Optimization of a Bleaching Process for the Conversion of Sugar Beet Pulp Alpha-Cellulose with a High Whiteness Index and Purity

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ABSTRACT: *A large amount of sugar beet pulps are produced annually as waste which causes environmental pollution. However, the composition of sugar beet pulp indicates the possibility of producing several value-added products. In this study, high-quality α-cellulose was extracted from sugar beet pulp. The purity and optical properties of α-cellulose have a significant effect on its application in various industries, such as the production of napkins or a variety of papers. The bleaching step has a significant effect on the quality of cellulose. So bleaching conditions including the concentration of NaClO (4.5-1 % w/w), temperature (25-60 °C), and time (15-45 minutes) were optimized based on response surface methodology. According to the results, good optical properties, low Kappa number, and high cellulose content (yield) could be achieved when the operating parameters were controlled. Severe bleaching conditions such as higher NaClO concentration or longer bleaching time caused cellulose oxidation and reduced efficiency. Optimal bleaching conditions for SBP were determined as 3.28% (w/w) NaClO at 25 °C for 45 minutes. These conditions led to the production of α-cellulose with a 69.38% of whiteness index, a Kappa number of 1.02, and a yield of 23.16%. The structure of the samples was investigated by SEM and FT-IR analysis. Therefore, the pulping and bleaching process has led to value-added products with industrial applications from sugar beet pulp.*

KEYWORDS: *Sugar beet pulp; Optimization; Response surface methodology; α-cellulose.*

INTRODUCTION

Sugar Beet Pulp (SBP) is one of the by-products of sugar factories [1]. Annually Significant amounts of SBP are produced in the world [2]. Just in European Union countries has been reported the annual sugar beet pulp dry weight production reaches around 14 million tons [2,3]. The principal end-use of SBP is in the animal feed area with low nutritional value [4,5]. This dry pulp contains 1.1% fat, 8.92% protein, 3.72% ash, and 86.26% polysaccharides [6]. The polysaccharides in sugar beet pulp include cellulose, hemicellulose, and pectin [7]. To extract cellulose, the first step in the pulping process involves baking the raw materials with hydroxide or sulfide at a temperature between 150-180 °C which this process decomposes the lignin-cellulose-hemicellulose

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complex [8–10]. The material of this process has a dark color due to the presence of hemicellulose and lignin residues, so the bleaching process is required to achieve high levels of whiteness and purity [11,12]. Generally, the pulp's color is due to the lignin coloring groups that should remove as an additional substance from the pulp in the bleaching unit. Phenolic lignin units could be oxidized by alkali to light-absorbing structures, resulting in darkened pulp [13]. therefore increasing the whiteness of the pulp is achieved by decolorizing or dissolving the color compounds of the pulp, especially the removal of lignin [14,15]. Increasing the whiteness improves the quality and value of the final product [16]. Pulping can dissolve about 90% of lignin without degrading cellulose fibers, so more delignification is done with bleaching [17]. Also, bleaching could be done in separate steps using different chemicals such as chlorine, sodium hydroxide, hypochlorite, chlorine dioxide, ozone, and hydrogen peroxide [18]. Parameters affecting the bleaching process including temperature, bleaching agent concentration, process time, and the source of the cellulose, are important in bleaching quality.

If bleaching conditions don't control, bleaching agents can reduce the amount of cellulose and yield besides removing lignin and increasing the cellulose whiteness index [19]. The bleaching mechanism involves the break specific bonds in lignin (e.g., aryl ether bonds, carbon-carbon bonds, or β-O-4 bonds) to produce water-soluble products, such as aromatic aldehydes and carboxylic acids [19–21].

Few studies have examined the factors affecting this process. fewer chemicals and milder conditions are required for the bleaching process of plant biomass such as fruit and vegetable pulp than wood-based cellulose because their lignin content is less than the woody source [19,22]. In the study by Wang et al., α-cellulose with the whiteness index of 72.36 were extracted from apple pomace, using 1 M HCl solution, 1 M NaOH, and 1-1.5% NaClO solution at 80 ℃ for 1 hour [19]. In the study of *Alves et al*., α-cellulose with a Kappa number of 2.7 was extracted from sorghum straw by 1.25% NaOH and 25 mL of 2% $NaClO_2$ solution [11].

