



# Optimizing the Cellulose Content of Shea Butter Bark Wood Fibre through Alkali Pretreatment for Composite Application

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## Abstract

**Objectives:** This study was aimed to optimize the cellulose content (CC) increase with the percentage removal of hemicellulose (HC) and lignin content (LC) of shell butter bark wood fiber (SBBWF) by alkalization process. The truth remains that natural fibers have many uses to be employed in all human activities. The minimization of plant waste is a secret for close economy by effectively converting it to useful ventures. **Methods/findings:** The SBBWF was inserted in variant solutions for the concentration of sodium hydroxide (CSH) at 3–9 wt% and the soaking time (ST) for 6–18 h. The SBBWF before and after pretreatment were analyzed through gravimetric method and Fourier transform infra-red (FTIR) to determine its composition and organic substance with corresponding functionality, respectively. The optimization was examined using the central composite design (CCD), an option in response surface methodology (RSM). The results at optimal conditions indicated that the increment in CC with the reduction of HC and LC was 97.9988%, 44.5245%, and 45.716%, respectively. The factors at this junction were CSH and ST at 3 wt% and 18 h, respectively. The  $R^2$  values of CC, HC and LC had an approximation nearer to 1. The errors approximated by RSM and experimental readings were <0.43%. **Application:** Therefore, the modified SBBWF at this state is potential material in the component of composite manufacturing to be applied in domestic utilization.

**Keywords:** Shell Butter Bark Wood Fiber, SBBWF, Wealth from Waste, Natural Source, Wood Fibre.

## 1. Introduction

Natural fiber from a wood source has been a potential material as a component for the field of science, engineering and technology in solution to mankind problems. The introduction of fiber from a woody substance in daily activities has geometrically increased globally. However, the huge deposits of these raw materials, lower cost of excavation and preparation, and lighter in terms of density are the reason behind its application in paper, polymer composites, and carbon sheet, building, automobile, and so on [1–11].

The bark from the tree of this wood species, SBBWF is regarded as waste after extracting the timber. In the world at present, technocrats in all area of studies have emphasized to utilize all wastes in the environment with the objectives that these can be translated to useful materials for manufacturers to be applied in both domestic and industrial purpose [12–15]. Regulations and attempt have to be put in place to ensure these wastes are utilized to zip up wastes by recycling and the economy of any nation will improve [16]. In addition, the advantage of land usage in other works of life increases when properly embarks on completely harnessing of wastes for a useful endeavor [17].

Although, SBBWF is basically an agro waste, its compositional part is mainly cellulose. The other major component is hemicellulose, followed by smaller compound identified as lignin and finally with some minute smallest substances in the fiber [18–19]. The percentile of cellulose in the SBBWF is variable depending upon age of the tree, location and plant variety, modification, the yield and period for the growth of the fiber [20].

The de-lignification and reducing some extra constituents of SBBWF such as hemicelluloses and soluble material are essential for its capacity to enhance the cellulosic content for effective application in multiple purposes [21–22]. Therefore, the need for pretreatment of SBBWF is the remedy for reduction of ineffective components. When SBBWF is not pretreated before employing in any industrial processes; it would lead to more water sorption, low adhesion of the fiber when combining with another material such polymer matrix for composite making and poor capability to inculcate stress to another substrate during processing [23]. Furthermore, many methods have been applied for treating the fiber for effectual bonding.

This involves the use of acidification, alkalization, bleaching agent, oxidation and peroxide chemical modifying procedure, etc. By acidifying the bark, denotes soaking of the fiber in either solution of  $\text{H}_2\text{CO}_3$ ,  $\text{HCl}$ ,  $\text{HNO}_3$  or  $\text{H}_2\text{SO}_4$ , etc [24]. The modification of plant fiber with chlorite remains the best option in treating the fiber by bleaching process [12]. In addition, subjecting the fiber through the passage of oxygen gas or in aqueous hydrogen peroxide is another method for activation of cellulosic percentage. Moreover, the simplest method presumes for surface modification of SBBWF is alkalization [22]. Alkalization is the immersion of the SBBWF in a basic solution for the improvement of roughness and cellulose percentage of the fiber. It allows the fiber to activate OH group which provides intermingling of fiber surface, and enhancing the bonding properties of the SBBWF. The reagents employ for this method are  $\text{NaOH}$ ,  $\text{Ca}(\text{OH})_2$ ,  $\text{KOH}$ ,  $\text{Mg}(\text{OH})_2$ , etc [25]. Hence, it is been narrated that bark with a large amount of cellulose gives better stiffness and strength [21].

The mechanism at the period for modification of SBBWF includes: components chain of the fiber loses, solubility and decreasing of undesired compositional compounds in

the bark and more content of cellulose. Furthermore, the crystalline characteristics are degraded; voids and surface area of bark are tremendously improved [26–28].

