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Optimizing delignification and mercerization treatments for cellulose extraction from Semantan bamboo (*Gigantochloascortechinii*) Fiber using response surface modelling (RSM)

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ABSTRACT
The exceptional strength-to-weight ratio, abundance and biodegradability of Semantan bamboo (Gigantochloascortechinii) have made it a popular choice as both a template material for composite manufacturing and an ideal candidate for utilization as an adsorbent material. The objective of this study was to obtain cellulose of excellent quality from bamboo fiber, considering its purity and optical qualities, which are important for various industrial. The bleaching phase, which is crucial for determining the quality of cellulose, was optimised using the response surface methodology. The parameters of $NaClO_2$ concentration (12 – 20% w/w), temperature (60 – 80°C), and duration (3-5 hours) were adjusted to achieve optimal results. The results showed that using high concentrated $NaClO_2$ or prolonging the bleaching period resulted in cellulose oxidation and decreased efficiency. The ideal conditions for bleaching bamboo fiber were determined to be a concentration of 16% (w/w) $NaClO_2$ at a temperature of 70°C for 4 hours. Under these conditions, the amount of cellulose produced was as high as 45.9%. The crystallinity degree yielding results of 57.87% and 64.29% for untreated and treated bamboo fiber, respectively. The effectiveness of the delignification and mercerizastion processes in producing valuable industrial products from bamboo fiber was proven by structural analysis using morphological analysis.

Keywords:*Cellulose optimization, Delignification, Mercerization, Semantan Bamboo, Industrial applications.*

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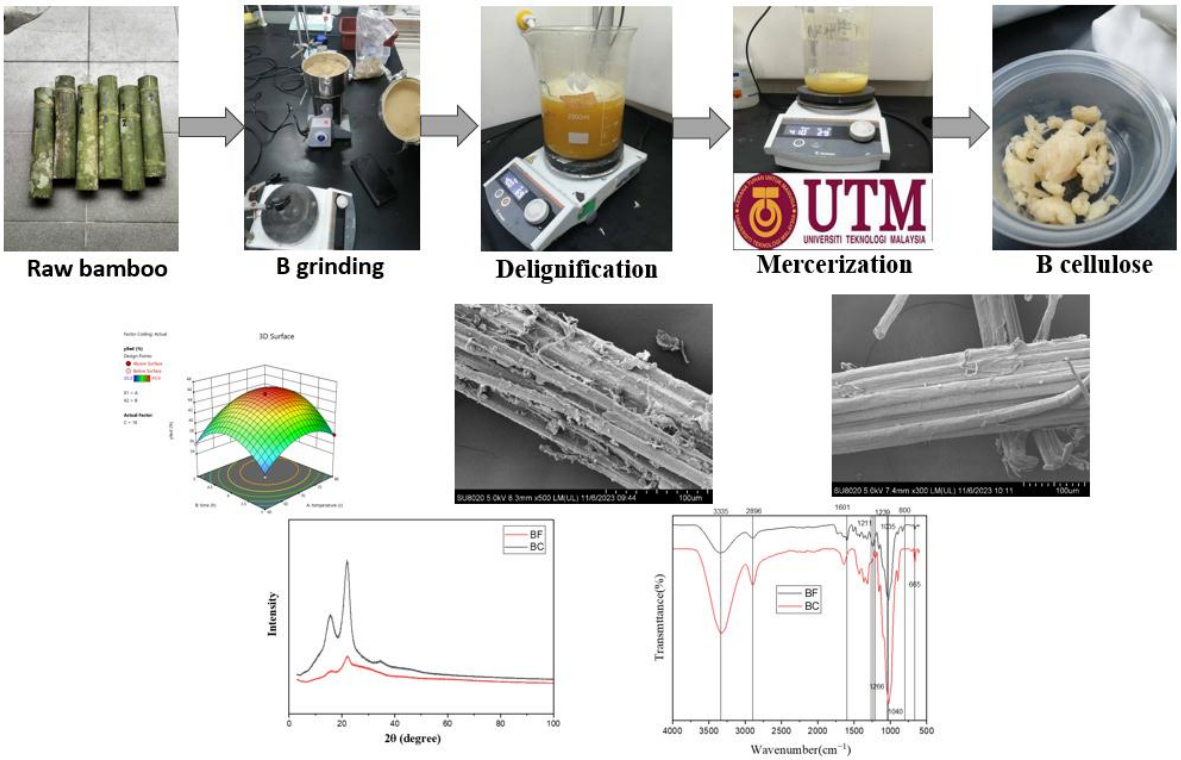
Graphical abstract



Graphical abstract

Highlight

Semantan Bamboo (Gigantochloascortechinii) by Response Surface Modelling (RSM) as a sustainable resource, and potential source of cellulose to treart polluted water from textile factories.

I. Introduction

There has been a notable increase in attention towards environmentally sustainable biomaterials as viable alternatives to synthetic fibres in recent times[1]. Natural lignocellulosic fibres offer a multitude of benefits, such as their low weight, economical nature, remarkable specific characteristics, thermal resilience, environmentallysustainable nature, and biodegradable nature [2]. Due to these attributes, they are attractive for a diverse range of applications in sectors including automotive manufacturing and construction [3]. Annual production of cellulose, the most prevalent natural biopolymer on a global scale, is 7.5× 1010 tonnes[4]. Carboxylic fibres, in their composition, consist of both disordered (amorphous) and highly ordered (crystalline) components [5]. These fibres possess distinctive qualities, including remarkable mechanical strength, a large surface area, and a high aspect ratio (length to diameter). These properties give cellulose fibres considerable commercial potential[6].Semantan bamboo (*Gigantochloascortechinii*), which is frequently encountered in Malaysia, exhibits considerable potential as a cellulose fibre source. Considerable investigation has been dedicated to theenhancement of the characteristics of these bamboo fibres[7]. Delignification and mercerization are two of the most significant treatments that have been investigated in relation to improving fibre cellulose quality[8].The procedure entails the extraction of lignin from the fibres of bamboo. Lignin, an intricate polymer, is responsible for imparting rigidity and hydrophobicity to the fibres[9]. By means of selective lignin degradation, scientists can increase the fibres' overall cellulose content and ductility[10].

Chemical techniques effectively produce cellulose with a high level of purity. A method called bleaching is used. This involves treating the material with $NaClO_2$ in acidic conditions to remove lignin and hemicellulose, as well as partially breaking down the substance. A short bleaching cycle, low bleacher concentration, and low temperature setting are critical for efficient output[11].Bleaching under acidic circumstances has not yet been fully investigated for its ability to optimise lignin and hemicellulose removal, especially for semantan bamboo (*Gigantochloascortechinii*) fiber. To achieve a successful $NaClO_2$ reaction during bleaching, the acidity levels are regulated after the temperature reaches 70°C using eco-friendly acetic acid[12].

Mercerization, also called alkali treatment, is the process of subjecting the natural fiber to the action of fairly concentrated strong base solution (aqueous NaOH or KOH solution), which depend on the type and

concentration of solution, temperature and time of treatment, as well as tension of material, to produce great swelling with resultant changes in the fine structure, morphology, dimensions, and mechanical properties; ASTM: D1695-07[13]. It has been reported that mercerization has four effects on the fiber: (1) it increases the number of possible reaction sites by exposing the surface of cellulose fiber (2) it increases surface roughness, resulting in better mechanical reinforcement; (3) it improves the mechanical behavior, i.e. strength and stiffness, and increases the percentage crystallinity index of alkali-treated fibers; and (4) decreases in the spiral angle, i.e. closer to fiber axis, and increase in molecular orientation [14]. The chemical process of mercerization, which involves the alkaline treatment, is extensively used in the extraction of cellulose from natural fibers and agricultural debris. After the structure of the hydrogen bond network is broken, the remaining lignin is removed through delignification [15]. Nevertheless, the existing body of literature is scant or nonexistent with respect to the impacts of treatment duration, as most of the research has concentrated on different concentrations, following **Table 1**.