In this research, by pulping and bleaching process using NaOH and NaClO, respectively, α-cellulose with high purity and whiteness index was extracted from SBP. Bleaching was optimized by Response Surface Methodology (RSM). The yield, Kappa number, and the whiteness index of the extracted cellulose were used as the optimization criterion.

EXPERIMENTAL SECTION

Materials

SBP was prepared by Kermanshah Sugar Company. Sodium chloride (NaClO), sodium hydroxide (NaOH), acetic acid, and anthraquinone were prepared from Sigma-Aldrich. All materials had an analytical grade.

SBP pulping process

The soda-anthraquinone pulping method was performed to remove lignin and hemicellulose from SBP, and bleaching was performed to remove maximum hemicellulose and lignin. Briefly, in a 2000 ml reactor, SBP was treated in 0.1 M NaOH solution containing anthraquinone (0.2% by weight of anthraquinone/kg pulp) at 75 °C for 40 min. After pulping, the mixture was filtered using distilled water until the filtered water was at neutral pH.

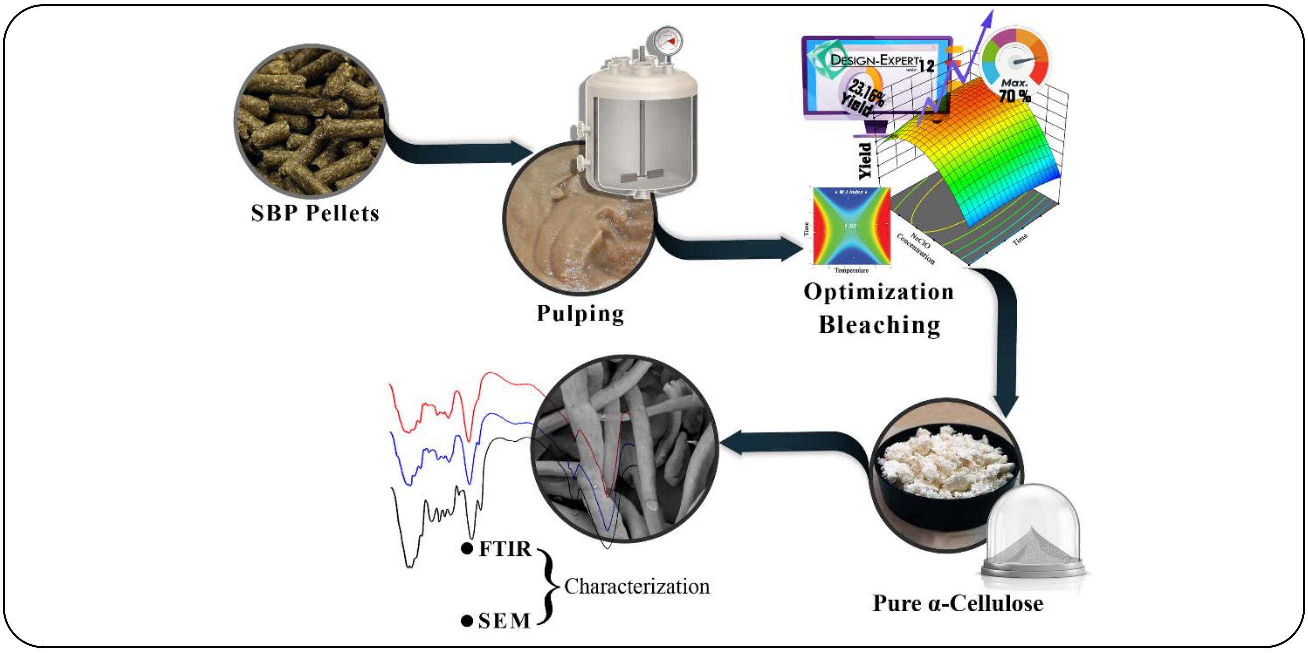
SBP bleaching process

The pulping can dissolve about 80-90% of lignin without destroying cellulose fibers, so further delignification is done by bleaching. After alkali treatment, the residue was bleached with NaClO solution with a mechanical stirrer at a specific temperature for a limited time. The pulp was then washed with distilled water to neutralize and remove the odor.