The factors that serve as the determinant for the composition of the fiber includes: temperature of modification, the ratio of bark content to solution concentration and pre-treated solution time [29].

Later scholars have implemented different reagent to improve the cellulosic component of multiple fibers. These are enlisted: *Pterocarpus angolensis* (wukwa) [27], pinewood oil palm empty fruit bunch [29–31] corn husk fiber [32], waste betel nut husk fiber [33], Mendong straw [19], rice straw [17], wood pulp [34], century fiber [21], sugar cane basse [35], flask fiber, banana fiber, the typha fiber, rice husk, etc [36–37].

In this research, the optimization of process parameters in alkali-treated novel SBBWF was studied with aim of improving the cellulose, reducing hemicelluloses and lignin content for its functionality in composite making on varieties of industrial applications.

## 2. Material and Methods

### 2.1. SBBWF

The SBBWF (*Vitellaria paracloxum*) called kadanya in Hausa was sourced in Tella, Taraba, Nigeria. The bark was peeled out from the shell butter wood tree. The bark was dried in 8 hours for 8 weeks in the sun, ground with aid of electric blender Model Vitamix A3500 and sieved employing a 20 mesh size (850  $\mu\text{m}$ ). The SBBWF of 10 g was soaked in NaOH solution at 3% wt, 6% wt and 9% wt for a time of 6, 12 and 18 h in a different laboratory beaker. The SBBWF was rinsed with distilled water for five times after completion of the absorption time. The SBBWF was placed in an oven at 90 °C and allowed to dry for 17 h.

### 2.2. Evaluation of Cellulose Content

The SBBWF of 10 g (m) was immersed in a mixture of nitric acid (ACS reagent grade), and ethanol solution (ACS grade A1040) for 1 h.

The SBBWF was separated with Whatman filter paper and cleaned with heated water for 50 °C. The solid SBBWF at the top of the filter was maintained at 100 °C in an oven for a fixed weight ( $m_1$ ). The SBBWF cellulose was estimated by applying eq. (1)

$$\% \text{SBBWF content of cellulose} = \frac{m_1}{m} \times \frac{100}{1} \quad (1)$$

where  $m_1$  and  $m$  are the weight of SBBWF before and after oven-dried.

### 2.3. Determination of the Hemicellulose Content (Neutral Detergent Fiber Method)

The SBBWF of 10 g was refluxed with a solution of 50 ml sodium lauryl sulfate (certified reference material pharmaceutical secondary standard). The SBBWF residue in the mixture was removed out and cleaned in distilled water at 50 °C. The SBBWF residue

heated in an oven for 8 h at 100 °C and weighed ( $m_2$ ). The procedure was repeated with 5 ml of 72% w/w  $H_2SO_4$  solution (HPLC grade) and the final residue after oven-dried tagged as  $m_3$ . The SBBWF hemicelluloses were computed employing eq. (2)

$$\% \text{ SBBWF hemicellulose} = \frac{m_2 - m_3}{m} \times \frac{100}{1} \quad (2)$$

where  $m_2$  and  $m_3$  are the residues after oven-dried with base and acid, respectively.

## 2.4. Determination of the Lignin Content

The SBBWF with the same initial weight was fired with 5 ml solution of 72% w/w  $H_2SO_4$  for 30 min. The precipitation of SBBWF recorded was rinsed with a mixture of ethanol and de-ionized water at 50 °C. The deposition of SBBWF after filtering and drying in the oven at 105 °C for 24 h weighed as  $m_3$ . The deposit of SBBWF was also moved to another porcelain crucible and heated to 600 °C at 5 h, finally cooled and weighed as  $m_4$ . The SBBWF lignin content was evaluated using eq. (3)

$$\% \text{ lignin content} = \frac{m_3 - m_4}{m} \times \frac{100}{1} \quad (3)$$

where  $m_3$  and  $m_4$  are the residue weight of SBBWF at 105 °C and 600 °C after cooling, respectively [38].

