CHEMICAL MODIFICATION OF LOCUST BEAN POD HUSK FIBRE AS A REINFORCING COMPONENT FOR POLYMER COMPOSITE APPLICATION

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J.A. OLOWOKERE[1]*, C.M. ODINEZE[1], U.G. AKPAN[2], J.O. OKAFOR [2], S.M. AUTA[3]
[1]Department of Chemical Engineering, Federal University Wukari, Taraba State, Nigeria.
[2] Department of Chemical Engineering, School of Infrastructure, Process Engineering and Technology, Federal University of Technology Minna, Niger State, Nigeria.
[3] Department of Civil Engineering, School of Infrastructure, Process Engineering and Technology, Federal University of Technology Minna, Niger State, Nigeria.
*Corresponding author: joariolo2@gmail.com

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Abstract: This paper focuses on the chemical modification of raw locust bean pod husk (LBPH) fibre using sodium hydroxide. The LBPH fibres were cleaned and reduced to a particle size range of (250-150µm). Sequential extraction method of biomass analysis was used to determine the chemical composition of the LBPH fibre. Analysis shows that the chemical composition consists of cellulose (59.3%), hemicellulose (20.2%), lignin (10.2%), extractive (9.1%) and ash (1.2%). 100 g of raw LBPH was mercerized at varying treatment concentration and contact time using NaOH to optimally improve the cellulose content, reduce the hydrophilic property of the LBPH fibre and improve the interfacial adhesion capacity of the LBPH fibre for polymer composite. Upon mercerization, a high optimum cellulose content of 80% was achieved using 1.5% NaOH at a contact time of 3 h. This indicates that the alkaline treatment process has a positive effect on the raw LBPH fibre. This is further confirmed by the FTIR spectrum of the treated LBPH fibre, recording minimal OH groups and aromatic lignin peaks. High NaOH treatment concentration weakens and denature the LBPH fibre. The LBPH treated fibre with the optimum cellulose content was preserved as a reinforcing material for polymer composite production.

Key Words: Chemical Treatment, Locust Bean Pod Husk, Cellulose Content, Mercerization, Contact time and Polymer Composite.

Introduction

The availability of abundant and cheap natural fibre alongside its ease of processing, for use as reinforcing materials in polymer composite, is currently of great interest to researchers. Several natural fibre such bagasse, kenaf, sisal, hemp, kapok, flax and coir have been extensively studied as reinforcing materials in polymer composite (PC), due to their eco-friendliness, light weight, high strength and corrosion resistance (Mohamed *et al*, 2021). However, a major challenge with polymer reinforced composites with natural fibre is their incompatibility leading to weak interphase and uneven dispersion within the (PC) (Government *et al,* 2019). Polymer is hydrophobic in nature while natural fibres are hydrophilic. These natural fibre majorly compose of hemicellulose, cellulose, lignin, water-soluble substance and wax (Radoor *et al*, 2020). This difference in character can be narrowed by chemical or physical modification of the fibre or polymer, thereby improving their compatibility (Kumar *et al*, 2018). The surface of natural sourced fibres can be chemically modified through processes like coupling agents, chemical treatment, and graft co-polymerization, with the essence of improving the mechanical properties of polymer composites (Kumar *et al*, 2018).

According to Malkapuram *et al* (2008), these processes reduce the hydrophilic property of the natural fibre. Alkali treatment is a suitable and cheap chemical treatment method of modifying natural fibre. The use of alkaline such as potassium hydroxide, sodium hydroxide, magnesium hydroxide and calcium hydroxide increases the surface roughness of fibre and also help to eliminate the network structure of the hydrogen bonding present within the natural fibre (Lee *et al,* 2009). This chemical treatment assists in removing the extractives (oils and wax) and lignin that form a coating around the cell wall of the fibre to a reasonable degree (Government *et al*, 2018). Exposition of the short length crystallites and depolymerization of the native structure of the cellulose is also observed (Li *et al,* 2009). The reaction which takes place between natural fibre (NF) such as locust bean pod husk and sodium hydroxide for the chemical treatment process is shown in Equation 1.1.

$$Fibre - OH + NaOH \rightarrow Fibre - O^{-}Na^{+} + H_2O \;\ldots\ldots\ldots(1)$$

Generally, the use of sodium hydroxide for the chemical treatment of natural fibre such as locust bean pod husk (LBPH) is referred to as mercerization.