The bleaching of the pulp was carried out by optimizing the NaClO concentration, time, and temperature of the bleaching process. The optimization of the experiment was carried out through 22 experiments based on cellulose content (yield), whiteness index, and Kappa number (Table 1). One gram of pulp was used for each treatment. The pulp bleaching was carried out in the same reaction system employed in the pulping process. After bleaching with NaClO, the product was first neutralized with distilled water and then treated with acetic acid in a mechanical stirrer. After this process, the mixture was allowed to cold and then filtered using distilled water to neutralize the pH of filtered water. Finally, the bleached samples were treated with diethylenetriaminepentaacetic acid (DTPA) as a chelating agent to chelate metal ions at 70 °C for 30 min, followed by washing with distilled water. Then the samples were dried by the freeze-drying method. The graphical design of these processes is indicated in Scheme 1.

Table 1: Response Surface Methodology (RSM) test for the bleaching process.

Std	Run	Factor 1	Factor 2	Factor 3	Response 1	Response 2	Response 3
		A:NaClO $\%\frac{w}{w}$	B:Time	C:Temp	Kappa Num.	Whiteness index	Yield
1	2	1	15	25	2.7	61.31	20.9
2	12	4.5	15	25	1.4	68	19.9
3	9	1	45	25	2.29	60.63	22.7
4	10	4.5	45	25	0.76	71.05	19
5	6	1	15	60	2.34	60.81	21
6	15	4.5	15	60	0.77	69.41	18.7
7	14	1	45	60	2.39	59.31	22.8
8	13	4.5	45	60	0.5	70.91	18.1
9	8	1	30	42.5	2.11	60.23	22
10	4	4.5	30	42.5	0.61	69.49	18.9
11	7	2.75	15	42.5	1.4	67.45	22.9
12	11	2.75	45	42.5	1.1	67.91	23.4
13	3	2.75	30	25	1.24	66.9	24.2
14	16	2.75	30	60	1.09	66.49	23.7
15	1	2.75	30	42.5	1.12	67.27	23.9
16	5	2.75	30	42.5	1.12	67.07	23
17	17	2.75	30	42.5	1.2	66.96	23.4



Scheme 1: The graphical design of α-cellulose extraction from SBP.

Characterization of samples

The Kappa number is a measure of the amount of lignin left in the pulp and was determined according to the methodology T236 om-06 of the Technical Association of the Pulp and Paper Industry [23].

The dried α-celluloses by freeze-drying were weighed and the yield of extracted α-celluloses (α-celluloses content %) was calculated by equation (1).

$$\text{Yield (\%)} = \frac{W_{\text{dried } \alpha-\text{celluloses}}}{W_{\text{dried SBP}}} \times 100 \qquad (1)$$

Color measurements were performed using a spectrophotometer (CM-3600A, Japan) by evaluating the values of L * (light), a * (red), b * (yellow), and the whiteness index (WI %) was calculated using Eq. (2).

$$\text{WI (\%)} = 100 - \sqrt{(100 - L^{*})^2 + a^{*2} + b^{*2}} \qquad (2)$$

The morphology of the dried samples of SBP, samples after pulping, and α-cellulose were examined by scanning electron microscopy (Tescan s.r.o. Brno, Czech Republic).

Fourier Transform Infra-Red (FT-IR) (Perkin Elmer, model Spectrum two, Beaconsfield, UK) was applied to identify the functional groups. Fourier transform infrared spectra of SBP, samples after pulping, and α-cellulose were recorded. The samples were mixed with KBr and pressed as tablets, and transmission was measured in the range of 400-4000 cm^{-1}.

RESULTS AND DISCUSSION

Characterization samples by SEM and FT-IR

In (Fig. 1a), the cell layers are joined together by lignin, and lignin is seen between and inside the cavities of the surrounding cells. These materials were removed by NaOH hydroxyl ions during the pulping, and lignin and hemicellulose were approximately dissolved. The cells separate from each other's layers and, cavities and porosity have appeared, but some impurities are seen on the fibers due to the presence of lignin and hemicellulose that remain after pulping (Fig. 1b). The residual lignin and hemicellulose content were removed by the sodium chloride used in the bleaching process (Fig. 1c). So the surface of cellulose fibers is free of impurities and looks smooth as shown in (Fig. 3c). The impurities were dissolved in the reaction solution, and the purity and whiteness index of cellulosic fibers were increased [24,25]