## 2.5. FTIR Examination of SBBWF

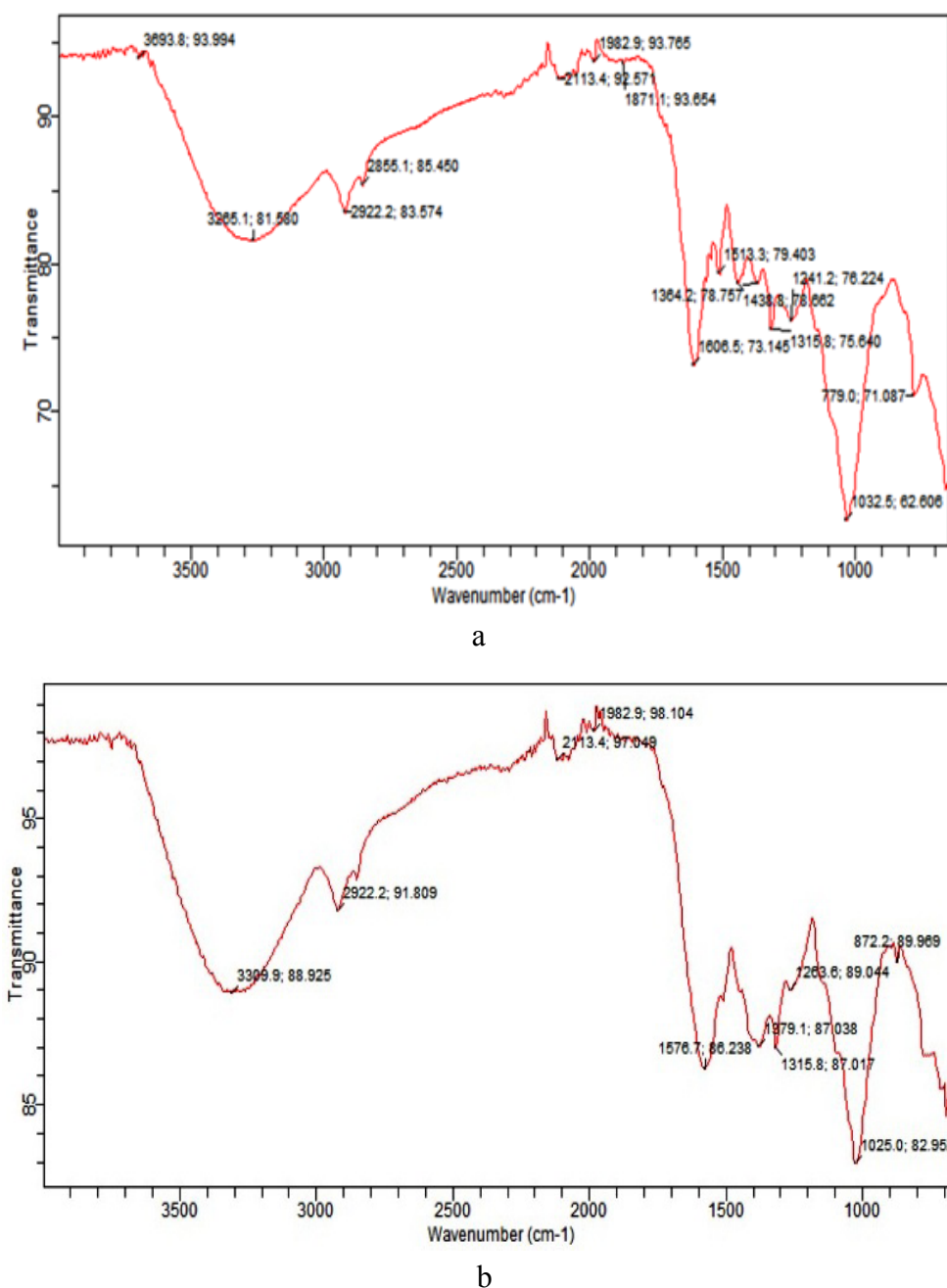
The FTIR features of SBBWF were examined using a Shimadzu spectrometer Model 8400S. The SBBWF of 1.5 mg was mixed with 50 mg KCl. The mixture was slotted in the FTIR equipment. After 50 s, the functionality in SBBWF is determined through the corresponding peak in the wavelength by the spectrum as display in the machine.

## 2.6. Modeling and Optimization

The software, design expert version 7.0 was employed using RSM of CCD. The faced center option of CCD was used to input the response and factors resulting in 13 experimental runs. The analysis of variance was structured to maintain a 95% confidence level.

# 3. Result and Discussions

Figure 1(a–b) illustrates the FTIR of unmodified and pre-treated SBBWF, respectively. As noticed in Figure 1(a), the occurrence of OH and C–H bond can be stared at  $3365.1 \text{ cm}^{-1}$  and  $2922.2$  at 81.58% and 83.574% in transmittance corresponding to alcohol and alkane, respectively. These were observed in Figure 1(b) at  $3309.9 \text{ cm}^{-1}$  (OH) and emergence of the bond C–H with an improvement of the percentile transmittance at 88.925% and



**FIGURE 1.** FTIR spectra of SBBWF for (a) unmodified (b) modified.

91.908% when immersed in NaOH solution, respectively. The NO<sub>2</sub> aromatic compound was found in Figure 1(a) at 1513.3 cm<sup>-1</sup> at 76.224%. After pre-treatment as narrated in Figure 1(b), the C–N and C=C bonds of pyridine emerged at 1576.7 cm<sup>-1</sup> at 86.238%. The observation is due to the activation of SBBWF with OH ions which improve the focal content of SBBWF and reducing unwanted substance in the fiber after treatment. In

conclusion, the enhancement of the transmitting percentile and formation new substance after alkali modification of SBBWF exhibited an increment in CC and removal of HC and LC, respectively as reported [39].

