

Optimization and Characterization of Cellulose Extracted from Sugarcane Bagasse

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Abstract- *The surplus in the amount of agricultural wastes leftover on the Egyptian farmlands after each harvest season, along with the presidential initiative and the current strategic vision 2030 towards sustainable development, are the main challenge and strong ambition to conduct this research study. This study evaluates the extraction of sugarcane bagasse (SCB) celluloses with alkali treatment, aided by an optimization for the obtained results by using the response surface methodology (RSM) as a statistical method for modeling and analyzing a process. Seventeen experimental runs were executed to study the influence of various process variables, such as extraction time (1-4 hrs), temperature (50-90 °C) and the concentration of NaOH (2-10 %) on the response (yield) of extracted cellulose. It was estimated that the optimum operating conditions for extracting the high yield (0.5871 g of cellulose/g of bagasse) of cellulose were obtained at time of (2.4 hrs), temperature of (60 °C) and NaOH concentration of (9.8 %). It was also evident from these experiments that the suitable coefficient of determination ($R^2=0.8772$), which is higher than 75 % showed that the quadratic polynomial model is significant and sufficient to represent the actual relationship between the response and significant variable. The extracted celluloses were characterized by Fourier transmission infrared spectroscopy (FTIR) and X-ray diffraction (XRD) analysis. The increase in crystallinity from 31.76 percent for the raw bagasse fibres to 51.13 percent for the extracted cellulose, as observed from the XRD analysis was found to be in complete agreement with the findings of FTIR analysis.*

Key words- *Agricultural Wastes, Cellulose, Sugarcane Bagasse, RSM, Crystallinity.*

I. INTRODUCTION

Green fillers, which are produced from industrial by-products and other waste materials with potential for recyclability, have recently gotten a lot of attention. Economic benefits should be realized through adding value to trash, lowering disposal costs, and achieving favourable environmental outcomes by reducing the amount of materials dumped in landfills. There is an exciting innovation opportunity to develop these alternative materials to function as replacement or supplementary fillers in a diversity of industrial products [1, 2].

Agricultural waste estimates are scarce, although it is widely assumed that it contributes an outsized share of total waste products within the developed world. Increased quantities of agricultural crop wastes and agro industrial by-products have naturally emerged from increased agricultural productivity. Globally, the rapid increase in agricultural

waste amounts has motivated many researchers to compile their studies toward using renewable natural fiber reinforced materials as a desire regarding eco lawn logical footprint and also to reduce reliance on petroleum. Natural fiber-reinforced materials offer target environmental, technical, and economic advantages over traditional ones. These numerous distinguishing advantages, such as reduced dependence on non-renewable energy/ material sources, fewer pollutants, and greenhouse emissions, as well as low cost, high toughness, low density, good specific strength properties, enhanced energy recovery, CO₂ neutral when burned and biodegradability [3]. Nowadays, the potent desire for environment-friendly materials has encouraged the development of industry and the use of biopolymer for various applications [4, 5].

Lignocellulosic biomass (cellulose, hemicellulose, and lignin) distribution varies considerably among the distinctive layers of plant cell walls [6]. Cellulose is one of the most abundant, natural, renewable, and biodegradable polymers [7, 8], that is found in almost pure form in cotton fiber at a concentration of 98 %. Cellulose has been utilized as a raw material for approximately 150 years, and it is known to be insoluble in water because it has a long chain and high molecular weight. Cellulose is the main constituent of biomass, forming approximately 40 % to 45 % of the dry substance in most lignocellulosic materials. The crystalline cellulose is a linear, unbranched homopolysaccharide material composed of anhydroglucose units, which are linked together by β -1,4-glycosidic bonds. This is confirmed by the presence of three hydroxyl groups with different reactivities in the cellulose structure. The ability of these hydroxyl groups to form hydrogen bonds is playing a crucial role in the compactness of the structure and the physical qualities of this crystalline material. Cellulose is found in amorphous innovative plant fibres, which are said to be in the crystalline phase through intermolecular and intramolecular hydrogen bonds. It has a good mechanical properties, low density, and biodegradable. Cellulose is found as the major constituent of plant cell wall, including lignocellulosic material such as wood, cotton, rice straws and SCB [8-11]. In this perspective, an intensive study has been dedicated to cellulose.

Every year, there are massive quantities of SCB wastes left after the crushing process of sugarcane used for juice extraction and in alcohol industry. SCB is used as a fuel for the sugar mills in the process of sugar production, as well other industries are using SCB as a raw material for electric generation, fermented products, paper production and has been developed for biomass [9]. SCB contains 40-50 % cellulose, much of which is in the crystalline structure. Another component in SCB is hemicellulose as much as 25-35 %, which is an amorphous polymer and mainly composed of xylose, arabinose, galactose, and mannose. The rest is mostly lignin with about 18-24 % [9, 12]. Bagasse is made up of two layers: a stiff fibrous substance called rind and a soft layer called pith [13]. Small fibres and the majority of the sucrose are found in the pith, but the rind contains longer and finer fibres that are scattered randomly throughout the stem and held together by lignin and hemicellulose. The filaments have been shown to be frequently close to the inner wall of the rind particle [14, 15]. SCB may be a by-product that is available in many countries and is usually used as fuel or disposed of for incineration. It has long served as a versatile raw material for the manufacture of artificial cellulose-based products as well as several stable cellulose derivatives used in a variety of industrial and home applications [16].