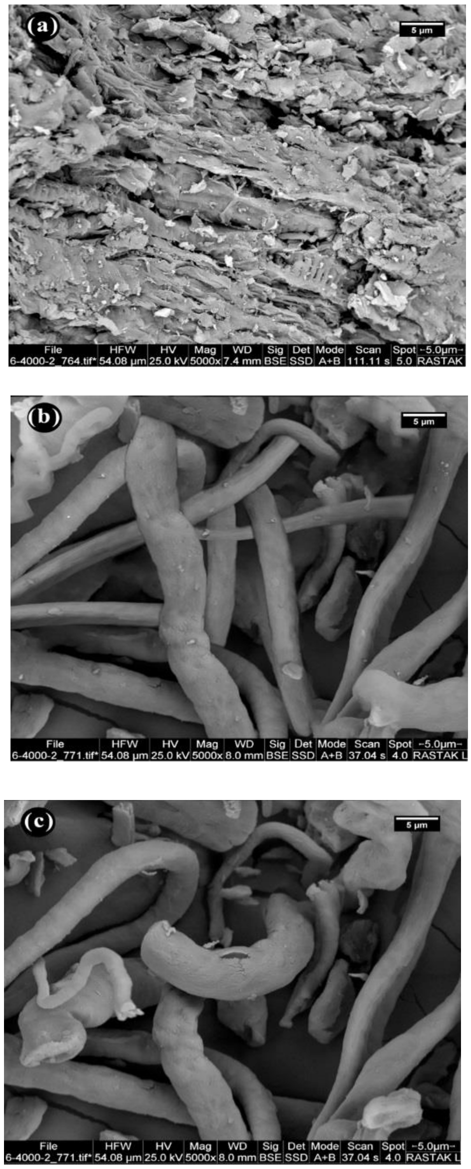


Fig. 1: SEM of (a) SBP before treatment, (b) cellulose after pulping, (c) bleached α-cellulose.

FT-IR analysis was performed to investigate the effect of alkaline treatment (pulping) and bleaching on cellulose structure. The FT-IR spectrum of SBP, cellulose after pulping, and bleached α-cellulose are shown in (Fig. 2). The band located at about 1739 cm^{-1} in the SBP spectrum is attributed to the tensile strength of O=C of the hemicellulose acetyl and uronic groups or the ester bonds of the carboxylic groups of ferulic and p-coumaric acids of lignin and xylan in hemicellulose. (Fig. 2a). After bleaching the fibers, the band at 1739 cm^{-1} was no longer observed in the FT-IR spectrum (Fig. 2c). However, the 1516 cm^{-1} and 1739 cm^{-1} bands related to lignin are still observed after pulping in the FT-IR spectrum of the fiber (Fig. 2b). This result showed that the alkaline treatment with sodium hydroxide did not remove completely the lignin or hemicellulose content. The 1516 cm^{-1} band is known as the aromatic skeletal vibrations of the lignin functional groups (Fig. 2a). The spectra of all samples show C-H aliphatic tensile vibrations in cellulose, hemicellulose, and lignin at approximately 2927 cm^{-1}. Due to the alkaline treatment with NaOH, the peaks attributed to hemicellulose and lignin were reduced by removing most of the hemicellulose and lignin from the spectrum during the pulping step (Fig. 2b). The FT-IR spectrum of bleached α-cellulose indicates the peaks attributed to hemicellulose and lignin were reduced or disappeared, and the peaks attributed to cellulose were increased in intensity (Fig. 2c). Similar results were reported for bleached α-celluloses from different sources [11,26–29].

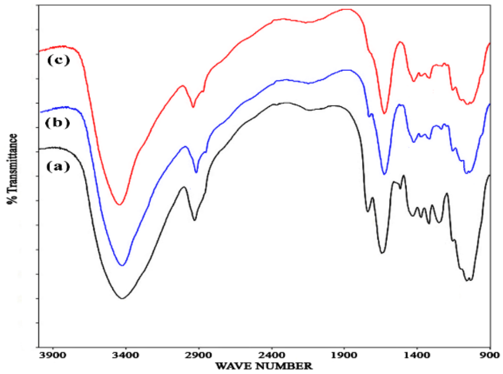


Fig. 2: FT-IR of (a) SBP before treatment (b) cellulose after pulping (c) bleached α-cellulose