Table 1. Reported works on the production of cellulose from bamboo fiber throughdiverse treatment methods.

Fibers	Pre-treatments	Treatments	Cellulose Yield	References
Kenaf	Acid Hydrolysis	H_2SO_4 hydrolysis	54.7%	[16]
Sugarcane bassage	Enzymatic Hydrolysis	Cellulase digestion	82%	[17]
cotton	Ionic Liquid	ionic liquids-based imidazolium	83%	[18]
Eucalyptus waste (*Eucalyptus citriodora*)	Mercerization NaOH	bleaching using hydrogen peroxide	55%	[19]
Thespesia populnea barks	-	alkali treatment	76.42%	[20]
Bamboo	Delignification	Mercerization	45.9%	Current work

An example of a process that can greatly benefit from Response Surface Methodology (RSM) is the extraction of bamboo fibres. Because it is a renewable resource, bamboo has great potential in many different fields. But it's not easy to get thecellulose out of bamboo fibres. The complexities of this process can be thoroughly examined and improved using RSM[21].The main goal is to maximise the yield of cellulose extraction, which is heavily influenced by critical variables like temperature, concentration, duration, and the presence of NaOH and $NaClO_2$. By using RSM, we can examine how these factors interact with one another, which sheds light on how they affect the extraction process as a whole[22]. Utilising RSM, scientists can identify the ideal delignification and mercerization conditions for bamboo fibres, increasing the cellulose yield. Not only does this optimisation improve the extraction process efficiency, but it also helps with sustainable material production by making full use of bamboo resources.

Delignification and mercerization of bamboo using RSM were the goals of this work. To determine how duration, temperature, and chlorite concentration affected cellulose production, the mercerization procedure, an alkaline treatment, was used. The Box-Behnken design (BBD) method was employed in conjunction with Design-Expert software (version 13.0) to analyse experimental data and ascertain the optimal conditions that would yield the greatest quantity of cellulose from BC. To determine the three best conditions, a methodical series of seventeen runs was carried out with the help of statistical modelling software (RSM) to guide the practical lab work. After that, XRD, FESEM, and FTIR were used to characterise these ideal circumstances.

II. Methodology

2.1 Materials

Samantan bamboo (Gigantochloascortechini) was collected from FRIM in Kepong, Malaysia. The chemical reagents used were 80% pure sodium chlorite ($NaClO_2$) from Sigma, 99.8% pure acetic acid (CH_3COOH) from QRëc, and 99% pure sodium hydroxide.

2.2 Extraction of bamboo cellulose

Various techniques are available for isolating cellulose from lignocellulosic biomass sources. In this study, the authors successfully obtained cellulose fibers from bamboo (BF) using the methodologies outlined by Ilyas et al. [23]the **Figure1** show for delignification and mercerization. The quantity of cellulose recovered from BF was significantly influenced by three key factors: the solution temperature, the duration of contact between sodium chlorite ($NaClO_2$) and BF powder, and the concentration of the bleaching solution. To gain a comprehensive understanding of the cellulose extraction procedure, it is advisable to adhere to this protocol[24].

The extraction process began with the addition of 5 grammes of powdered and dried bamboo powder to 200 ml of water and sodium chlorite at three distinct strengths (12, 16, and 20 %). After that, the mixture was

agitated continuously for 3 to 5 hours at temperatures between 60 and 80°C using a magnetic stirrer. Then, the residue of hemicellulose solids was collected after filtering with filter paper.

After that, the hemicellulose solids residue was treated with 200 ml of NaOH 5% solution and left to agitate for two hours. Then, it underwent another round of filtering to extract the cellulose solids residue. Sodium hydroxide was used to bring the pH of the samples to 7.5. After going through another round of filtration, the remaining cellulose particles, which we'll call Bamboo cellulose (BC), were carefully measured, and then dried at 75°C overnight to ensure that the sample was completely devoid of moisture.

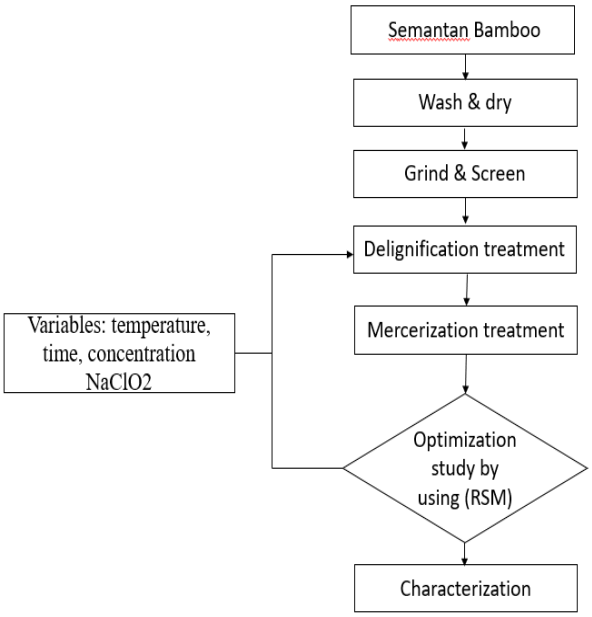


Figure 1. Schematic illustration of BF extraction

2.3 Cellulose extraction yield

Following each phase of processing, the specimens are dried in an oven preheated to 70°C until their mass remains constant. The cellulose yield, expressed as a percentage, is calculated using Equation (1). By employing this methodological approach, the cellulose extraction process **(Figure2)** is subjected to a rigorous and standardised evaluation, which facilitates the accurate assessment of treatment efficacy and the optimisation of cellulose yield from bamboo substrates.

$$\text{Yield}(\%)=\frac{Wf}{Wi}100 \qquad (1)$$

W_i = initial weight, W_f= final weight.

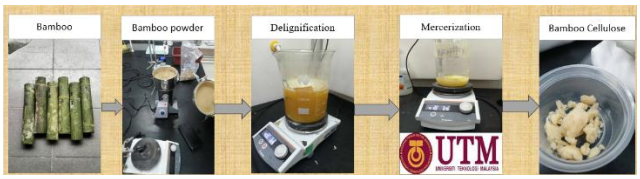


Figure2. The procedure for cellulose extraction from bamboo fiber

2.4 Optimization of bamboo cellulose

2.4.1 Experimental Design from Response Surface Methodology.

The primary data on the chemical quality of cellulose were collected from each treatment, which included water extractives, hemicellulose, cellulose, and lignin. The treatment protocol was created using an optimisation method that utilised Response Surface Methodology (RSM), with experimental data analysed using Design-Expert software (version 13.0) with three components. The optimisation concentrated on the bleaching process's temperature (A), time (B), and $NaClO_2$ concentration (C), which were studied at low, moderate, and high levels, denoted by (-1), (0), and (+1), respectively, as shown in **Table 2**. The experimental design included seventeen experiments, five repeated experiments at centre points, and twelve factorial points, with the goal of discovering the ideal circumstances for the experiments and identifying the maximum yield (Sayed and Khalaf, 2023). The total number of experiments was obtained using a previous study (2).

$$\boldsymbol{N = 2K\,(K - 1) + C_0} \quad (2)$$

Where N is the number of experiments, K is the number of variables (time, temperature, and concentration), 12 experiments with 5 replications, (Co) have been enhanced to evaluate the pure error.