Recently, many researchers had focused their studies on the potential importance of cellulose derived from solid agricultural wastes. However, our country has limited investigations on the beneficial effect of lignocellulosic fibres extracted from agricultural waste, particularly SCB on a diversity of advanced high engineering applications. The treatment and safe disposal of bagasse is one of the most significant challenges in waste agricultural management. Without enough regard to the environmental effects, the significant amount of sludge generated every year inevitably leads to a material with a high pollution potential. Additionally, there is a requirement to transfer new non-traditional technology into our developed country. In this respect, cellulose extraction from SCB has been the topic of in-depth research.

Meanwhile, the ambitious strategic vision for achieving an economical sustainable development was the principal motivation for an enlightened undergraduate researching team to implement this research for the extraction and characterization of cellulose from bagasse. The process of pure cellulose isolation has become the subject of extensive research work for many decades due to the cell wall structure complexity. Steam explosion, solvent extraction, alkaline treatment, and organosolv pre-treatment are some of the well-known methods used for pre-treating lignocellulosic materials for extracting cellulose. The disruption of hydrogen bonding within the network structure could be a significant change caused by alkaline treatment [14, 17-19].

This study aimed to extract cellulose from SCB by using alkaline treatment or sometimes called mercerization method [20], to investigate the effect of varying the different effective factors (i.e., time, temperature, and concentration

of the used alkali) on the yield of the obtained cellulose. This practical lab work was performed through the directive aid from statistical modelling software (RSM) program, which enables the co-workers from executing a systematic sequence for seventeen runs to obtain the five optimum ones that have been characterized by using an FTIR and XRD analytical techniques.

2. MATERIALS AND METHODS

2.1 Materials

Cellulose could be successfully derived from many lignocellulosic biomass materials such as cotton, wool, rice husk, and SCB. In this study, the investigated cellulose was extracted from the raw SCB fibres collected from local mills in (Minea El Amh, Ash Sharqia Governorate, Egypt). These fibres were carefully prepared and treated to extract the maximum yield of cellulose, as mentioned in the next section. A selection of analytical grade chemicals used in the process of cellulose extraction such as sodium hydroxide and hydrochloric acid were all supplied from Alpha Chemika and Medical Lab Supplies.

2.2 Methodology

2.2.1 Desilication of SCB

Raw bagasse contains a significant amount of sand, picked up from the harvesting grounds. Bagasse should be thoroughly washed with plenty of water for 15 minutes, this step was repeated to get rid of high silica content and any dirt present. The wet bagasse was left to dry by exposure to the sunlight for 5 days followed by drying in an oven for 24 hours at 105 °C. Screening is performed to allow the silica to fall through the mesh. The desilicated bagasse is then subjected to the next step called milling of sugarcane.

Fig. 1 Retsch SK 100 laboratory mill (grinding) machine.



2.2.2 Milling of SCB

The dried desilicated bagasse contains a mixed content from both inner (pith) and outer (rind) bagasse fibres. These fibres are well ground by using a deluxe-standard Retsch SK 100 laboratory mill operating with a rotor speed of 2850 rpm at 50 HZ, and the size of the metal sieves used inside the grinding machine is 1.0 mm and 0.5 mm, as shown in Fig. 1, to obtain a ground bagasse fiber with size less than 500 microns. The milled bagasse contains a significant amount of pith, which is completely removed in the following step.

2.2.3 Depithing of SCB

This pith removal is achieved by hammer-milling and screening to remove the pith as “fines.” The centrifugal force of the spinning drum allows adequate separation of the fine pith from the bagasse fibres. Successfully removing the pith is necessary to avoid wastage of chemicals during the forthcoming chemical treatment. The dried depithed milled bagasse fibres were stored in plastic polyethylene bags for further treatment. Bagasse fibres (before grinding) and bagasse powder (after grinding) are seen in Fig. 2.

2.2.4 Extraction of Cellulose

Several methods can be employed to extract cellulose from lignocellulosic biomass materials. Herein, this study has focused on using an alkali pretreatment method. The temperature of the solution, duration time of contact between the sodium hydroxide solution (NaOH) and the SCB powder, and the concentration of alkali (NaOH) solution are considered to be the most three significant parameters that can potentially affect the yielded cellulose extracted from SCB. The following procedure can clearly explain the employed process of cellulose extraction:

- 1- Three different concentrations of sodium hydroxide (0.5, 1.5, 2.5 N) were prepared.
- 2- Hydrochloric acid (HCl) with concentrations (0.2 %, 0.6 %, and 1 %) necessary for neutralization was prepared.
- 3- 200 ml of NaOH solution was added to 10 grams of the dried milled (depithed) bagasse powder.
- 4- A magnetic stirrer was used to allow good mixing at an adapted temperature ranging from (50-90 °C) at a constant stirring rate, and at a different constant time ranging from (1-4 hr).
- 5- The alkaline treated samples were left to cool down at room temperature.
- 6- HCl was then added to adjust a pH range for the prepared samples from (8-8.5)
- 7- The sample was filtered using filter paper and the cellulose solids residue was collected.
- 8- The cellulose solids residue was left in the dryer for 12 hours at 105 °C to remove moisture until reaching a constant weight.



