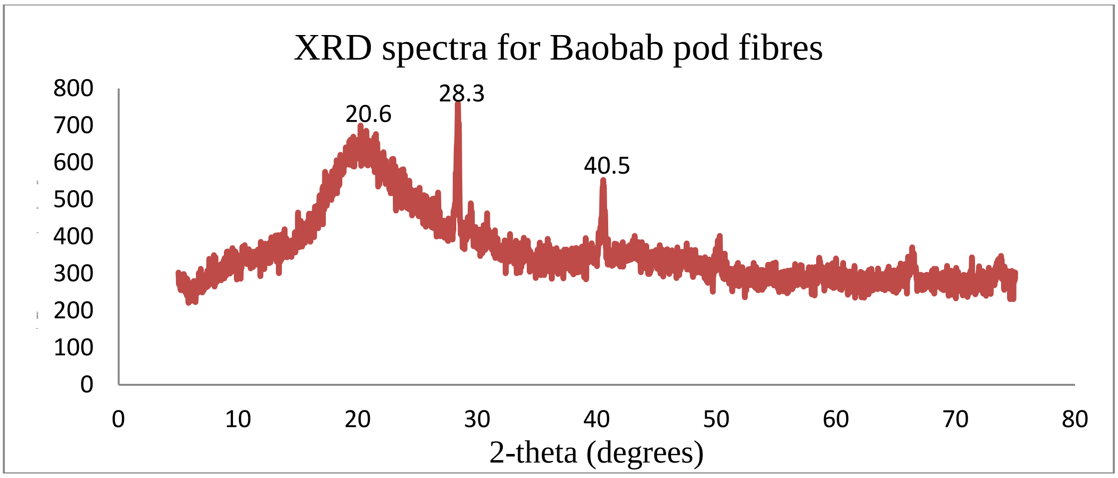


Figure 4: XRD for raw BPFs

3.7 Optimization using Response surface Methodology

Response Surface Methodology (RSM) is a collection of statistical and mathematical techniques useful for developing, improving, and optimizing processes. The most extensive applications of RSM are in the particular situations where several input variables potentially influence some performance measure or quality characteristic of the process.

Table 4: ANOVA Response for Single Fibre Tensile Strength Model

Std. Dev.	0.39	R-Squared	0.9992
Mean	35.53	Adj R-Squared	0.9981
C.V. %	1.09	Pred R-Squared	0.9925
PRESS	9.30	Adeq Precision	78.981
-2 Log Likelihood	1.04	BIC	29.37
		AICc	57.70

The "Pred R-Squared" of 0.9925 is in reasonable agreement with the "Adj R-Squared" of 0.9981; i.e. the difference is less than 0.2. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 78.981 indicates an adequate signal. This model can be used to navigate the design space.

Table 5: ANOVA Response for Single Fibre Young Modulus Model

Std. Dev.	0.53	R-Squared	0.9919
Mean	21.45	Adj R-Squared	0.9815
C.V. %	2.48	Pred R-Squared	0.9228
PRESS	18.84	Adeq Precision	45.738
-2 Log Likelihood	11.70	BIC	40.03
		AICc	68.37

The "Pred R-Squared" of 0.9228 is in reasonable agreement with the "Adj R-Squared" of 0.9815; i.e. the difference is less than 0.2. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 45.738 indicates an adequate signal. This model can be used to navigate the design space.

Table 6: Optimization solution for Alkali Treatment of BPFs

Number	NaOH Conc.	Soaking Time	Soaking Temp	Tensile Strength	Young Modulus	Desirability
1	6.212	169.388	45.632	34.157	20.884	1
2	2	360	67.5	31	19.081	1
3	2	120	67.5	26.25	15.791	1
4	2	240	100	24.425	20.545	1
5	2.533	256	39.333	25.078	14.155	1
6	8.489	138.667	43.185	29.794	21.394	1
7	3.081	180.889	90.491	32.657	20.561	1
8	8.844	313.086	36.658	27.622	21.523	1
9	6	360	35	31.775	22.376	1
10	6	120	35	26.825	21.366	1
11	3.042	286.37	92.698	33.573	22.932	1

12	10	240	35	20.975	19.315	1
13	8.14	263.185	42.47	32.487	21.212	1
14	9.148	326.06	36.858	26.906	21.642	1
15	4.23	302.672	36.019	29.677	18.438	1

3.8 Validation of predicted Model

In order to validate the optimization solution, the first and last predictions were taken and real experiments were carried out to determine the authenticity of the model. The percentage error for both TS and YM was obtained by subtracting the predicted results from the actual. Table 7 shows the validation test results for both TS and YM.

Table 7: Validation test results

Concentration (%)	Time (min)	Temp (℃)		TS (MPa)	YM (Pa)
			Actual	39.21	27.47
6.112	359.291	99.788	Predicted	44.27	30.738
			Error%	5.06	3.268
			Actual	43.78	28.66
6.963	359.963	99.763	Predicted	45.716	30.778
			Error%	1.936	2.118

4. Conclusions

In this investigation, the optimization of NaOH treatment conditions of Baobab Pod Fibres was achieved using Box-Behnken Method. Conclusions from this study are as follows:

1. NaOH treatment of baobab pod fibres alters the physical and mechanical properties of the fibres.
2. The higher the concentration of the sodium hydroxide used for the treatment, the lower the tensile strength and the young modulus.
3. A very high concentration of NaOH would certainly deteriorate the fibre and consequently reduce its tensile strength.
4. The removal of surface impurities of fibres increases the adhesion properties with polymer matrix.
5. The optimal conditions were found when the fibres were soaked in 6.2% NaOH concentration at 45.63◦C for 169.3 min (i.e., run # 1) in terms of tensile strength and young modulus.

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