The equation above shows that 17 experiments under various operating circumstances are required. During these trials, five optimal conditions (centre points) were determined to measure potential human error. The temperature ranges from 60°C to 80°C, and the stirring times are 2 hours, 4 hours, and 5 hours, with sodium chlorite concentrations of 12g, 16g, and 20g. Each experiment used 5 grammes of powder.

To characterise the impacts of the three variables, a second-order polynomial regression model was used to fit the quadratic mathematical model for cellulose yield, which is shown in Equation (3) [25].

$$Y = \beta_0 + \beta_1 A + \beta_2 B + \beta_3 C + \beta_{11} A^2 + \beta_{22} B^2 + \beta_{33} C^2 + \beta_{12} AB + \beta_{13} AC + \beta_{23} BC \quad (3)$$

In the equation, the symbol Y stands for the anticipated reaction (cellulose yield). ß0 is the constant, whereas ß1, ß2, and ß3 are the linear coefficients. ß11, ß22, and ß33 stand in for quadratic coefficients, whereas ß12, ß13, and ß23 stand for interaction coefficients. Factors A, B, and C correspond to the variables being investigated. The link between these variables and the outcome was examined using analysis of variance (ANOVA). In addition, p-values were determined for model comparisons, and F values were evaluated to determine the effects of factors on responses.

Table 2.Minimum and maximum levels of the variables**.**

Factor	Variables	Unit	Level -1 0 +1	Output Response
A	Temperature	◦C	60 70 80	Cellulose Y(%)
B	Time	h	3 4 5	
C	concentration	g	12 16 20	

2.6 Characterization of raw bamboo and bamboo cellulose

2.6.1 Moisture content

To examine the moisture content, a set of five samples was prepared with great attention to detail. An oven setting of 105°C was used to heat each sample for a period of 24 hours. Both the pre-heating weight (represented as Mi in grammes) and post-heating weight (represented as M_f in grammes) of the samples were precisely determined. Subsequently, the moisture content was computed utilising these measurements in accordance with the method described in Equation and as suggested by (4) [26,27].

$$Moisture\ content\ (\%) = \frac{M_i - M_F}{M_i} \times 100 \quad (4)$$

where $M_f$is mass of the samples after heating and M_i is mass of the samples before heating.

2.6.2 Chemical composition

According to [28].The chemical composition analysis entailed the utilization of gravimetry to ascertain the quantitative presence of cellulose, hemicellulose, and ligninwere present in the original material on a dry basis. The Semantan bamboo (BF) was soaked in a 5% (w/v) sodium chlorite ($NaClO_2$) solution with a pH of 4 to 5 for 90 minutes at 70°C. The samples were then oven-dried for 24 hours before being chilled in a desiccator to achieve a constant weight. The weight reduction from this treatment revealed lignin composition, while the residual weight reflected holocellulose, which included hemicellulose and cellulose. The holocellulose was rinsed with deionized water before being chemically treated with a 6% (w/v) sodium hydroxide (NaOH) solution at 25°C for 24 hours to remove hemicellulose while retaining cellulose. Following treatment, the samples were oven-dried for 24 hours, chilled in a desiccator, and analysed. The cellulose content was calculated based on the sample's ultimate weight.

2.6.3 Field emission scanning electron microscopy (FESEM)

Field emission scanning electron microscopy (FE-SEM) is a sophisticated technique for capturing detailed microstructure images of materials. In this study, a SEM (Hitachi TM 3000, USA) operating at 5 kV was used. Prior to imaging, the dried samples were coated with a thick layer of gold, approximately 1 to 10 nm. At least five SEM pictures were taken from different areas on each sample[49].

2.6.4 FourierTransform Infrared Spectroscopy (FTIR)

To analyse potential alterations in functional groups that may have occurred because of the extraction process, Fourier transform infrared spectroscopy (FTIR) was utilised at various phases of Semantan bamboo fibre (BF) extraction. The infrared spectra of the substance were acquired using a Thermo Scientific Nicolet iS10 spectrometer. The FTIR spectra spanned the spectral range of 4,000 – 500 cm^{-1}, which facilitated a comprehensive examination of the functional groups that were found in the samples.

2.6.5 X-Ray diffraction (XRD)

The crystallinity of the materials was evaluated employing X-ray particle diffraction (XRD)[29]. The Rigaku D/max 2500 X-ray diffractometer (Rigaku, Tokyo, Japan) used to analyse the X-ray diffraction patterns of untreated and treated semantan bamboo fibre (BF) had a generator voltage of 40 kV, a current of 40 mA, and a goniometer speed of 0.02 (2θ) s^{-1}. The instrument was equipped with CuKα radiation (λ = 0.1541 nm) within the 2θ range of 10° to 40°. Using an empirical procedure [30]the crystallinity index (Xc) of the samples was calculated (Xie et al. 2016),as shown in Equation 5, where I_{002} and I_{am} represent the peak intensities of the crystalline and amorphous regions, respectively.

$$Xc = \frac{I_{002} - I_{am}}{I_{002}} \times 100 \qquad (5)$$

where I_{002} and $I_{am}$are represent the maximum levels of intensity observed in crystalline indicative of the lattice plane and minimum intensity of the two highest peaks of I_{am}, respectively

$$Xc = \frac{I_{002} - I_{am}}{I_{002}} \times 100$$

where I_{002} and $I_{am}$are represent the maximum levels of intensity observed in crystalline indicative of the lattice plane and minimum intensity of the two highest peaks of I_{am}, respectively [32].

III. Result and Discussion

3.1 Extraction and optimization of cellulose

3.1.1Synthesis optimization using RSM

The composition of hemicellulose, cellulose, and lignin in fibrous materials varies greatly according to their source. To extract cellulose from semanatan bamboo fibre (*Gigantochloascortechinii*), 17 experiments were carried out using the RSM design and the parameters given in **Table 3.** The cellulose yield responses from these tests are shown in the same table. Significant factors, such as temperature, reaction time, and $NaClO_2$ concentration, were assessed at three levels. Under these settings, the cellulose yield from BF ranged from 36% to 45.9%. The model's correctness was assessed using a variety of statistical approaches, including ANOVA, lack of fit, residual distribution, overfitting tests, and coefficient of determination (R^2).

The statistical validity and correctness of the fitted model were assessed using ANOVA[33]. with results based on the quadratic model shown in **Table 4.Figure 3 (A,B)** displays the surfaces of response of cellulose yield response on the interaction effects between temperature, time, and concentration; as well as Diagnostic plots of BBD optimization. An astonishingly low p-value of less than 0.0001 confirms the regression model's statistical reliability and demonstrates its outstanding importance. Furthermore, a model F-value of 83.50 indicates the model's relevance, with only a 0.01% probability of such a high F-value being attributable to noise. Significant model terms are those with P-values less than 0.0500, such as A, B, C, AB, BC, A^2, B^2, and C^2.

The lack of fit F-value of 5.31 indicates a 7.03% possibility that such a high Lack of Fit F-value could be caused by noise, indicating poor fit. The predicted R^2 of 0.8791 is comparable to the adjusted R^2 of 0.9789, indicating good model agreement. Furthermore, Adeq Precision reported a signal-to-noise ratio of 22.849, indicating appropriate signal strength. These results support the model equation's ability to describe BF cellulose extraction under a variety of operational situations. Equation (5) represents the response surface model, which may effectively express BF cellulose yield (%).