(a) Before grinding (b) After grinding
Fig. 2 Bagasse before (a) and after (b) grinding.

2.2.5. Experimental Design and Optimization

The design expert software was used to obtain the optimal process conditions. These optimum conditions will be applied, to obtain the maximum yield of extracted

cellulose. The three main factors, which are found to significantly affect the yield of extracted cellulose, are the temperature, time of stirring, and concentration of sodium hydroxide. Box-Behnken design with the three optimal processing independent variables was used to calculate the ideal conditions for extracting cellulose from SCB. The ranges of the actual values for these selected variables and their respective coded levels, were as shown in Table 1. The experimental design comprising a total of seventeen experiments with 5 repeating experiments at center points and 12 factorial points were performed, in order to determine the optimum conditions applied for the experiments and to identify the maximum yield [21]. The total number of experiments was obtained from “(1)”.

$$N = 2K(K - 1) + C_0 \quad (1)$$

Where:

N is the number of experiments.

K is the number of variables (time, temperature, and concentration).

There are 12 experiments with 5 replications, (Co) that have been enhanced to evaluate the pure error.

Table 1. Coded and actual values of cellulose extraction

Variable	Symbol	Coded and actual levels		
		-1	0	+1
Temperature	X1	50	70	90
Time	X2	1	2.5	4
Concentration	X3	2	6	10

It is shown from the above equation that 17 runs of experiments at different operating conditions are required to be performed, and among these 17 experiments there are five optimum experiments derived at the same conditions (center point) to know the human error, as shown in table 2. The three variable temperature values are (50 °C, 70 °C, and 90 °C), the time of stirring is (1 hr, 2.5 hr, and 4 hr) and the values of sodium hydroxide concentration are 2 % (0.5 N), 6 % (1.5 N) and 10 % (2.5 N). The amount of bagasse powder used in every experiment was 10 gm.

Table 2. Actual values of experiments

Run	Temperature (°C)	Time (hr)	Concentration (%)
1	50	2.5	2
2	70	1	10
3	90	2.5	10
4	70	2.5	6
5	90	1	6
6	50	2.5	10
7	50	1	6
8	70	2.5	6
9	50	4	6
10	70	2.5	6
11	70	1	2
12	70	4	10
13	90	2.5	2
14	70	2.5	6
15	70	2.5	6
16	90	4	6

2.2.6. Characterization of the Extracted Cellulose

The dried extracted cellulose that was successfully obtained at the optimum conditions via the above-mentioned procedures was characterized, by using the following analytical (physical and material) characterization techniques.

2.2.6.1 Infrared Spectroscopy (FTIR)

The raw untreated bagasse together with the extracted cellulose were dried and stored, to be examined by FTIR analysis. The analysis was performed in the 4000 – 400 cm⁻¹ range to determine the chemical composition of bagasse and the respective extracted cellulose, by using the FTIR model, class 1 Laser product IEC/EN 60825-1/A2:2001 Avatar series (USA) in The Egyptian Academy for Engineering and Advanced Technology. The resulting spectrum depicts molecular absorption and transmission, assigning molecular fingerprint of the sample.

2.2.6.2 X-ray Diffraction (XRD)

X-ray diffraction (XRD) is an analytical technique used to determine whether a sample contains crystalline or amorphous phases of a wide variety of materials, most commonly for mineralogical examination and material identification. Powder diffraction data are primarily generated from crystallography physics-described atomic and molecular configurations. The X-ray diffraction technique relies on the constructive interference of monochromatic X-rays with crystalline material. The mineralogical composition is evaluated in the Science and Technology Center of Excellence using an X-ray diffraction Bruker D8 advanced computerized X-ray Diffractometer equipment with monochromatic Cu K α radiation that runs at 40KV and 40mA.

3. RESULTS AND DISCUSSION

In this section, the quality and characterization of the raw bagasse and the extracted cellulose are well demonstrated through a set of systematic experimental techniques.

3.1 Cellulose Extraction

The response surface methodology was used to investigate the individual and interaction effect of the three-factor on cellulose extraction from SCB. Based on the obtained statistical results, the effects of experimental factors on cellulose extraction and their corresponding three-dimensional response surface plots were shown in Figs. 3-5. The NaOH concentration, time and temperature have significant effects on the yield of cellulose extracted from bagasse. The three coded independent variables obtained from the statistical analysis software at the design point, the actual experimental results with different parameters and the cellulose yield are shown in Table 3. The interaction between reaction parameters, 2D models, as well as the yield equation of extracted cellulose will be further illustrated.