LBPH is dark brown in colour with a density of l0.97kg/m^3 and pH of 6, indicating its weak level of organic acidity. The chemical composition shows that it has a tannin content of 27-44%. LBP concentrated extract are known to strengthen the water resistance of walls, ceramic pots and floors. Ash obtained from burnt LBP husk has been used for dying and tanning of indigo clothes and for soap making (Aguwa and Okafor, 2012). Ndububa and Uloko (2015) ascertained the water absorption capacity and compressive strength of the production of concrete mortar, using LBPH ash as a partial replacement of cement in the concrete mix. Adama and Jimoh (2011) reported that LBPH ash can be used as a pozzolan- which is a cementitious stabilizing additive in weak laterite for road construction.

This paper explores the alkaline treatment of LBPH fibre as a reinforcing material for PC production. The modification of the LBPH will help to improve its hydrophobic property and interfacial bonding capacity with polymer matrix, when used as reinforcing material for PC production. The raw LBPH fibre was subjected to sequential extraction of biomass analysis to determine the chemical composition. Mercerization was carried out on the fibre. The optimum parameters of the mercerisation process was statistically determined, and reported using design expert software. After which the effect of the mercerization process, as it relates to the functional groups was investigated using Fourier transform intra-red (FTIR) analysis.

Materials and Method

Materials

Raw LBPH fibres was collected and used for this research as the starting material, Soduum hydroxide was used for the treatment or mercerization process, alongside distilled water of analytical grade for dilution, washing and experimental purposes.

Sample Collection and Preparation of Locust Bean Pod Husk

Locust bean pod husk (LBPH) fibre was collected from the heap of agricultural waste of farmers involved in the processing of locust bean condiment in Bosso, Minna, Niger State. The husks were cleaned, dried in the sun until it attained a constant weight. After which the fibre was reduced into a particle size range of 60 -100 mesh (250-150 µm). It was further dried in an oven at a temperature of 80 ºC for 24 h to further reduce the moisture content. This was stored for chemical treatment (Muhammad *et al,* 2011).

Chemical Composition and Treatment of Locust Bean Pod Husk

The sequential extraction method of biomass analysis according to Sridevi *et al*, (2015) was used to determine the chemical composition of the raw and treated LBPH fibre. The prepared LBPH fibre was dried in an oven for 24 h at 80ºC to a constant weight. 100 g of LBPH each was taken and treated with sodium hydroxide based on the experimental design input factors. The treated LBPH was washed with enough distilled water until the pH of the solution attains neutrality to modify the LBPH particles in order to enhance better bonding and interaction with the polymer matrix. Thirteen (13) runs were carried out for the treatment process at different combination of NaOH concentrations and contact time. This was based on the experiment layout using central composite design. The run that was observed to have the highest optimum response of cellulose content was noted. This was stored and preserved as a reinforcing filler for polymer composite production (Olowokere *et al*, 2022).

Table 2.1 shows the layout of the experiment using central composite design (CCD) that was used in carrying out the chemical treatment process. The design layout on Table 2.1 shows two input factors with five level combinations to give 13 runs. This was carried out at varying treatment concentrations and contact time. Thirteen (13) runs were generated in all.

Table 2.1: Experiment Layout Using Central Composite Design

Input Factors	**-∝**	**-1**	**0**	**+1**	**+∝**
Treatment Concentration (%)	0.88	1.5	3	4.5	5.12
Contact Time (hours)	0.59	1	2	3	3.41

Fourier Transform Infrared (FTIR) Spectroscopy

The observations and changes in the functional group present in the raw and treated reinforcing fibre was determined and captured by Fourier Transform Infrared (FTIR) spectroscopy machine of model NICOLET 155 thermo scientific Nicolet corporation, madison USA. The FTIR spectra of the samples were analyzed in the range of 4000 cm^{-1} to 500 cm^{-1}.