Table 3. Experimental design matrix of BBD with cellulose yield.

		Factor 1	**Factor 2**	**Factor 3**	**Response 1**
Std	Run	A:temperature	B:time	C:concentration	Yiled
		C	H	G	%
2	1	80	3	16	37.8
17	2	70	4	16	45.9
1	3	60	3	16	35.3
9	4	70	3	12	37.1
8	5	80	4	20	40
11	6	70	3	20	36.8
14	7	70	4	16	45.3
7	8	60	4	20	38.4
4	9	80	5	16	42.1
16	10	70	4	16	45.6
15	11	70	4	16	45.4
12	12	70	5	20	40.8
13	13	70	4	16	45
3	14	60	5	16	36
6	15	80	4	12	40.3

10	16	70	5	12	36.5
5	17	60	4	12	36.1

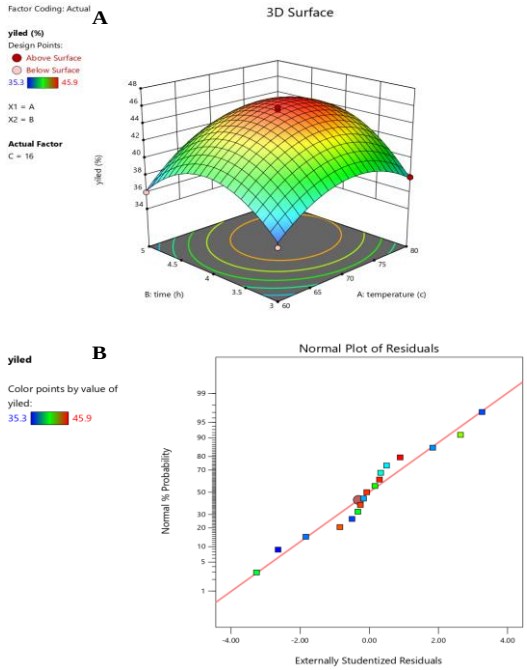


Figure3(A) Surfaces of response of cellulose yield response on the interaction effects between temperature, time, and concentration; **(B)** Diagnostic plots of BBD optimization.

Table 4. Results of the quadratic model using ANOVA.

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	241.77	9	26.86	83.50	< 0.0001	Significant
A-temperature	25.92	1	25.92	80.57	< 0.0001	
B-time	8.82	1	8.82	27.42	0.0012	
C-concentration	4.50	1	4.50	13.99	0.0073	
AB	3.24	1	3.24	10.07	0.0156	
AC	1.69	1	1.69	5.25	0.0556	
BC	5.29	1	5.29	16.44	0.0048	
A^2	47.82	1	47.82	148.64	< 0.0001	
B^2	76.77	1	76.77	238.63	< 0.0001	
C^2	47.82	1	47.82	148.64	< 0.0001	
Residual	2.25	7	0.3217			
Lack of Fit	1.80	3	0.6000	5.31	0.0703	not significant
Pure Error	0.4520	4	0.1130			

In regression analysis, coefficient estimates are used to indicate the expected change in the response variable when there is a one-unit change in the value of a factor while keeping all other factors constant. The intercept in an orthogonal design represents the mean response across all experimental runs.

Within these designs, coefficients represent modifications made to the average value, considering the specific settings of components. When components are orthogonal, variance inflation factors (VIFs) of 1 indicate the absence of multicollinearity, meaning that each factor is independent of the others. Nevertheless, VIFs exceeding 1 suggest the existence of multicollinearity, indicating interdependencies between variables. The strength of this link intensifies as the VIF values grow.

Typically, VIFs that are less than 10 are considered acceptable. However, this threshold may differ based on the context and goals of the investigation.

3.1.2Effect of pretreatment variables on cellulose yield

Figure 2A. shows the quadratic model produced three-dimensional response surface graphs, illustrating the interplay between the response yield and independent process factors. There was notable consistency between anticipated and empirically observed outcomes. Findings revealed that optimal conditions for cellulose production entailed high temperature (70°C) and prolonged pretreatment duration (4 hours), underscoring the importance of meticulous optimization of crucial process variables to achieve desired outcomes.

Repeated testing confirmed the adequacy of the model, with three replications closely aligning with projected values. Maximum cellulose yield (45.9%) was achieved with optimal substrate concentration (20g), pretreatment temperature (70°C), and pretreatment time (4 hours). Statistical validation of validation run data further bolstered the robustness of the findings.

According to [23], delignification and mercerization methods were employed to extract cellulose in this investigation. [34,35] have indicated that $NaClO_2$ can effectively oxidize water-insoluble lignin, generating carboxyl groups and enhancing its water solubility. An ANOVA analysis revealed that time and $NaClO_2$ concentration had greater significance in determining the yield, water-insoluble content (WI), and Kappa number of the extracted cellulose compared to temperature.

3.1.3 Validation of experimental model

Experiment results were utilised for validation purposes in conjunction with response surface methodology (RSM) to determine the optimal conditions for cellulose yield maximisation from SBF. With a cellulose yield of 45.9%, the optimised BF extraction was conducted at 70°C for four hours with a $NaClO_2$ concentration of 16. The observed result, which was near the anticipated value of 49.9%, serves as evidence of the model's dependability.

Emphasising the sufficiency and precision of the selected extraction process parameters is a 95% confidence interval between experimental results and RSM-predicted values. The results of this study validate the reliability of the approach, suggesting that it can be effectively utilised in practical domains.

3.2 Characterization

3.2.1 Moisture content

The efficacy of a natural material as a filler in polymer blends is contingent upon its moisture content to a significant extent. Studies have shown that high amounts of moisture can make bio-composites less stable, slow the growth of pores, and shorten their overall life [36]. In this case, the study showed that the amount of water in the bamboo samples varied, with averages of 23%, 17%, and 26%[37,38]. According to [39], there may be variations in the amount of lignin present. Lignin is known for its ability to keep water away.

In addition, the number of parenchyma cells affects the moisture content of bamboo because they tend to hold on to water better than arterial bundles. As a result, changes in this cellular makeup can cause changes in the amount of water present. It's important to note that the amount of water in bamboo has a big effect on its mechanical power. Higher amounts of moisture can weaken structures, which shows how important it is to control moisture when using bamboo as a composite filler.

3.2.2 Chemical composition

In determining their physical, mechanical, and thermal properties, the chemical composition of natural fibres is crucial. In general, natural fibres comprise cellulose, hemicelluloses, lignin, and ash, although the exact proportions may differ due to variables including the origin of the fibre, the methods used for extraction, and the age of the fibre [40]. Regarding bamboo, its fundamental chemical components consist of cellulose, hemicelluloses, and lignin, which are all intricately interwoven in a multifaceted framework [41]. The cellulose content comparison between bamboo cellulose (BC) and unprocessed bamboo fibre (BF) is presented in **Table 5.** The results indicate that BC contains a considerably higher percentage of cellulose (72.5%) than raw fibre (46%), this result was similar to extracted (crude) cellulose from argan press cake by a procedure involving alkaline treatment with 12 wt% NaOH and 8 wt% sodium sulfate (Na_2SO_4) solution at 80 °C for 90 min.This approach led to the extraction of cellulose with high purity, beyond 71% [42]. Also comparison bleached cellulose fibers from Tó leaf petioles (Calathea lutea) with 3.5% $NaClO_2$, at 30 °C to pH 9.2, followed by 20% NaOH for 1 h, and finally 0.5% $NaClO_2$ for 1 h, all under agitation where was yield 26.25% Bolio-López et al.[43] . It is thought that the removal of extractive components during the delignification and mercerization processes is what caused the increase in cellulose content in BC [27]. These procedures successfully isolated a larger percentage of insoluble compounds. As part of the extraction process, the whitening step gets rid of hemicellulose, silica, soluble mineral salts, and ash isolating a larger percentage of insoluble compounds. Temperature and pH are two things that can change this process. Reactions usually happen when the temperature and pH are high. In the second step, chemical treatment breaks down and removes more parts of the fibre, making it even better.Notably, bamboo's cellulose content tends to diminish as it age [44]. Due to its elevated cellulose content, BC has deemed the most favourable option for the mechanical treatment of nanocellulose in a wet-disc mill.