3.2 Statistical Analysis

The experimental data was analyzed and the following regression equations in terms of coded factors were obtained for the predicted values of output responses (Yield) for

cellulose extracted from bagasse. The Yield (Y) was obtained from statistical design analysis in coded value, as seen below in “(2 and 3)”.

$$Yield = 0.5831 - 0.0052 B + 0.0043 C - 0.0069 AB - 0.0076 AC - 0.0087 B^2 \quad (2)$$

$$A = \frac{t-2.5}{1.5} \quad B = \frac{T-70}{20} \quad C = \frac{c-6}{4}$$

$$Yield = 0.00336 Temp + 0.00424 Conc + 0.0237 time - 0.00023 time * Temp - 0.001267 time * Conc - 0.00002175 Temp^2 + 0.434 \quad (3)$$

Where:

t= Time (hours)

T= Temperature (°C)

C: NaOH Concentration (%)

The R-squared value for the model equation is 87.72 percent, which is higher than 75%, indicating that the quadratic models are well-fitting.

Table 3. Coded results, actual experimental results, and the cellulose yield.

Run	Temperature (°C)	Time (hr)	Concentration (%)	Yield (g/g)
1	-1 (50)	0 (2.5)	-1 (2)	0.5722
2	0 (70)	-1 (1)	1 (10)	0.5871
3	1 (90)	0 (2.5)	1 (10)	0.5744
4	0 (70)	0 (2.5)	0 (6)	0.5761
5	1 (90)	-1 (1)	0 (6)	0.5669
6	-1 (50)	0 (2.5)	1 (10)	0.5868
7	-1 (50)	-1 (1)	0 (6)	0.5621
8	-1 (70)	0 (2.5)	0 (6)	0.5843
9	-1 (50)	1 (4)	0 (6)	0.586
10	0 (70)	0 (2.5)	0 (6)	0.5845
11	0 (70)	-1 (1)	-1 (2)	0.5684
12	0 (70)	1 (4)	1 (10)	0.5713
13	1 (90)	0 (2.5)	-1 (2)	0.5612
14	0 (70)	0 (2.5)	0 (6)	0.5844
15	0 (70)	0 (2.5)	0 (6)	0.5864
16	1 (90)	1 (4)	0 (6)	0.5632
17	0 (70)	1 (4)	-1 (2)	0.5832

3.3 Combined Effect of Studied Parameters on Cellulose Yield

A contour plot (Level Plot) is a way to show a three-dimensional surface on a two-dimensional plane. It indicates the minimum and maximum value and also indicates the effect of two factors on a certain response on a two-dimensional plot. In the contour statistical model graph, two factors change while the third factor remains constant at its minimum, average or maximum value to determine the maximum cellulose yield that can be obtained at these values. Changing the colour from blue to red indicates that the yield increases. The bluish the colour the lowest the yield, whereas the reddish the colour the highest the yield at any condition. Each line shows the yield obtained at any condition corresponding to the other two parameters through the line. The contour plot is useful for establishing the response values and operating conditions as required.

3.3.1. Effect of Time and Temperature on Cellulose Yield

When the concentration of sodium hydroxide remained constant and there is a change in the value of temperature or time, it was found that there was a change in the value of cellulose yield.

It was obvious from Fig. 3 (a, b, and c) that at minimum, average and maximum values for sodium

hydroxide concentration, while increasing the time and temperature the yield of cellulose was found to be increased. This may be attributed to increasing the time of stirring will increase the interaction between NaOH solution and bagasse powder, which in turns lead to increase the removal of lignin and large amount of hemicellulose. When temperature of the reaction between sodium hydroxide and bagasse powder increases the rate of the reaction will increase. This will lead to an increase in the cellulose yield until it exceeds 90 °C, this is because above 90 °C the solid to liquid ratio start to change as water start to evaporate gradually [22].

3.3.2. Effect of Temperature and Concentration on Cellulose Yield

When the time remained constant and there is a change in the value of temperature and concentration, it was found that there was a change in the value of cellulose yield. It was evident from Fig 4. (a, b, and c) that at minimum, average and maximum values for time, while increasing the concentration of NaOH the cellulose yield increases and this is due to the weight loss of the sample resulting from the strong breakage of the bonded network between hemicellulose and lignin. But when the reaction temperature increases the yield of cellulose started to decrease because the solid to liquid ratio begins to change as water start to gradually evaporate. Whereas, in case of Fig. (4 c) as the concentration and reaction temperature increases the cellulose yield decreases, because when the reaction time increases the concentration start to decrease and increasing in temperature reduces the amount of water, so this changes the solid to liquid ratio [22].