Optimization of bleaching conditions

The optimization of the bleaching process was carried out based on high yield, high WI, and low Kappa numbers. Bleaching time, temperature, and NaClO concentration were 15-45 minutes, 25-60 °C, and 1-4.5% $^{w}/_{w}$, respectively (Table 1) for 1 g of pulp. By using the Central Composite Design (CCD) method for experimental data, second-order polynomial equations have been proposed to explain Kappa, WI, and yield of α-cellulose with only significant parameters (Equations 3-4-5). The results were analyzed according to P values and F values provided by the design expert 12 software (Table 2). According to the values of the sum of squares, the mean of the squares, and the total F, the models effectively reduce the fluctuations, and the results obtained for these models (Kappa number, WI, and yield) are significant. A higher F-value means a correct prediction for the experimental data, and a lower P-value indicates its validity. P values must be less than 0.05, and the P-value of the model is less than 0.0001 for Kappa number, WI, and yield, indicating that the models are significant and stable. The insignificant terms should be removed from the models to increase accuracy. In the proposed models, the R square (0.9930, 0.9968, and 0.9993, for yield, Kappa number, and whiteness index, respectively) shows the multiple correlation coefficient of the models, and their values are close to one, which means more coordination of the models to estimate the results. As shown in (Fig. 3), most of the responses are in the very close range or on the line, indicating the accuracy of the models for predicting Kappa number (Fig. 3a), WI (Fig. 3b), and yield (Fig. 3c). The important interaction between variables was considered in the proposed models.

The Kappa number results indicate the lignin decomposition capacity related to the combination of the process's time, NaClO concentration, and temperature parameters during the bleaching process. According to Table 2 (p and f values), all three parameters (temperature, time, and NaClO concentration) were affect the Kappa number. The obtained bleached samples are considered high quality because they have low Kappa number values (between 0.5 and 2.7). The lowest value of Kappa number 0.5 is related to the combined treatment of 4.5% $^{w}/_{w}$ of NaClO solution and temperature of 60 °C for 30 minutes, while the highest value of Kappa number is 2.7 with a combination of 1% $^{w}/_{w}$ NaClO, 25 °C, in 15 minutes. The second-order polynomial equation Equation (3) provides a good fit with the experimental data for the Kappa number, with the value $R^2 \cong 1$. The response level diagram for the Kappa number was constructed using Equation (3) and is shown in (Fig. 4a).

$$\text{Kappa Number} = 4.79011 - 0.755210A - 0.064663B - 0.035044C - 0.002619AB - 0.002571AC + 0.000395BC + 0.090532A^2 + 0.000743B^2 + 0.000269C^2 \quad (3)$$

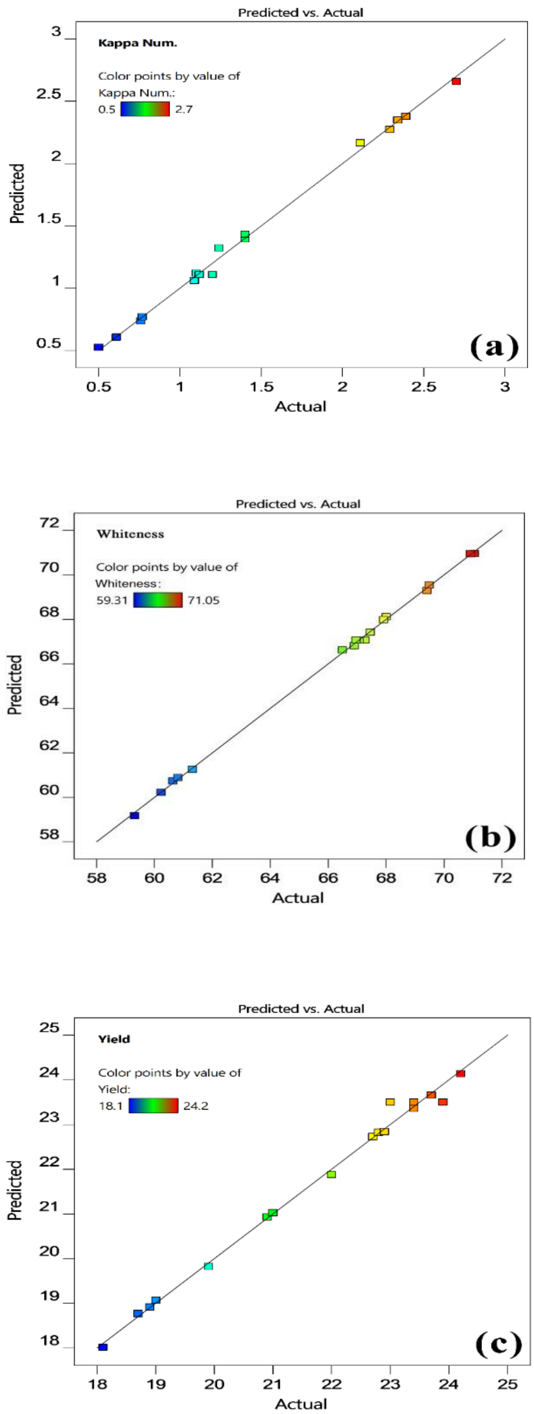


Fig. 3: Actual vs. predicted response values (a) Kappa number (b) WI (c) yield of α-cellulose.