Table 5. Chemical compositions of BF and BC.

Samples	Cellulose (%)	Hemicellulose (%)	Lignin (%)
BF	46	24.5	29
BC	72.5	17	10.5

3.2.3 Morphological analysis

Chemical and mechanical pretreatments can change the shape and size of fibres' surfaces in big ways. Upon examination of the surface of unprocessed bamboo fibers, an abundance of hemicelluloses, lignin, waxes, oils, and surface impurities becomes apparent. [45]These constituents are characteristic features observable in natural fibers such as hemp and jute. [46,47]The image in **Figure 4** shows raw bamboo fibre, which is made up of bundles of cells that are joined together by lignin, hemicelluloses, and sticky substances [48]. This means that the surface of raw bamboo fibre is usually rough and uneven because it contains noncellulosic materials[49,50]. The FESEM picture of pulp BC shows a fibre surface that is uniform and smooth, which means that noncellulosic materials have been removed. Even though pulping separates fibres from groups, these fibres may still be knotted [51]. Delignification and mercerization, on the other hand, make the fibres smoother and smaller. It can be observed in **Table 6** that the sizes of the raw fibre, cellulose BF, and BC were 110nm to 9.51nm, which is the same as what was found in other bamboo species [52].Due to the removal of lignin and hemicellulose during delignification and mercerization, BC width is smaller than that of the cellulose microfibers discovered by Alemdar and Sain in 2008 [53]This process cuts apart BF bundles, which frees up cellulose microfibrils [54]. This makes the fibres smaller.

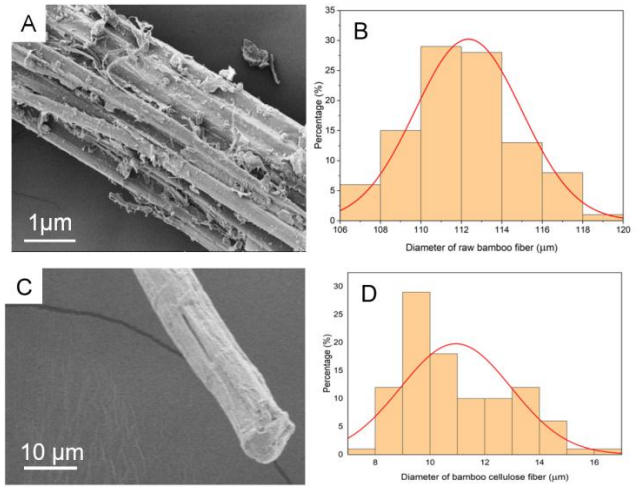


Figure4. FESEM images of raw bamboo (A) and size distribution (B); SEM image of cellulose bamboo (C) with size distribution (D).

Table 6. Diameter of BF and BC.

Samples	Diameter (μm)
BF	111.61
BC	9.51

3.2.4 FTIR analysis

To further investigate the modifications in chemical composition that take place in bamboo fibres after delignification procedures, a comparative analysis of Fourier-transform infrared (FTIR) spectra was performed on untreated bamboo fibres and those that had undergone delignification techniques [55]. The Fourier transform infrared (FTIR) analysis unveiled notable variations in the wave numbers and figures of characteristic peaks, with treated fibres in **Figure 5** exhibiting the most pronounced differences in the range of 1700 cm^{-1} to 600 cm^{-1}[56].

Significantly, absorption peaks were identified at specific locations: 3324 cm^{-1} and 3335 cm^{-1}, which correspond to O-H stretching; and 2895 – 2896 cm^{-1}, which corresponds to CH stretching in the CH_2 and CH_3 groups, respectively. The presence of lignin was indicated by bands between 1601 cm^{-1} and 1211 cm^{-1}, which were associated with skeleton stretching vibrations of aromatic rings, methoxy C-Hdeformations, and lignin bending. In lignin and hemicellulose, the vibrational band at 1239 cm^{-1}suggested the presence of a clove ring

and C-O stretching [57]. Significantly, the elimination of the vibrational band at 800 cm^{-1}, which is attributed to C-H vibrations in derivatives derived from Guaiac after delignification, served to emphasise the effectiveness of the treatment in eliminating lignin. Moreover, alterations were noted in the peaks corresponding to hemicellulose and cellulose constituents, specifically within the range of 1023 $cm^{-1}$to 1035 cm^{-1}. These peaks are suggestive of stretching of the C–O and C–H groups in cellulose and hemicellulose, respectively. Furthermore, marginal alterations were observed in the intensity of the vibrational bands associated with cellulose at 1307 cm^{-1}, 1367 cm^{-1}, 1266 cm^{-1}, and 1157 cm^{-1} after the delignification process [58]. Absorption maxima at 665.85 cm-1 in bamboo and 664.42 cm-1 in extracted cellulose indicate the out-of-plane bending of cellulose C-OH bonds. Moreover, the presence of the 1428.91 cm-1 FTIR absorption band in the extracted cellulose spectrum, commonly referred to as the "crystallinity band" due to its link with sample crystallinity, corresponds to the symmetric vibrating motion of CH_2 groups in cellulose[59].

Molecular recognition of peaks or bands with precise intensity within the range of 1072-1040 cm^{-1} suggested that the fibres had retained cellulose. The results of this study indicate that the bleaching methods successfully eliminated a substantial proportion of lignin and hemicellulose components from the BF [48]

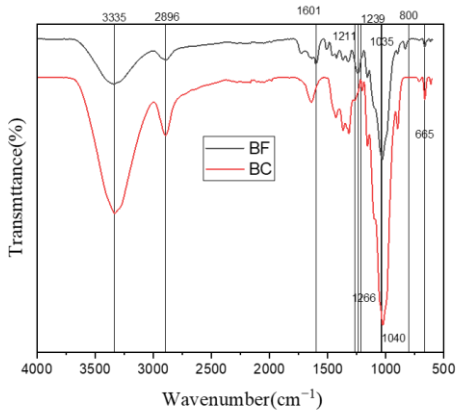


Figure 5. FTIR spectra of raw BF and BC

3.2.5 XRD crystallinity

Individual fibre crystallinity has a substantial influence on material thermal and mechanical properties [27].**Figure6**depicts the X-ray diffraction (XRD) patterns for both untreated and treated bamboo fibres, which show comparable diffraction peaks but with variable intensities. The XRD analysis revealed distinct peaks at 2θ values around 16° and 22.8° in all diffractograms, indicative of the characteristic cellulose form. Conversely, the presence of low peaks at approximately 18° indicated the amorphous region, these peak shifts indicate an increase in the treated fibres' interlayer spacing, which is caused by the disorder induced during the fibre treatment procedure. The crystallinity degree yielding results of 57.87% and 64.29% for untreated and treated bamboo fiber, respectively. These findings are consistent with a previous study, which showed thattreatment with alkali improve crystallinity by eliminating lignin from fibres crystallinity by eliminating lignin from fibres [60,61]. It is worth mentioning that the crystallinity of BF is comparatively lower than that of extracted cellulose. This observation aligns with prior research that has shown an increase in the crystallinity index after the extraction of cellulose from bamboo fiber. The elevated crystallinity index observed in extracted cellulose can be attributed to its heightened exposure after extraction. On the other hand, the lower crystallinity index in materials that have not been treated is mostly due to the presence of lignin and amorphous hemicelluloses [62].