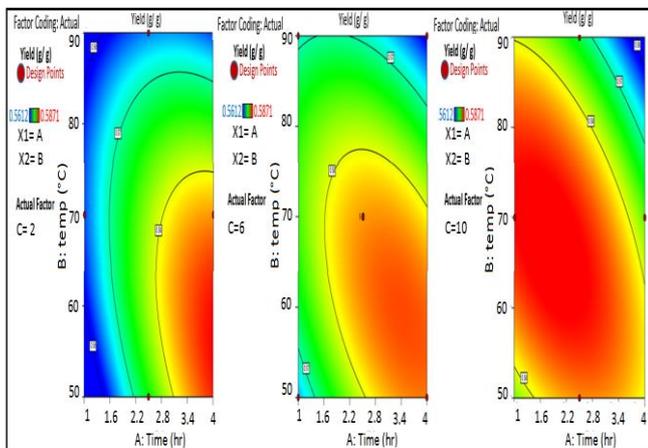


Fig. 3 Contour graph for effect of time and temperature on cellulose yield at 2 % (a), 6 % (b) and 10% (c) conc. of NaOH

3.3.3. Effect of Time and Concentration on Cellulose Yield

At constant temperature, the values of time and concentration will change and also the value of cellulose yield will change. It was clear from Fig. 5 (a, b, and c) that at minimum, average and maximum values of temperature, while increasing the time and NaOH concentration the yield of cellulose was found to be increased. Besides, the yield was found to increase with time increase till it reaches 3.4 hours as shown in Fig (5 b) and (5c), and this is because at high temperatures the rate of reaction between NaOH and bagasse powder increase and breakdown of lignin macromolecules increase so more lignin were separated from cellulose and thereby cellulose purity increases, but at

high concentrations after certain time the yield began to decrease as no more impurities to be removed and maximum yield is achieved [22]. Then it starts to decrease as shown due to the change in solid to liquid ratio at elevated temperature.

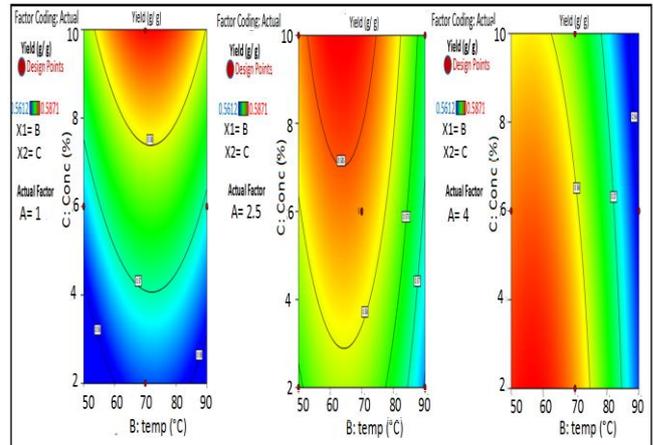


Fig. 4 Contour graph for effect of temperature and concentration on cellulose yield at time 1 hr (a), 2.5 hr (b) and 4 hr (c).

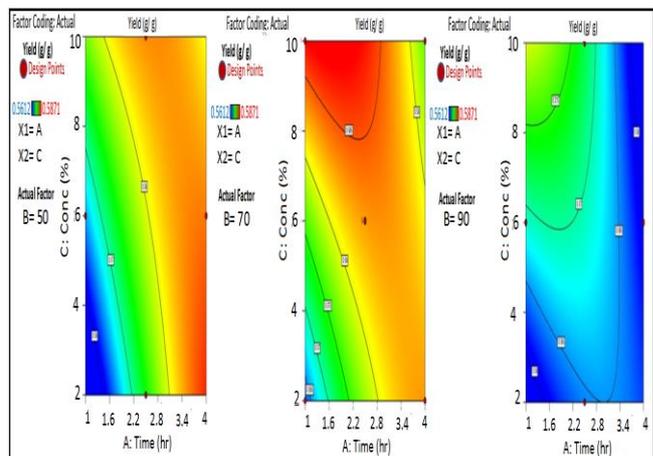


Fig. 5 Contour graph for effect of time and concentration on cellulose yield at temperature 50 °C (a), 70 °C (b) and 90 °C (c).

3.4 Response Optimization

The vital part of the experimental study was to determine the optimum process condition where maximum cellulose can be extracted from SCB. Optimization of the extracting variable parameter was conducted in a numerical optimization method, which reveals that the optimal predicted operating parameters for cellulose yield were obtained at 9.8 % NaOH concentration by volume, reaction temperature of 60 °C and extraction time of 2.4 hours. The target is to have maximum cellulose yield of 0.5871 g/g. The practical confirmatory experimental results were found to be in a good agreement and closest to the predicted results. The ultimate errors of 2 % were very low for the predicted responses (Yield) obtained using the developed models for cellulose extraction. The detail of optimization condition is shown in Table 4.

Table 4. Optimum experimental results

Run	Yield g/g
1	0.575
2	0.576
3	0.576
4	0.575
5	0.576

3.5. Characterization for Bagasse and Extracted Cellulose

The characterization for the dried raw bagasse and its respective extracted cellulose obtained at the optimum conditions was done, by using the different analytical quantitative following techniques.

3.5.1 Fourier Transform Infrared (FTIR)

In this investigation, the qualitative analysis of bagasse before and after alkali treatment at the optimized condition was determined. FTIR spectroscopy is a powerful tool for analyzing the characterization of bagasse and the extracted cellulose, as it signifies the comprehensive view about the structure/composition of the vital active groups in the bagasse fiber. Infrared (IR) spectroscopy is used for the study of interactions between the matter and electromagnetic fields in the IR region. The probability of a particular IR frequency being absorbed depends on the actual interaction between the frequency and the molecule.