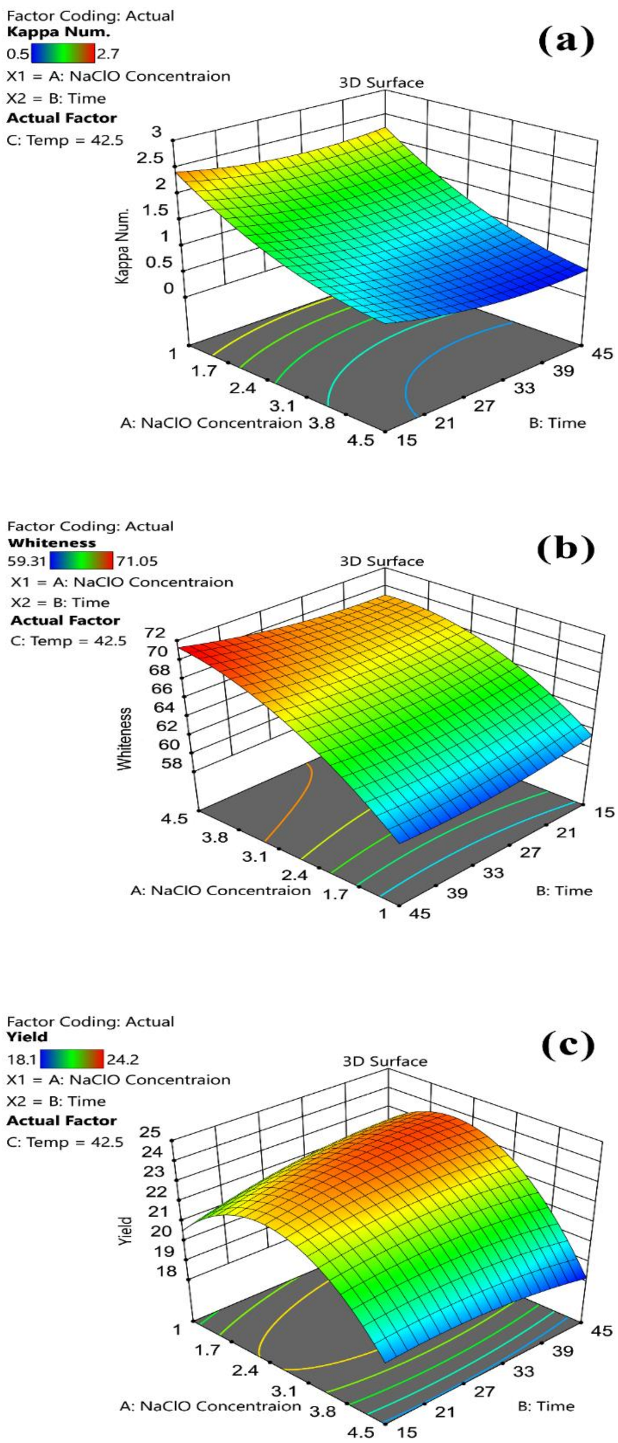


Fig. 4: Response level diagram for (a) Kappa number, (b) WI, and (c) yield.

From Table 2, it is clear that the WI of extracted celluloses is more affected by the NaClO concentration parameter and the processing time. In this regard, WI increases with increasing NaClO concentration or process time. The second-order polynomial equation (Equation (4)) presents the fit of the experimental data for WI of α-cellulose, and the response level diagram for WI of α-cellulose using Equation 4 is shown in (Fig. 2b).

Table 2: ANOVA of CCD model for all responses in the bleaching process.