Significantly, the structural integrity of the predominant component of the isolated cellulose is preserved, suggesting that the treatment has no impact on the lattice structure of cellulose. The results of this study highlight the efficacy of the treatment method in augmenting the crystallinity of cellulose obtained from semantan bamboo fiber. As a result, more is known regarding the structural characteristics and possible uses of this material.

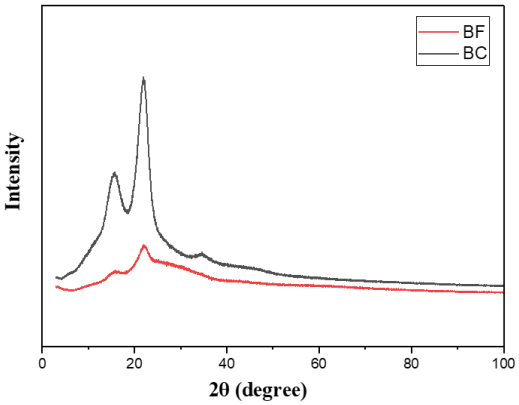


Figure6. XRD patterns of BF and BC

IV. Conclusion

Delignification and mercerization were employed in this investigation to extract cellulose fibres from bamboo fibres (BF). The cellulose extraction efficiency of 45.9%, which was consistent with the predicted values of 49.9%, was achieved under the optimised delignification conditions (temperature: 70°C, time: 4 hours, $NaClO_2$ concentration: 16g). Through studying FTIR spectra, it was found that interventions greatly decreased the amounts of hemicellulose and lignin in BF, which made it easier to extract cellulose. The XRD analysis of bamboo cellulose (BC) revealed a noteworthy increase in crystallinity, reaching 64.29 percent. Further examination by means of SEM analysis revealed substantial modifications in the surface morphology of BF after treatment.

Through delignification and mercerization, selectively removing lignin and hemicellulose from BF makes it possible to extract cellulose efficiently while preventing substantial fibre degradation. Because of the scalable cellulose extraction methods that these insights reveal for BF and similar biomasses, BF is seen as a possible raw material for making nano-cellulose. Except for delignification and mercerization, additional research is necessary to conduct a thorough analysis of the associated costs.In summary, the research emphasises the revolutionary capacity of cellulose extraction from BF, which opens the door to the advancement and innovation of sustainable materials.

Availability of data and materials

All data and materials are available within this research article.

Author Contribution declaration

J.A.S.O. drafted the original manuscript. J.A.S.O., R.A.I., N.N., A.H.N., M.F.M.A. have revised the manuscript. J.A.S.O. conceptualized the idea of the manuscript.

Competing interests

The authors declare that they have no conflict of interest.

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References

[1] T.D. Moshood, G. Nawanir, F. Mahmud, F. Mohamad, M.H. Ahmad, A. AbdulGhani, Sustainability of biodegradable plastics: New problem or solution to solve the global plastic pollution?, Current Research in Green and Sustainable Chemistry 5 (2022). https://doi.org/10.1016/j.crgsc.2022.100273.

[2] A. Karimah, M.R. Ridho, S.S. Munawar, Ismadi, Y. Amin, R. Damayanti, M.A.R. Lubis, A.P. Wulandari, Nurindah, A.H. Iswanto, A. Fudholi, M. Asrofi, E. Saedah, N.H. Sari, B.R. Pratama, W. Fatriasari, D.S. Nawawi, S.M. Rangappa, S. Siengchin, A comprehensive review on natural fibers: Technological and socio-economical aspects, Polymers (Basel) 13 (2021). https://doi.org/10.3390/polym13244280.

[3] A.A. Salih, R. Zulkifli, C.H. Azhari, Tensile properties and microstructure of single-cellulosic bamboo fiber strips after Alkali treatment, Fibers 8 (2020). https://doi.org/10.3390/FIB8050026.

[4] B. Thomas, M.C. Raj, B.K. Athira, H.M. Rubiyah, J. Joy, A. Moores, G.L. Drisko, C. Sanchez, Nanocellulose, a Versatile Green Platform: From Biosources to Materials and Their Applications, Chem Rev 118 (2018) 11575–11625. https://doi.org/10.1021/acs.chemrev.7b00627.

[5] M. Rahimi Kord Sofla, R.J. Brown, T. Tsuzuki, T.J. Rainey, A comparison of cellulose nanocrystals and cellulose nanofibres extracted from bagasse using acid and ball milling methods, Advances in Natural Sciences: Nanoscience and Nanotechnology 7 (2016). https://doi.org/10.1088/2043-6262/7/3/035004.

[6] J. Guo, X. Guo, S. Wang, Y. Yin, Effects of ultrasonic treatment during acid hydrolysis on the yield, particle size and structure of cellulose nanocrystals, CarbohydrPolym 135 (2016) 248–255. https://doi.org/10.1016/j.carbpol.2015.08.068.

[7] N. Husna, M. Hassan, S. Muhammed, R. Ibrahim, Properties of Gigantochloascortechinii Paper Enhancement by Beating Revolution, J. Trop. Resour. Sustain. Sci 2 (2014) 59–67.

[8] R.A. Ilyas, S.M. Sapuan, M.R. Ishak, E.S. Zainudin, Delignification of palm fibre, 2017.

[9] Verma, Hashmi, Mili, et al., Extraction and applications of lignin from bamboo: a critical review, European Journal of Wood and Wood Products 79 (2021) 1341–1357.

[10] E. Małachowska, M. Dubowik, P. Boruszewski, J. Łojewska, P. Przybysz, Influence of lignin content in cellulose pulp on paper durability, Sci Rep 10 (2020). https://doi.org/10.1038/s41598-020-77101-2.

[11] S. Susi, M. Ainuri, W. Wagiman, M.A.F. Falah, High-Yield Alpha-Cellulose from Oil Palm Empty Fruit Bunches by Optimizing Thermochemical Delignification Processes for Use as Microcrystalline Cellulose, Int J Biomater 2023 (2023). https://doi.org/10.1155/2023/9169431.

[12] S. Susi, M. Ainuri, W. Wagiman, M.A.F. Falah, High-Yield Alpha-Cellulose from Oil Palm Empty Fruit Bunches by Optimizing Thermochemical Delignification Processes for Use as Microcrystalline Cellulose, Int J Biomater 2023 (2023). https://doi.org/10.1155/2023/9169431.

[13] H.-M. Ng, L.T. Sin, T.-T. Tee, S.-T. Bee, D. Hui, C.-Y. Low, A.R. Rahmat, Extraction of cellulose nanocrystals from plant sources for application as reinforcing agent in polymers, Compos B Eng 75 (2015) 176–200. https://doi.org/10.1016/j.compositesb.2015.01.008.

[14] R.A. Ilyas, S.M. Sapuan, M.R. Ishak, E.S.Zainudin, Effect of delignification on the physical, thermal, chemical, and structural properties of sugar palm fibre, Bioresources 12 (2017) 8734–8754. https://doi.org/10.15376/biores.12.4.8734-8754.