The FTIR spectroscopy was used to identify changes in functional groups and/or molecular conformation caused by the pre-treatment process. It was previously observed that the three components of biomass (cellulose, hemicellulose, and lignin) are most likely composed of alkene, esters, aromatics, ketone, and alcohol, with various oxygen-containing functional groups observed, such as OH ($3400\text{--}3200\text{ cm}^{-1}$), C=O ($1765\text{--}1715\text{ cm}^{-1}$), and others [23]. However, they continue to exhibit distinct IR structures. Table 5 shows the main differences in FTIR spectra this table summarizes the higher bands observed in the FTIR spectrum of SCB and their assignments to chemical group vibrations and molecules, as well as the main functional groups of the biomass components.

Through the analysis of its functional groups, FTIR spectroscopy was used to demonstrate that lignin and hemicellulose were removed during the cellulose isolation process. The most noticeable differences in the spectra of unmodified SCB and extracted cellulose fibers were found between 3500 and 600 cm^{-1} . According to the FTIR spectrum, there are several peaks in SCB that are not found in the cellulose spectrum. Peaks are approximately 1250 cm^{-1} , 1516 cm^{-1} , and 1736 cm^{-1} . These absorptions are attributed to lignin functional groups that are associated with cellulose and hemicellulose prior to the delignification of SCB. The 1250 cm^{-1} absorption peak is caused by the C-O stretching vibration of the aryl group in lignin. The C=C stretching vibration of the aromatic ring in lignin is responsible for the peak at 1516 cm^{-1} . The peak at 1736 cm^{-1} in the bagasse FTIR spectrum is related to the C=O stretching vibration of carboxylic groups in lignin and hemicellulose. Fig. 6a depicts two significant absorption bands that must be emphasized, the bands at 1516 and 1250 cm^{-1} .

In the spectrum of extracted cellulose fibers obtained after the alkaline chemical treatment process, the band at 1516 cm^{-1} is absent, and the band at 1250 cm^{-1} is nearly

absent, indicating that most of the lignin and hemicellulose were removed from SCB [9 - 24, 25]. Due to a decrease in fiber hydrophilicity, favourable phenomena for using such fibers as reinforcement in certain polymer matrix have been discovered [26]. The removal of lignin was confirmed by X-ray diffraction, which revealed that the extracted cellulose fibers had a higher crystallinity degree.

Table 5. Infrared Main Transitions for SCB.

Source	Vibration	Wavenumber (cm^{-1})	
		Investigated results	Literature [25]
Polysaccharides	O-H linked shearing	3434.5	3300
Polysaccharides	C-H symmetrical stretching	2924.9	2885
Xylans	C=O unconjugated stretching	1735.9	1732
Water	OH (water)	1628.6	1650–1630
Cellulose	C-O-C asymmetrical stretching	1162.8	1162
Cellulose	C-OH out-of-plane bending	665.1	670

The observed FTIR spectroscopic investigations demonstrated the main differences in functional groups between SCB and its respective extracted cellulosic material by displaying the different absorption bands that characterize the raw biomass bagasse and its respective extracted cellulosic material regenerated after alkali pre-treatment, as shown in Fig. 6 (a and b). A change in crystalline organization results in a significant simplification of the spectral contour due to a reduction in intensity or even the disappearance of the bands characteristic of the crystalline domains.

The FTIR spectra for both samples showed similar patterns, with the dominant peaks observed around 3434.5 cm^{-1} and 3426.1 cm^{-1} for the bagasse and extracted cellulose, respectively, which are characteristic for the OH-stretching vibration of the hydroxyl groups in cellulose, hemicellulose, and lignin [27], and this broad band gives useful information about the formation of hydrogen bonds. The spectra also revealed a C-H stretching vibration at a band of around 2924.9 cm^{-1} for bagasse, and a slightly shifted band at around 2921.3 cm^{-1} for the extracted cellulosic material.

Absorbance peaks caused by O-H bending of adsorbed water appear at around 1629 cm^{-1} in the bagasse spectrum and at around 1637 cm^{-1} in the cellulose spectrum. The absorption peak around 1163 cm^{-1} in the bagasse spectrum and its corresponding shifted one around 1162 cm^{-1} in the cellulose spectrum are caused by the cellulose/hemicellulose C-O-C pyranose ring asymmetric stretching vibration [9, 11].

Another prominent absorption band in the FTIR spectra appears around 899 cm^{-1} , which is attributed to C-O-C stretching at cellulosic β -(1 \rightarrow 4)-glycosidic linkages between glucose [28-30]. The absorption peaks for bagasse and extracted cellulose appearing at 665 cm^{-1} and 663 cm^{-1} respectively, are indicating the cellulosic C-OH out-of-plane bending. Furthermore, the 1426 cm^{-1} FTIR absorption band in the extracted cellulose spectrum was assigned to a

symmetric CH₂ scissoring vibrating motion in cellulose [31]. This band is also known as the "crystallinity band," because a decrease in its intensity corresponds to a decrease in the degree of crystallinity of the samples.