Results and Discussion

The chemical compositions of locust bean pod husk (LBPH) for the untreated sample are presented in Table 3.1.

Table 3.1: Chemical Composition of untreated Locust Bean Pod Husk (LBPH) Fibre

Sample	**Cellulose %**	**Hemi-cellulose %**	**Lignin %**	**Extractive %**	**Ash %**
Untreated LBPH	59.3	20.2	10.2	9.1	1.2

Untreated Locust Bean Pod Husk (LBPH)

The chemical composition of the raw LBPH fibre, as shown in Table 3.1 is made up of cellulose, hemicellulose, lignin, extractives and ash. The extractives may include wax and oils. The cellulose content (59.3%) of the untreated LBPH is the highest among other components and the lowest is ash with 1.2%. This result is similar to that obtained by Pereira *et al* (2013) on several species of *Eucalyptus globulus* sawdust (EGS) wood for charcoal yield, and for untreated sugar palm fibre by Iiyas *et al* (2017). In most cases, plant and fruit fibre have higher cellulose content than wood fibre (Olowokere *et al,* 2022; Khairiah and Khairul, 2006).

The results of the treated LBPH are presented in Tables 3.2.

Table 3.2: Chemical Composition of Treated Locust Bean Pod Husk at Different Treatment Concentration and Contact Time

Std Run	**Factor 1**	**Factor 2**	**Responses**				
	Treatment concentration (%)	**Contact Time (h)**	**Cellulose %**	**Hemi-cellulose %**	**Lignin %**	**Extractives %**	**Ash %**
1	1.50	1.00	74.20	9.00	7.00	8.80	1.00
2	4.50	1.00	55.70	15.50	20.10	8.20	0.50
3	1.50	3.00	80.00	6.10	5.00	8.00	0.90
4	4.50	3.00	53.80	14.40	23.50	7.70	0.60
5	0.88	2.00	60.20	13.00	23.10	2.60	1.10
6	5.12	2.00	51.20	18.00	25.20	5.10	0.50
7	3.00	0.59	67.40	10.70	9.60	11.50	0.80
8	3.00	3.41	61.10	12.30	22.10	3.40	1.10
9	3.00	2.00	60.1	16.1	13.1	9.5	1.2
10	3.00	2.00	60.0	16.1	13.2	9.6	1.1
11	3.00	2.00	59.9	16.2	13.5	9.4	1.0
12	3.00	2.00	60.2	16.0	13.0	9.6	1.2
13	3.00	2.00	60.0	16.0	13.3	9.7	1.0

Alkaline Treatment of Locust Bean Pod Husk (LBPH)

The results on Table 3.2 for LBPH alkaline treatment shows that as the treatment concentration and contact time continue to increase from 0.87% to 1.5% for 1 – 2 h, the cellulose content increases, while other components like hemicellulose, lignin extractives and ash content continues to decrease. Iiyas *et al* (2017) recorded a similar observation with the exception of the ash content for sugar treated palm fibre. However, beyond 1.5% treatment concentration, the cellulose content begins to decrease progressively at 3%, 4.5% and 5.12% concentration. In this case, the highest cellulose content obtained for LBPH was 80% at experimental design conditions of 1.5% alkaline treatment concentration and contact time of 3 hours. It is evident that excessive alkaline treatment beyond 1.5% has adverse effects on LBPH fibre. This is an indication that LBPH fibre requires mild treatment process to achieve high cellulose content. In this case, LBPH fibre treatment concentration has a stronger effect on the fibre content than contact time. This is also observed by Jabar (2017).