Table 1 indicates the design matrix for the composition of modified SBBWF. As shown in Table 1, the combination of the variation of CSH and ST to yield the increase in CC and the percentile reduction of HC and LC after pre-treatment. The combined CSH, ST, and the corresponding improvement in CC, the HC and LC removal formed the empirical models whose equations were applied to forecast the enhancement of main compositional constituent of SBBWF. The regression equations for CC, HC, and LC are stated using eq. 4, eq. 5, and eq. 6, respectively.

$$CC = 52.62 - 36.6CSH + 5.52ST - 1.42CSH ST + 2.59CSH^2 - 0.75ST^2 \quad (4)$$

$$HC = -3.49306 + 8.1707CSH + 2.27124 ST - 0.085389CSH ST - 0.47544CSH^2 - 0.026194ST^2 \quad (5)$$

$$LC = +72.05883 - 14.93440CSH - 5.96737ST + 0.26747CSH ST + 1.33251CSH^2 + 0.30690ST^2 \quad (6)$$

The ANOVA for the chemical constituent of chemically modified SBBWF is mentioned in Table 2. The model for CC, HC, and LC was thoroughly significant. In addition, the coefficient of the variables, CSH, ST, CSH\*ST, CST<sup>2</sup>, and ST<sup>2</sup> for CC, HC, and LC sustained probability value <0.042. The R<sup>2</sup> of CC, HC and LC retained decimal values close to 1. The predicated R<sup>2</sup> showing related values of the adjusted R<sup>2</sup>. Moreover, the differentials of both R<sup>2</sup> were minimal. These indicate a complete increase in CC, high experimental overhauling of HC and LC in the SBBWF by NaOH modification process. With the high values of R<sup>2</sup> for CC, HC, and LC; these mean that the precision and reliability of the study were carefully carried out. The results for CC, HC, and LC were the familiar trends by authors in the research area [40–44].

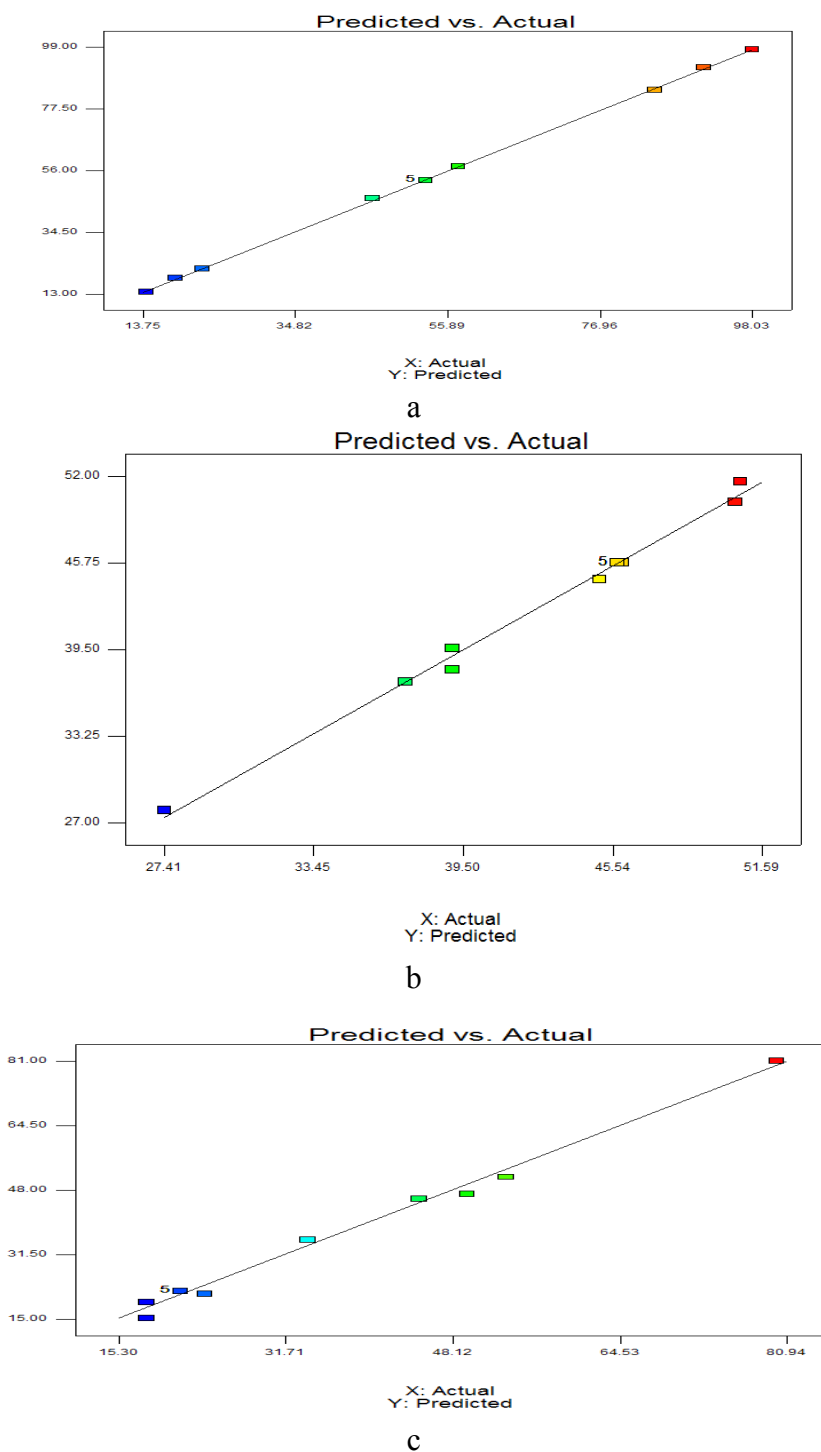
**TABLE 1.** Design matrix for the composition of modified SBBWF