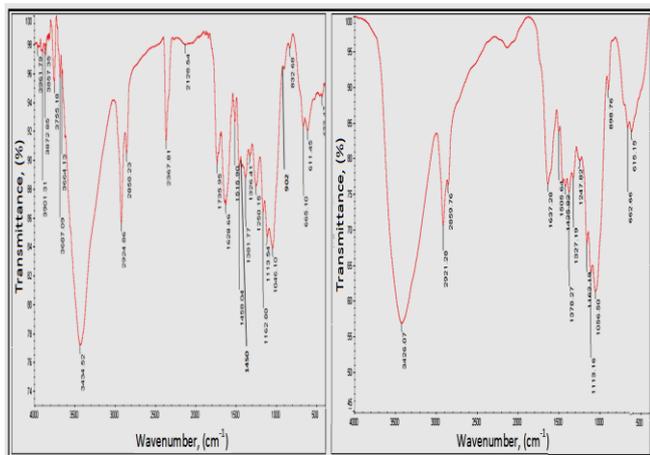


Fig. 6 FTIR for untreated bagasse (a) and extracted cellulose fibers (b) at (wavenumber 4000 to 400 cm⁻¹).

The peak in the bagasse spectrum with an absorption band around 1382 cm⁻¹ is assigned to C-H and C-O vibrations contained in the polysaccharide rings of cellulose, and its corresponding one in the cellulose spectrum is shifted at around 1378 cm⁻¹. Furthermore, the peak at 1113 cm⁻¹ is consistent with aromatic C-H in plane lignin deformation and can be attributed to the C-O-C glucosidic ring vibration in cellulose. The absorption peaks at 1326 cm⁻¹ and 1327 cm⁻¹ for bagasse and extracted cellulose, respectively, were assigned to the cellulose CH₂ wagging frequency. Furthermore, bands at 1458 cm⁻¹ and 1450 cm⁻¹ for bagasse and extracted cellulose, respectively, are caused by C-H asymmetric deformations.

These observed peaks are typical of lignocellulosic materials and match those reported in the literature [26]. However, after the chemical treatment, some peaks in the bagasse FTIR spectrum linked to aromatic skeletal vibrations of lignin and hemicelluloses were reduced, and others were almost reduced in the corresponding regions for the extracted cellulose FTIR spectrum. This indicates lignin and hemicellulose removal, as well as cellulose exposure.

3.5.2. X-ray diffraction analysis (XRD) for Bagasse and Extracted Cellulose

X-ray diffraction analysis (XRD) is a technique used in materials science to determine the crystallographic structure of a material. XRD works by irradiating a material with incident X-rays and then measuring the intensities and scattering angles of the X-rays that leave the material. X-ray diffraction examinations were conducted on bagasse fiber and the respective extracted cellulose to analyze the changes in the crystal form and to establish the impact of alkali (NaOH) treatment on the crystallinity of the final fibers.

Fig. 7 (a and b) shows the XRD profile of treated and untreated bagasse fiber, which reveals that cellulose is a powder composed of two phases, amorphous and crystalline phases with high purity and crystallinity. Using the software with diffraction angles 2θ ranging between 5° and 60°, a wide range is captured for all the intensities of both the amorphous and crystalline fractions of the samples.

The major peaks in the SCB and cellulose diffractograms are at 2 values that correspond to 15.5° and 22.5° of the cellulose I polymorphs in natural fibers [32, 33]. Results for crystallinity indicated that SCB had a lower level of crystallinity than the extracted cellulose, and an increase in crystallinity index following the extraction of cellulose from SCB was noticed in similar findings [34]. They claimed that the increased cellulose exposure compared to untreated SCB was the cause of the higher crystallinity index in the respective extracted cellulose. In addition, the high amorphous hemicelluloses and lignin contents present in the untreated materials are the main causes for their relatively lowest crystallinity index. Results of XRD studies show two distinctive peaks around 2θ value of 15.33° and 22.09° for the bagasse fiber, characteristic of typical forms of cellulose I structure. Similarly, the extracted cellulose exhibited a distinctive cellulose type I pattern with clear peaks at 2θ of 16.35° and 22.47° that were significantly strengthened in comparison with that of the bagasse. The only difference is a slight intensity change in the peaks, indicating some change in the crystallinity index of samples. The first peak (2θ at 22°) correlates to the crystallographic field of plane 200 cellulose lattices, while the later peak at 16° represents the amorphous region in plane 110 of cellulose type I (Fig. 7b). Presence of these peaks in both samples indicates that chemical structure of cellulose was not changed during the chemical treatment. The crystallinity index (CI) of the sample was calculated according to "(4)", by measuring the height of the maximum peak (I₂₀₀) and the height of the minimum peak (I_{am}) between the 002 peak and the 001 peaks.

$$CrI (\%) = I_{200} - I_{am} / I_{200} \quad (4)$$

In this equation, I₂₀₀ stands for the lattice diffraction peak's greatest intensity at a 2θ of approximately 22°, and I_{am} for the intensity dispersed by the sample's amorphous component, which was assessed as having the lowest intensity at a 2θ of about 16°.