[15] S.M. Mohomane, L.Z. Linganiso, T. Buthelezi, T.E. Motaung, EFFECT OF EXTRACTION PERIOD ON PROPERTIES OF SUGARCANE BAGASSE AND SOFTWOOD CHIPS CELLULOSE, 2017.

[16] V.G. Firsty, J.Y. Jeong, Y.M. Gu, J.H. Lee, S.-J. Shin, High Cellulose Purity by Acid Hydrolysis Pretreatment on Kenaf Outer Bast, Applied Sciences 13 (2022) 334. https://doi.org/10.3390/app13010334.

[17] M. Brienzo, L. Tyhoda, Y. Benjamin, J. Görgens, Relationship between physicochemical properties and enzymatic hydrolysis of sugarcane bagasse varieties for bioethanol production, N Biotechnol 32 (2015) 253–262. https://doi.org/10.1016/j.nbt.2014.12.007.

[18] K. Aghmih, A. Bouftou, M. El Bouchti, A. Boukhriss, S. Gmouh, S. Majid, Synthesis and application of functionalized ionic liquids-based imidazolium as solvent for cotton fibre cellulose dissolution, Cellulose 30 (2023) 1467–1481. https://doi.org/10.1007/s10570-022-04974-z.

[19] H. Zhang, S. Wu, Generation of lignin and enzymatically digestible cellulose from ethanol-based organosolv pretreatment of sugarcane bagasse, Cellulose 22 (2015) 2409–2418. https://doi.org/10.1007/s10570-015-0678-z.

[20] M. Kathirselvam, A. Kumaravel, V.P. Arthanarieswaran, S.S. Saravanakumar, Characterization of cellulose fibers in Thespesia populnea barks: Influence of alkali treatment, CarbohydrPolym 217 (2019) 178–189. https://doi.org/10.1016/j.carbpol.2019.04.063.

[21] A.S. Norfarhana, R.A. Ilyas, N. Ngadi, M. Hafiz Dzarfan Othman, Optimization of ionic liquid pretreatment of sugar palm fiber for cellulose extraction, J Mol Liq 398 (2024) 124256. https://doi.org/10.1016/j.molliq.2024.124256.

[22] S. Lamidi, N. Olaleye, Y. Bankole, A. Obalola, E. Aribike, I. Adigun, Applications of Response Surface Methodology (RSM) in Product Design, Development, and Process Optimization, in: Response Surface Methodology - Research Advances and Applications, IntechOpen, 2023. https://doi.org/10.5772/intechopen.106763.

[23] R. Ilyas, S. Sapuan, M. Atikah, M. Asyraf, S.A. Rafiqah, H. Aisyah, N.M. Nurazzi, M. Norrrahim, Effect of hydrolysis time on the morphological, physical, chemical, and thermal behavior of sugar palm nanocrystalline cellulose (Arenga pinnata (Wurmb.) Merr), Textile Research Journal 91 (2021) 152–167. https://doi.org/10.1177/0040517520932393.

[24] D.T. Ebissa, T. Tesfaye, D. Worku, D. Wood, Characterization and optimization of alkali-treated yushaniaalpina bamboo fiber properties: case study of ethiopia species, SN Appl Sci 4 (2022). https://doi.org/10.1007/s42452-022-04965-6.

[25] U. Qasim, S. Rafiq, F. Jamil, A. Ahmed, T. Ali, J. Kers, M.S. Khurram, M. Hussain, A. Inayat, Y.-K. Park, Processing of lignocellulose in ionic liquids: A cleaner and sustainable approach, J Clean Prod 323 (2021) 129189. https://doi.org/10.1016/j.jclepro.2021.129189.

[26] OO Antia, W Olosunde, A Offiong, Determination of Optimum Moisture Content of Palm Nut Cracking for Efficient Production of Whole Kernel, 2014.

[27] R.A. Ilyas, S.M. Sapuan, M.R. Ishak, E.S. Zainudin, Delignification of palm fibre, 2017.

[28] T. Arisyah, T. Yasim-Anuar, H. Ariffin, M. Nor, F. Norrrahim, M.A. Hassan, Spinnability of cellulose, 2017.

[29] Y. Kan, H. Kan, Y. Bai, S. Zhang, Z. Gao, Effective and environmentally safe self-antimildew strategy to simultaneously improve the mildew and water resistances of soybean flour-based adhesives, J Clean Prod 392 (2023) 136319. https://doi.org/10.1016/j.jclepro.2023.136319.

[30] L. Segal, J.J. Creely, A.E. Martin, C.M. Conrad, An Empirical Method for Estimating the Degree of Crystallinity of Native Cellulose Using the X-Ray Diffractometer, Textile Research Journal29 (1959) 786–794. https://doi.org/10.1177/004051755902901003.

[31] J. Xie, C. Hse, C.F. De Hoop, T. Hu, J. Qi, T.F. Shupe, Isolation and characterization of cellulose nanofibers from bamboo using microwave liquefaction combined with chemical treatment and ultrasonication, CarbohydrPolym 151 (2016) 725–734. https://doi.org/10.1016/j.carbpol.2016.06.011.

[32] X. Han, J. Wang, J. Wang, L. Ding, K. Zhang, J. Han, S. Jiang, Micro- and nano-fibrils of manau rattan and solvent-exchange-induced high-haze transparent holocellulose nanofibril film, CarbohydrPolym 298 (2022) 120075. https://doi.org/10.1016/j.carbpol.2022.120075.

[33] A.Y. Melikoğlu, S.E. Bilek, S. Cesur, Optimum alkaline treatment parameters for the extraction of cellulose and production of cellulose nanocrystals from apple pomace, CarbohydrPolym 215 (2019) 330–337. https://doi.org/10.1016/j.carbpol.2019.03.103.

[34] S. Susi, M. Ainuri, W. Wagiman, M.A.F. Falah, High-Yield Alpha-Cellulose from Oil Palm Empty Fruit Bunches by Optimizing Thermochemical Delignification Processes for Use as Microcrystalline Cellulose, Int J Biomater 2023 (2023) 1–15. https://doi.org/10.1155/2023/9169431.

[35] S. Meisam, S. Morteza, A. Reza, S. Ghorban, Optimization of a Bleaching Process for the Conversion of Sugar Beet Pulp Alpha-Cellulose with a High Whiteness Index and Purity, 2023.

[36] R. Jumaidin, N.A. Diah, R.A. Ilyas, R.H. Alamjuri, F.A.M. Yusof, Processing and Characterisation of Banana Leaf Fibre Reinforced Thermoplastic Cassava Starch Composites, Polymers (Basel) 13 (2021) 1420. https://doi.org/10.3390/polym13091420.

[37] R. Anokye, R.M. Kalong, E.S. Bakar, J. Ratnasingam, M. Jawaid, K. Awang, Variations in Moisture Content Affect the Shrinkage of Gigantochloascortechinii and Bambusa vulgaris at Different Heights of the Bamboo Culm, Bioresources 9 (2014). https://doi.org/10.15376/biores.9.4.7484-7493.

[38] M. Sharma, C.L. Sharma, D. Laishram, Variation in anatomical and physical properties of some Schizostachyum species of Manipur, India, Journal of the Indian Academy of Wood Science 14 (2017) 79–90. https://doi.org/10.1007/s13196-017-0191-0.

[39] R.M. Rowell, Handbook of wood chemistry and wood composites, Taylor & Francis, 2013.