Comparing the chemical composition of treated LBPH fibre and *Eucalyptus globulus* sawdust fibre, it is clear that the LBPH has higher cellulose content for both raw and treated fibre. This is an indication that sources of fibres from fruit husk are higher in cellulose content than in bast fibres such as wood sawdust (Olowokere *et al,* 2022; Khairiah and Khairul, 2006).

In most cases, natural fibres such as LBPH are subjected to chemical modification inorder to minimize their hydrophilic character. This is because, water-loving reinforcing materials poses a threat to the mechanical property and durability of polymer composites. This mercerization process assists in breaking the hydrogen bonds which exist between cellulose and other components such as lignin and hemicellulose. Similarly, free hydroxyl groups in the LBPH fibres capable of bonding with moisture are eliminated (Dittenber and GangaRao, 2012). The LBPH treated fibre with the optimum cellulose content was reserved as a reinforcing material for polymer composite production.

The FTIR Spectra of the raw and treated Locust Bean Pod Husk is presented in Figure 3.1 and Figure 3.2.

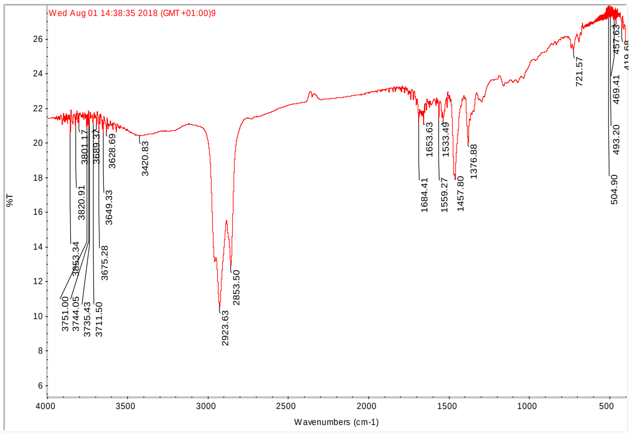

Figure 3.1: FTIR Spectrum for Raw Locust Bean Pod Husk

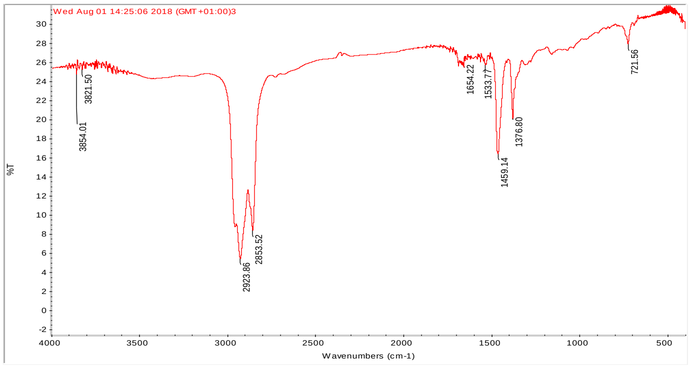

Figure 3.2: FTIR Spectrum for Treated Locust Bean Pod Husk

FTIR Analysis for Raw and Treated Locust Bean Pod Husk

Figure 3.1 presents the FTIR spectrum of raw locust bean pod husk (LBPH) fibre. The absorbance region is within the wavelength range of 500 cm^{-1} - 4000cm^{-1}. This extends from the fingerprint skeletal vibration region to single bond stretch region. The FTIR spectrum shows the three (3) basic chemical constituents found in natural fibre. These include cellulose, lignin and hemicellulose at their designated functional group and wavelength peaks. Starting from the single bond stretch region, multiple hydroxyl (OH) groups of not less than 12 peaks in the range of 3751.00 –3628.69cm^{-1} were observed. Functional groups within this range of peaks are commonly found in most natural fibres having hemicellulose and cellulose as part of their chemical compositions. This report is similar to Jabar (2017) and Ilyas *et al* (2017). The presence of these OH groups is a confirmation of the hydrophilic property of raw natural fibres, linked to the hemicellulose and cellulose component of the raw LBPH. These OH groups are held and bonded together by hydrogen bond. However, the hydrophilic nature of raw LBPH fibre can be a limiting factor for it to be suitable for use as a reinforcing material in polymer composite application. This is because of its incompatibility with the hydrophobic character of polymer matrix (Jabar, 2017; Olowokere *et al*, 2022). Furthermore, Figure 3.1 presents the alkane C-H peaks observed at 2923.63 and 2863.50 cm^{-1}. This is also an indicative presence of hydrocarbons in raw LBPH fibre, which also reported by Khalil *et al* (2001). Further observations reveals that variable C=C aromatic rings and C=C double bond stretch groups are present in the spectrum. These are indicative of the presence of lignin components, having the attributes of aromatic as observed at (1684.41 – 1653.63 cm^{-1}) and (1559.27 – 1533.49 cm^{-1}) respectively. Similarly, C –Cl stretch peak for aliphatic chloro compound is captured at a wavelength of 721.57 cm^{-1}. This is similar to the results obtained by Olowokere *et al* (2022).