It was evident that the crystallinity is increased from 31.76 % for the raw bagasse fibers to 51.13 % for the extracted cellulose, as in agreement with the earlier findings of the FTIR analysis. It was observed that the proportion of crystalline to amorphous regions in the cellulose had increased, which might be due to the removal of hemicelluloses, lignin, and amorphous cellulose during the treatment [35]. The primary component of the extracted cellulose is still cellulose I in its structural form. This suggests that the treatment has insignificant effect on the cellulose's lattice structure. When compared to their equivalents on the bagasse curve, the peaks at 16° and 22° for the extracted cellulose are found to be more clearly defined, showing that alkali treatment can raise the crystallinity index of cellulose.

The appearance of the peaks at 15° and 22° is evidence that the treatments had an impact on the fibers. These peaks locations suggest that the interlunar distance for the treated fibers had increased. This pattern develops as a result of the disorder that is produced when fibers are treated. An increase in the interfibrillar distance is linked to the projection of replacing groups along the axis [36].

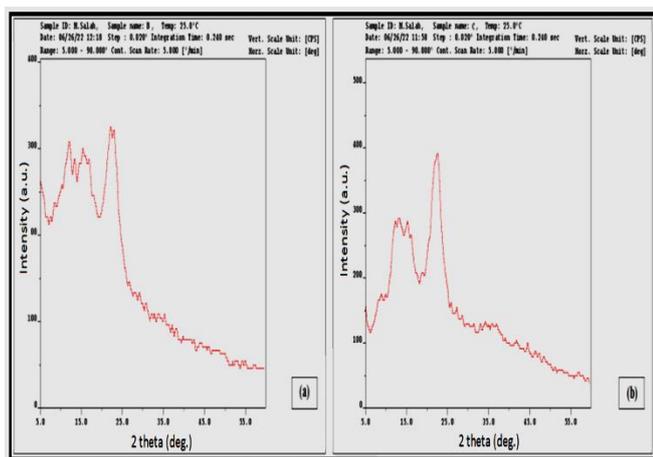


Fig. 7 X-ray diffractograms of SCB: unmodified bagasse (a) and extracted cellulose (b).

The lignin and hemicellulose content were eliminated during the NaOH treatment, which also raised the crystallinity index over 62 percent [37, 38]. This verifies that the elimination of lignin and hemicellulose suggested by FTIR data actually occurred. Additionally, the OH groups in the fiber react with NaOH during an alkaline treatment in the manner described below:



As Na⁺ ions enter the cellulose unit cell, the cell [thermal stability, crystallinity, etc.] increases [39]. Hemicelluloses and the lignin found in lignocelluloses are thought to be the amorphous components, whereas cellulose was thought to be the crystalline part. The partial removal of hemicelluloses and amorphous cellulose fractions during the alkaline pre-treatment resulted in pre-treated samples with better crystallinity indices [40]. Although the alkali treatment of the bagasse in this study resulted in an obvious increase in the crystalline index of the cellulose as seen in the XRD diffractograms, there is still hope for an additional increase if the treated bagasse is subjected to additional bleaching and nanocellulosic extraction processes.

4. CONCLUSION

In this work, a successful demonstration for an alkali (NaOH) treatment aided with the assessment of an RSM design expert software for extracting cellulose from raw SCB have ensured that economically and environmentally the extracted SCB celluloses could be potentially utilized in a diversity of industrial applications. The distinguished influence of NaOH concentration, time, and temperature as different selected process parameters on the yield (response) of extracted cellulose was studied using RSM. The Response Surface Methodology model is able to estimate the experimental results very well. The Box-Behnken design used the three optimal processing independent variables to screen and adjust the ideal conditions for extracting cellulose from SCB. It was observed that the optimum yield of 0.5871 g was achieved when NaOH concentration, time, and temperature were 9.8 %, 2.4 hrs and 60 °C respectively. The second-order polynomial equation that relates the response to the extraction optimum parameters was developed and the predicted and actual values are in agreement with each other. Overall, it was evident that the

optimization of the selected process variables to a certain extent imparts a significant impact on the enhancement of the yield for the extracted cellulose. The bagasse and extracted cellulose's functional group as well as the crystallinity index were determined. The crystallinity index of the bagasse and its respective extracted cellulose was 31.76 % and 51.13 % respectively. The major role of the alkali (NaOH) treatment is obvious through the FTIR findings, which confirmed the appearance of the peaks characteristic to the amorphous (lignin and hemicellulose) region in the bagasse spectrum and then diminished or disappeared from the extracted cellulose spectrum. The yield and results of different characterizations obtained in this work revealed that the natural raw bagasse fiber is a good agricultural waste that can be a source for cellulose extraction, and consequently could be valorized in a diversity of engineering industrial applications.

ACKNOWLEDGMENT

The authors extend their sincere thanks, appreciation and respect to Prof. Shereen Mohamed Samir, Head of Chemical Engineering Department and under the enlightened leadership of Prof. Fawzy Ibrahim, Dean of Egyptian Academy of Engineering & Advanced Technology in Egypt for their sincere efforts, for successfully achieving this research work in all its stages.

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