Run	Factors		Responses		
	CSH	ST	CC	HC	LC
	%	h	%	%	%
1	6	12	52.788	45.72	21.326
2	6	12	52.788	45.72	21.326
3	9	18	21.959	50.5	79.904
4	6	12	52.788	45.72	21.326
5	6	12	52.788	45.72	21.326
6	3	12	91.357	37.155	23.745
7	3	6	84.55	27.407	17.989
8	6	12	52.788	45.72	21.326
9	9	6	14.146	39.056	33.883
10	9	12	18.221	45.938	49.475
11	6	18	57.362	50.709	53.331
12	3	18	98.029	44.999	44.752
13	6	6	45.53	39.056	18.001

**TABLE 2.** ANOVA of compositional modified SBBWF

Source	Sum of squares	df	Mean square	F value	p-value Prob > F	
CC						
Model	8247.719	5	1649.544	7669.797	<0.0001	Significant
A CSH	8038.092	1	8038.092	37374.29	<0.0001	
B-ST	182.8666	1	182.8666	850.265	<0.0001	
AB	8.025889	1	8.025889	37.31755	0.0005	
A^2	18.55428	1	18.55428	86.27084	<0.0001	
B^2	1.558146	1	1.558146	7.244831	0.0310	
Residual	1.50549	7	0.21507			
Lack of fit	1.50549	3	0.50183			
Pure error	0	4	0			
Cor total	8249.225	12				
Std. dev.	0.463756		R-squared	0.999817		
Mean	53.46877		Adj R-squared	0.999687		
			Pred R-squared	0.998563		
HC						
Model	469.4282	5	93.88565	236.1121	<0.0001	Significant
A-CSH	112.0867	1	112.0867	281.8858	<0.0001	
B-ST	275.9325	1	275.9325	693.9397	<0.0001	
AB	9.449476	1	9.449476	23.76439	0.0018	
A^2	50.56963	1	50.56963	127.1771	<0.0001	
B^2	2.455931	1	2.455931	6.176397	0.0419	
Residual	2.783422	7	0.397632			
Lack of fit	2.783422	3	0.927807			
Pure error	0	4	0			
Cor total	472.2117	12				
Std. dev.	0.63058		R-squared	0.994106		
Mean	43.34		Adj R-squared	0.989895		
			Pred R-squared	0.94022		
LC						
Model	4208.439	5	841.6878	181.5719	<0.0001	Significant
A-CSH	982.4257	1	982.4257	211.9324	<0.0001	
B-ST	1948.106	1	1948.106	420.2524	<0.0001	
AB	92.71764	1	92.71764	20.00138	0.0029	
A^2	397.2207	1	397.2207	85.68987	<0.0001	
B^2	337.1471	1	337.1471	72.73057	<0.0001	
Residual	32.44894	7	4.635562			
Lack of fit	32.44894	3	10.81631			
Pure error	0	4	0			
Cor total	4240.888	12				
Std. dev.	2.153036		R-squared	0.992349		
Mean	32.90077		Adj R-squared	0.986883		
			Pred R-Squared	0.947539		

Figure 2(a-c) pictures the graph of real experimental values against the RSM values for the improvement in the composition of the treated SBBWF for CC, HC, and LC, respectively. From Figure 2(a), (b), and (c), it was captured that the plotted points coincided with the diagonal line for CC, HC and LC, respectively. This assertion indicated that the models for CC, HC, and LC using RSM gave better forecast on the upgrading of the CC,



**FIGURE 2.** Actual versus predicated plots for (a) CC (b) HC (c) LC of modified SBBWF.



and degrading of HC and LC by treated SBBWF, respectively. The similarity of the study has been emphasized by earlier workers [44–47].