The subjection of raw fibre to chemical treatment is of great importance, if it must serve as valuable reinforcing materials in polymer composite application. The FTIR spectrum in Figure 3.2 presents the treated LBPH fibre with the optimum cellulose content yield at 1.5% NaOH for 3 h. Observation from the FTIR spectrum shows there are minimal peaks associated with the OH hydroxyl group at 3854.01 – 3821.50 cm^{-1}. This reduction in OH group peaks is a manifestation of the effect of the alkaline treatment process, which has shifted and transformed the hydrophilic character of the raw LBPH fibre to a hydrophobic one, for ease of compatibility with any available polymer matrix. This transformation is possible through the breaking of the hydrogen bond network within the LBPH fibre by the NaOH, thereby exposing more crystalline cellulose components with active surface site for good interfacial bonding. Similarly, there are minimal aromatic lignin peaks at 1654.22 and 1533.77 cm^{-1}. In this case, the chemical treatment is able to initiate and promote the depletion of some of the amorphous aromatic lignin components, which also poses a setback to good interfacial bonding within a polymer composite.

Statistical Analysis of the Optimization Treatment Process

The analysis of variance (ANOVA), model summary, model equation and optimum parameters of the LBPH treatment process, is presented in this section.

The statistical analysis of variance (ANOVA) for LBPH cellulose response is presented in Table 3.3.

Table 3.3: ANOVA for LBPH Cellulose Response Surface Quadratic Model

source	Sum of Squares	df	Mean square	F value	p- value prob>F	
Model	415.38	2	207.69	6.23	0.0175	significant
A- treat conc	412.25	1	412.25	12.38	0.0056	
B-contact time	3.14	1	3.14	0.094	0.7652	
Residual	333.12	10	33.31			
Lack of fit	333.07	6	55.51	4270.17	<0.0001	significant
Pure error	0.052	4	0.013			
Cor.Total	748.51	12				

In Table 3.3, the model F- value of 6.23 signifies that the model is significant. There is only a 1.75% chance that a "model F-value" of this magnitude could probably occur as a result of noise. Values of "prob >F" that are less than 0.05 indicate a significance in their model terms. In this case, treatment concentration (A) is a significant model term since the "prob>F" value is less than 0.05 as shown in Table 3.3. While values greater than 0.1 indicate the model terms are not significant. In this case, B which is 0.7652 is not a significant model term in relation to "prob>F". The lack of fit F-value of 4270.17 is significant and this actually influences the fitness of a model. This is similar to the report of Salako *et al* (2020).

The model summary of the statistical analysis of LBPH cellulose response is presented in Table 3.4.

Table 3.4: Model Summary for LBPH Cellulose Response

Std. Deviation	Mean	C.V (%)	PRESS	R-squared	Adj. R-squared	Pred. R-squared	Adeq. precision
5.77	61.83	9.33	718.69	0.7549	0.6659	0.0398	7.323

The information on Table 3.4 shows that the "Pred R-Squared" of 0.0398 is not as close to the "Adj R-Squared" of 0.6659 as one might normally expect. This may indicate a large block effect or a possible problem with the model and/or data. Things to consider are model reduction, response tranformation, outliers. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. This ratio of 7.323 indicates an adequate signal. This model can be used to navigate the design space. This is somewhat similar to the report of Salako *et al* (2017).