[40] A. Karimah, M.R. Ridho, S.S. Munawar, D.S. Adi, Ismadi, R. Damayanti, B. Subiyanto, W. Fatriasari, A. Fudholi, A review on natural fibers for development of eco-friendly bio-composite: characteristics, and utilizations, Journal of Materials Research and Technology 13 (2021) 2442–2458. https://doi.org/10.1016/j.jmrt.2021.06.014.

[41] M.A. Azeez, J.I. Orege, Bamboo, Its Chemical Modification and Products, in: Bamboo - Current and Future Prospects, InTech, 2018. https://doi.org/10.5772/intechopen.76359.

[42] Y. Hu, O. Hamed, R. Salghi, N. Abidi, S. Jodeh, R. Hattb, EXTRACTION AND CHARACTERIZATION OF CELLULOSE FROM AGRICULTURAL WASTE ARGAN PRESS CAKE, 2017.

[43] G.I. Bolio-López, G. Cadenas-Madrigal, L. Veleva, R. Falconi, P. De La Cruz-Burelo, M. Mateo Hernández-Villegas, L. Pelayo-Muñoz, EXTRACTION OF CELLULOSE FIBERS FROM TÓ LEAF PETIOLES (Calathea lutea) AND CHARACTERIZATION, 2015. www.ijiset.com.

[44] L. Wang, Evaluation of Properties of Natural Bamboo Fiber for Application in Summer Textiles, Journal of Fiber Bioengineering and Informatics 3 (2010) 94–99. https://doi.org/10.3993/jfbi09201006.

[45] Z.H. Kamaruddin, R. Jumaidin, R.A. Ilyas, M.Z. Selamat, R.H. Alamjuri, F.A. Md Yusof, Influence of Alkali Treatment on the Mechanical, Thermal, Water Absorption, and Biodegradation Properties of Cymbopogancitratus Fiber-Reinforced, Thermoplastic Cassava Starch–Palm Wax Composites, Polymers (Basel) 14 (2022). https://doi.org/10.3390/polym14142769.

[46] D. Verma, K.L. Goh, Effect of mercerization/alkali surface treatment of natural fibres and their utilization in polymer composites: Mechanical and morphological studies, Journal of Composites Science 5 (2021). https://doi.org/10.3390/jcs5070175.

[47] K.J. Wong, B.F. Yousif, K.O. Low, The effects of alkali treatment on the interfacial adhesion of bamboo fibres, Proceedings of the Institution of Mechanical Engineers, Part L: Journal of Materials: Design and Applications 224 (2010) 139–148. https://doi.org/10.1243/14644207JMDA304.

[48] W. Klunklin, S. Hinmo, P. Thipchai, P. Rachtanapun, Effect of Bleaching Processes on Physicochemical and Functional Properties of Cellulose and Carboxymethyl Cellulose from Young and Mature Coconut Coir, Polymers (Basel) 15 (2023) 3376. https://doi.org/10.3390/polym15163376.

[49] N.A. Sri Aprilia, M. Asniza, F.A.T. Owolabi, S. Rizal, M.I. Syakir, M.T. Paridah, U.S. Uthaya Kumar, R.C.L. Nasrullah, M.K. Haafiz, H.P.S. Abdul Khalil, Role of dispersion time on the properties of enzymatic-treated bamboo cellulose nanofibers, Mater Res Express 5 (2018) 105014. https://doi.org/10.1088/2053-1591/aadaca.

[50] C.K. Saurabh, A. Mustapha, M.Mohd. Masri, A.F. Owolabi, M.I. Syakir, R. Dungani, M.T. Paridah, M. Jawaid, H.P.S. Abdul Khalil, Isolation and Characterization of Cellulose Nanofibers from Gigantochloascortechinii as a Reinforcement Material, J Nanomater 2016 (2016) 1–8. https://doi.org/10.1155/2016/4024527.

[51] L.A. Worku, A. Bachheti, R.K. Bachheti, C.E. Rodrigues Reis, A.K. Chandel, Agricultural Residues as Raw Materials for Pulp and Paper Production: Overview and Applications on Membrane Fabrication, Membranes (Basel) 13 (2023) 228. https://doi.org/10.3390/membranes13020228.

[52] J.-H. Jang, N. Hayashi, S.-Y. Han, C.-W. Park, F. Febrianto, S.-H. Lee, N.-H. Kim, Changes in the Dimensions of Lignocellulose Nanofibrils with Different Lignin Contents by Enzymatic Hydrolysis, Polymers (Basel) 12 (2020) 2201. https://doi.org/10.3390/polym12102201.

[53] A. Alemdar, M. Sain, Isolation and characterization of nanofibers from agricultural residues – Wheat straw and soy hulls, BioresourTechnol 99 (2008) 1664–1671. https://doi.org/10.1016/j.biortech.2007.04.029.

[54] B. Song, S. Zhao, W. Shen, C. Collings, S.-Y. Ding, Direct Measurement of Plant Cellulose Microfibril and Bundles in Native Cell Walls, Front Plant Sci 11 (2020). https://doi.org/10.3389/fpls.2020.00479.

[55] Y. Bai, W. Wang, Y. Zhang, X. Wang, X. Wang, J. Shi, Effects of Different Delignification and Drying Methods on Fiber Properties of Moso Bamboo, Polymers (Basel) 14 (2022) 5464. https://doi.org/10.3390/polym14245464.

[56] E. Galiwango, N.S. Abdel Rahman, A.H. Al-Marzouqi, M.M. Abu-Omar, A.A. Khaleel, Isolation and characterization of cellulose and α-cellulose from date palm biomass waste, Heliyon 5 (2019) e02937. https://doi.org/10.1016/j.heliyon.2019.e02937.

[57] J. Liu, Q. Zhang, F. Ma, S. Zhang, Q. Zhou, A. Huang, Three-step identification of infrared spectra of similar tree species to Pterocarpus santalinus covered with beeswax, J Mol Struct 1218 (2020) 128484. https://doi.org/10.1016/j.molstruc.2020.128484.

[58] J. Zhuang, M. Li, Y. Pu, A. Ragauskas, C. Yoo, Observation of Potential Contaminants in Processed Biomass Using Fourier Transform Infrared Spectroscopy, Applied Sciences 10 (2020) 4345. https://doi.org/10.3390/app10124345.

[59] X. Li, Y. Wei, J. Xu, N. Xu, Y. He, Quantitative visualization of lignocellulose components in transverse sections of moso bamboo based on FTIR macro- and micro-spectroscopy coupled with chemometrics, Biotechnol Biofuels 11 (2018) 263. https://doi.org/10.1186/s13068-018-1251-4.

[60] H. Amirulhakim, A.L. Juwono, S. Roseno, Isolation and characterization of Cellulose Nanofiber from Subang Pineapple Leaf Fiber waste produced using Ultrafine Grinding method, IOP Conf Ser Mater Sci Eng 1098 (2021) 062067. https://doi.org/10.1088/1757-899X/1098/6/062067.

[61] L.A. Worku, R.K. Bachheti, M.G. Tadesse, Isolation and Characterization of Natural Cellulose from Oxytenantheraabyssinica (Lowland Ethiopian Bamboo) Using Alkali Peroxide Bleaching Stages Followed by Aqueous Chlorite in Buffer Solution, Int J Polym Sci 2022 (2022) 1–15. https://doi.org/10.1155/2022/5155552.

[62] Q. Lin, Y. Huang, W. Yu, Effects of extraction methods on morphology, structure and properties of bamboo cellulose, Ind Crops Prod 169 (2021) 113640. https://doi.org/10.1016/j.indcrop.2021.113640.