From Figure 3(a) and (b) presents a graphical description of the contour and 3-D surface of the increase in pre-treated SBBWF CC, respectively. It was depicted that the optimum prediction of CC increment was seen in the reddish region of the contour at 97.9966% in Figure 3(a). However, the maximum CC corresponded to CSH at 3% wt and ST of 18 h.

In addition, in Figure 3(b), for constant ST, the CC reduced at high CSH increase. This phenomenon is due to a higher CSH, the cellulosic constituent of SBBWF is shattered. Meanwhile, at steady CSH, the CC inclined at more ST of SBBWF in NaOH. The existence of this fact is as a result of longer ST yields more CC of modified SBBWF. These observed facts were in conformity with previous reporters [48].

The HC removal of pre-treated SBBWF contour and 3-D surface plots is presented in Figure 4(a) and (b), respectively. Moreover, the optimum HC removal was traced at the yellowish vicinity at the prediction of 44.539% as shown in Figure 4(a). At this point of optimum HC, the CSH and ST were cited at 3 wt% and 18 h, respectively.

With respect to Figure 4(b), the HC removal was found to increase with the increment in CSH at steady ST. This is attributed to the modification of SBBWF enhances the elimination of HC. In addition, at fixed CST, the HC removal capacity was upgraded during higher ST. Similar expectations were recorded by earlier scholarly articles [49].

Figure 5(a) and (b) portrays the removal of LC in treated SBBWF contour and 3-D surface plots, respectively. As shown in Figure 5(a), the optimum prediction of LC removal in treated SBBWF is situated in the green region of the contour plot at 45.716%. The CSH and ST were domiciled at 3 wt% and 18 h, respectively.

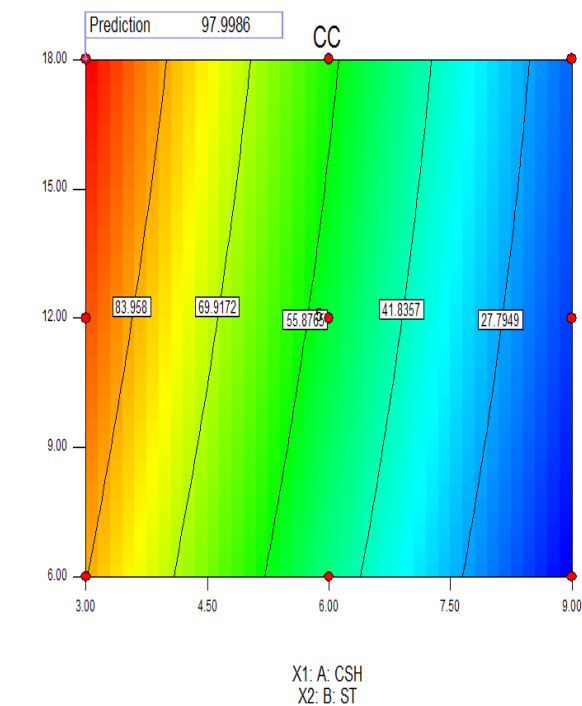
It was traced in Figure 5(b) at constant CSH, the LC removal amplified at more ST. This trend in HC was observed in LC. When ST is steady, the LC removal increases after absorbing in more CSH. The reason behind the phenomenon is that during immersion of SBBWF in NaOH, most of the undesirable component has been eliminated making the fiber to boost its intermingle ability and the cellulosic amount due to activation of the fiber surface by OH radicals. Preceding scholarly works have obtained a close outcome [50–51].

Figure 6 displays the overlay plot for enhancing the CC, the reduction of HC and LC from treated SBBWF. From the above plot, the optimal CC, HC, and LC of the pre-treated SBBWF occurred at 97.9988%, 44.5245%, and 45.718%, respectively. These coincided with CSH and ST of SBBWF at 3 wt% and 18 h, respectively. Therefore, the SBBWF at this pre-treating condition is required for composite production in domestics use.

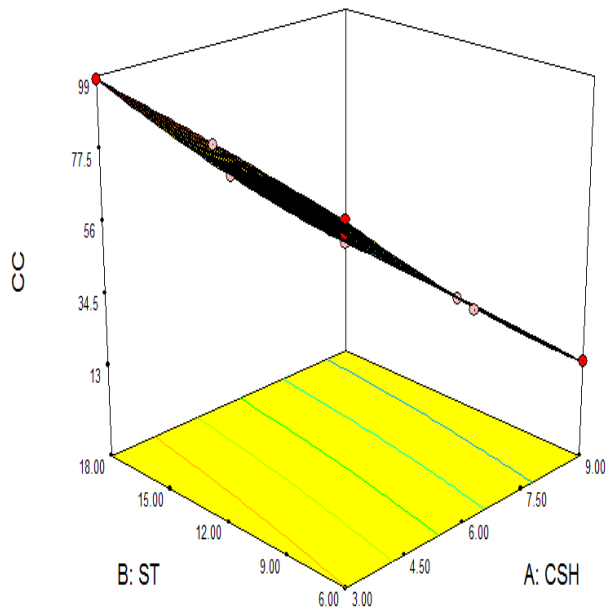
Table 3 presents the comparison of the predicted CC, HC, and LC for modified SBBWF at the optimal situation. From the observation in Table 3, the percentage relativities between CC, HC, and LC of SBBWF chemically modified were barely <0.43%. This is a significant affirmation that the RSM software forecasted the experimental data with lower errors.