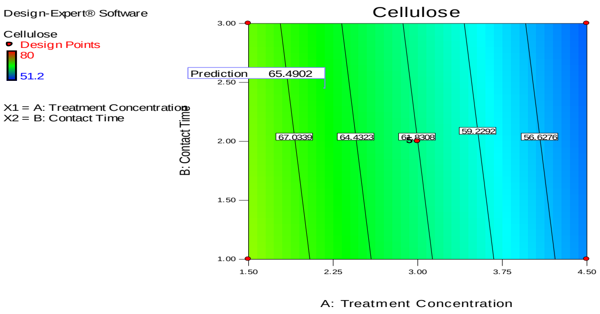

Figure 3.3: Predicted Optimum Cellulose Response Contour Plot for LBPH

The final model equation that describes the LBPH treatment process in terms of coded factors is expressed in Equation 2.

Given that A = Treatment Concentration and B = Contact Time

Cellulose = 61.83 -7.18A – 0.63B ----------------------------(2)

Equation 2 is valid within the range of 1.5 to 4.5% treatment concentration and contact time of 1 to 3 h.

The predicted optimum cellulose response contour plot and the overlay plot for LBPH are presented in Figure 3.3 and 3.4 respectively.

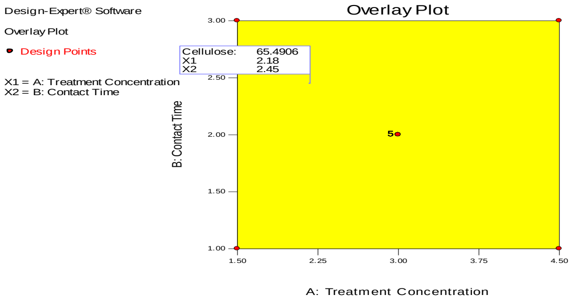

Figure 3.4: Overlay Plot for Optimum Cellulose Response Parameter for LBPH

Figures 3.3 and 3.4 present the optimum treatment conditions that give the optimum composition of cellulose and other non-cellulosic chemical composition of the treated LBPH fibre. The predicted optimum alkaline treatment concentration is 2.18% at a contact time of 2.45 h, to give a cellulose composition of 65.4906%. This value is less than the actual experimental value obtained upon confirmation. In the actual experimental analysis of the optimum treated LBPH fibre, a high cellulose content of 80% was achieved using 1.5% NaOH at a contact time of 3 h. the actual cellulose content obtained from the experiment is slightly higher than the predicted value. This observation is similar to Government *et al* (2018).

Conclusion

Natural fibres such as locust bean pod husk are promising reinforcing materials for polymer composite production. However, these fibres need to be chemically modified to better suit this purpose through mercerization. This will assist in optimally improving the cellulose content, reduce the hydrophilic property and ultimately improve the interfacial adhesion bonding capacity of the natural fibre with the polymer matrix in a composite. Using sequential extraction method of biomass analysis, the chemical composition of raw locust bean pod husk fibre was characterized. It was found to be composed of cellulose, hemicellulose, lignin, extractives and ash. Out of these 5 components, the cellulose content recorded the highest composition of 59.3%. This value increased to 80% upon exposure to alkaline treatment at a NaOH concentration of 1.5% for 3 h. Furthermore, the alkaline treatment is able to improve the crystallinity nature of the LBPH fibre, while reducing its hydrophilic character for better interfacial bonding for polymer composite application. This is confirmed by the FTIR result. All of these observations are confirmation of the positive effect of the alkaline treatment process of the LBPH fibre.

Author Contributions

This work was carried out in collaboration among all the authors.

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