## 4. Conclusion

The work presents SBBWF as novel and low cost material which can be applied in the field of composite engineering. The process parameters; CSH and ST were able to show

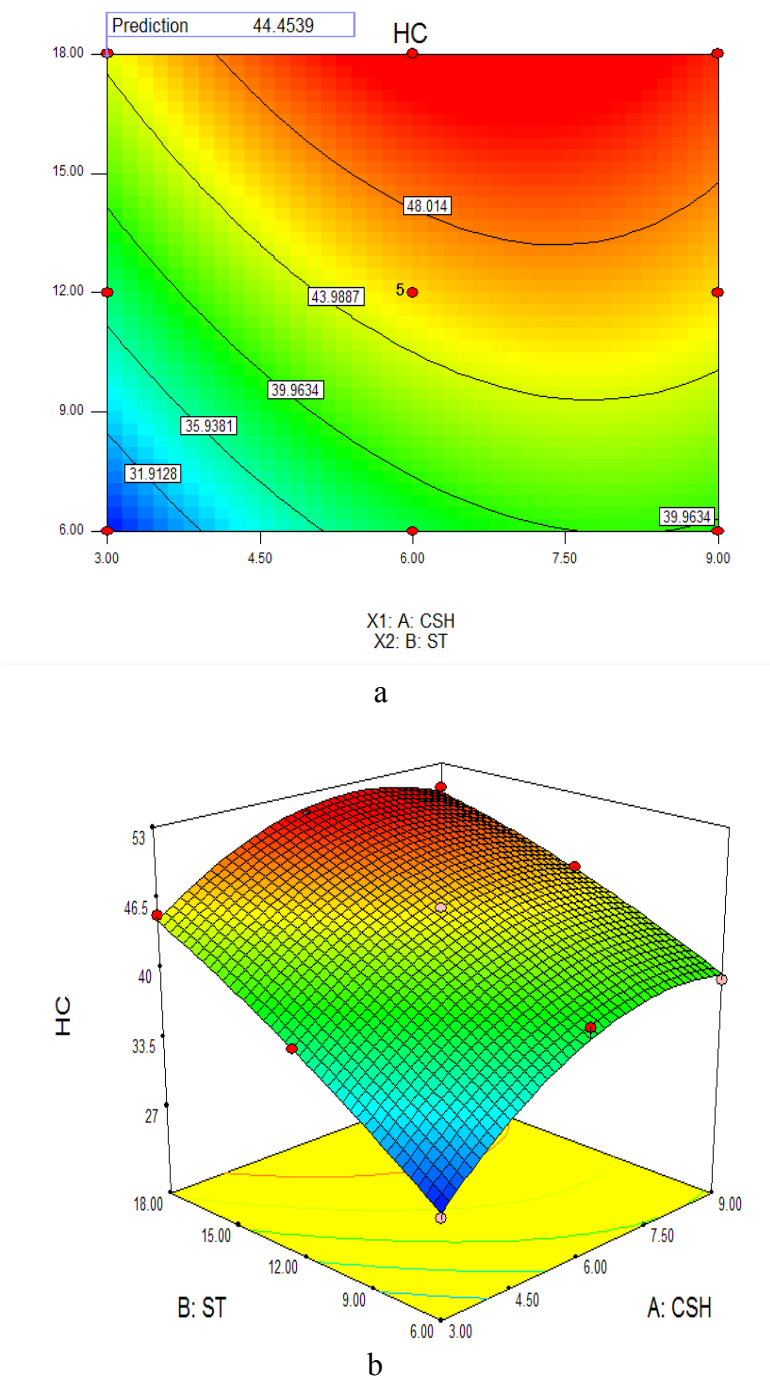


a

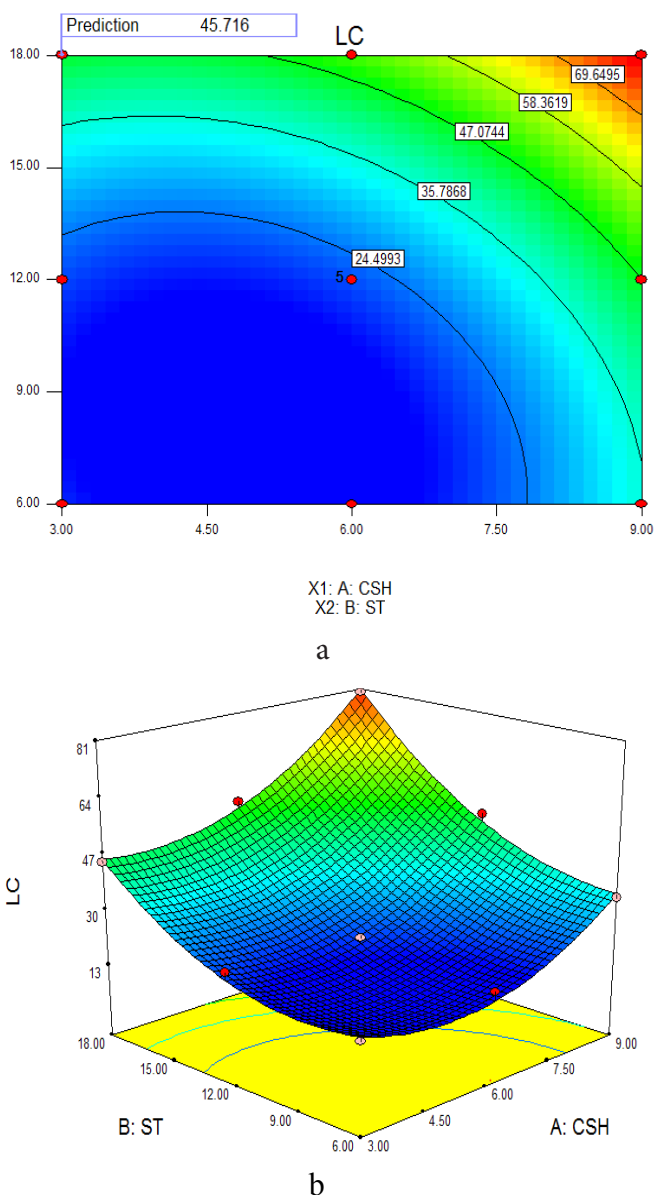


b

**FIGURE 3.** The enhanced CC of pre-treated SBBWF graph for (a) contour (b) 3-D surface.

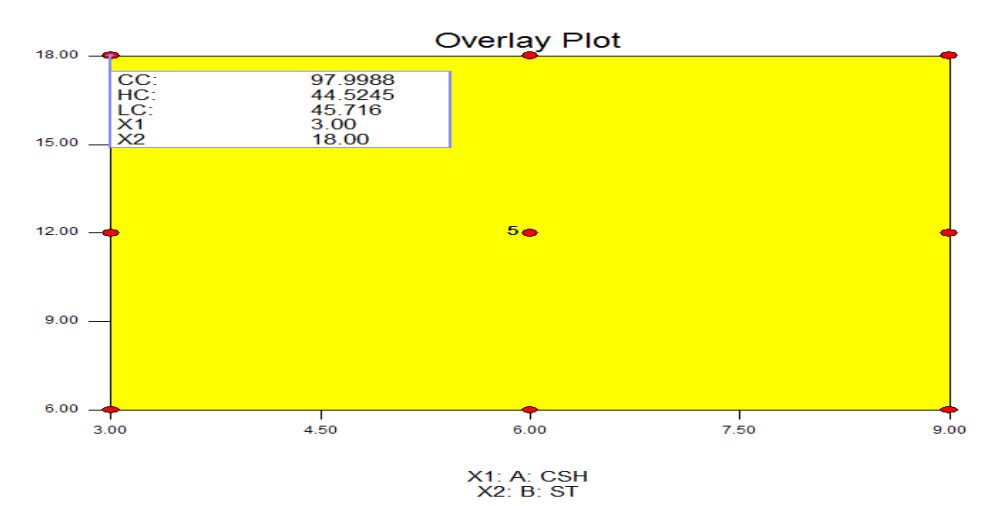


**FIGURE 4.** The percentage removal of HC in pre-treated SBBWF for (a) contour (b) 3-D surface.



**FIGURE 5.** The percentage removal of LC in pre-treated SBBWF for (a) contour (b) 3-D surface.

more impact on the composition of SBBWF. The pre -treatment process of SBBWF was able to enhance the CC and facilitated the HC and LC removal. FTIR spectra were able to show the main difference before and after treatment. The RSM was able to forecast the CC, HC and LC with minimal error. The data obtained from the estimated values of RSM prediction of pre-treated SBBWF shows that it is a promising material for reinforcement in composite application.



**FIGURE 6.** The overlay plot for the percentage improvement of CC, HC, and LC removal of modified SBBWF.

**TABLE 3.** Comparison of the predicted results by RSM and experiment values of CC, HC and LC of treated SBBWF at optimum condition

SBBWF compositional content	CST	ST	Prediction/experiment		Error difference
	%	h	%	%	%
CC	3	18	97.99859	97.6924	0.313428
HC	3	18	44.52456	44.3379	0.420988
LC	3	18	45.71598	45.5817	0.294586

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