Source	ANOVA of CCD model for Kappa number				ANOVA of CCD model for the whiteness index				ANOVA of CCD model for the yield			
	Sum of Squares	Mean Square	F-value	p-value	Sum of Squares	Mean Square	F-value	p-value	Sum of Squares	Mean Square	F-value	p-value
Model	7.47	0.8298	242.56	< 0.0001	243.52	27.06	1172.45	<0.0001	67.28	8.41	142.69	<0.0001
A-NaClO %	6.07	6.07	1773.87	< 0.0001	216.88	216.88	9397.6	<0.0001	21.9	21.9	371.65	<0.0001
B-Time	0.2465	0.2465	72.05	< 0.0001	0.8009	0.8009	34.7	0.0006	0.676	0.676	11.47	0.0095
C-Temp	0.169	0.169	49.4	0.0002	0.0922	0.0922	3.99	0.0858	0.576	0.576	9.77	0.0141
AB	0.0378	0.0378	11.05	0.0127	5.66	5.66	245.33	<0.0001	3.25	3.25	55.16	<0.0001
AC	0.0496	0.0496	14.5	0.0066	1.19	1.19	51.72	0.0002	0.6613	0.6613	11.22	0.0101
BC	0.0861	0.0861	25.17	0.0015	0.7021	0.7021	30.42	0.0009				
A^2	0.206	0.206	60.2	0.0001	12.77	12.77	553.41	<0.0001	25.75	25.75	436.86	<0.0001
B^2	0.0749	0.0749	21.91	0.0023	1.09	1.09	47.06	0.0002	0.4287	0.4287	7.27	0.0272
C^2	0.0181	0.0181	5.3	0.0548	0.325	0.325	14.08	0.0071	0.4287	0.4287	7.27	0.0272
Residual	0.0239	0.0034			0.1615	0.0231			0.4715	0.0589		
Lack of Fit	0.0197	0.0039	1.85	0.3877	0.1121	0.0224	0.9081	0.5985	0.0648	0.0108	0.0531	0.9974
Pure Error	0.0043	0.0021			0.0494	0.0247			0.4067	0.2033		
Cor Total	7.49				243.68				67.75			

$$\text{Whiteness index} = 57.19667 + 5.08474A - 0.191084\text{B} + 0.090362\text{C} + 0.032048\text{AB} + 0.012612\text{AC} - 0.001129\text{BC} - 0.712918\text{A}^2 + 0.002830\text{B}^2 - 0.001137\text{C}^2 \quad (4)$$

The yield of extracted α-celluloses generally decreases under severe conditions of high NaClO concentration, high bleaching process temperature, and time. The Kappa number and yield parameter of extracted α-cellulose indicate that the weight loss of the samples was caused not only due to the removal of lignin but also by the destruction of cellulose. The second-order polynomial equation Equation (5) fits well with the experimental data for the α-cellulose extraction yield, and the response level diagram for the α-cellulose extraction yield using Equation (5) is shown in (Fig. 2c).

$$\text{Yield} = 15.89189 + 5.84918A + 0.190786B - 0.098918\text{C} - 0.024286\text{AB} - 0.009388\text{AC} - 1.01224\text{A}^2 - 0.001778\text{B}^2 - 0.001306\text{C}^2 \quad (5)$$

In this study, a lower temperature range than other studies (25-60) was selected for bleaching [11,19]. The reason for this, in addition to reducing the need for energy, is to prevent pulp loss through the hydrolysis of carbohydrates, which reduces the yield of cellulose extraction. In addition, higher temperature leads to the formation of undesired by-products through reactions with bleaching chemicals [30]. According to previous studies, NaClO is capable of oxidizing water-insoluble lignin, resulting in the formation of carboxyl groups in lignin and increased water solubility [19,31,32]. Generally, based on ANOVA analysis, NaClO concentration and time had a more effect on yield, WI, and Kappa number of extracted α-celluloses than temperature. When the bleaching time was at the higher level (45 min), greater cellulose quality change was observed as the increase of NaClO concentration. The optimal conditions of the bleaching process were NaClO concentration of 3.28% $^w/_w$, the temperature of 25 ℃, and the time of 45 minutes. Under optimal conditions, 23.16% α-cellulose was obtained with a Kappa number of 1.02 and a whiteness index of 69.38%, which indicates the appropriate quality and quantity of extracted α-cellulose. According to previous studies, cellulose with a low Kappa number and high purity (such as the optimized values in this study) is a good non-conventional source of cellulose, and therefore a cellulosic material for the synthesis of chemical derivatives [25]. In some studies, the degree of whiteness (equivalent to the whiteness index) of some products in the cellulose industries including, napkins (67.2%), toilet paper (67.6%), paper towels (67.9%), and newsprint paper (60.90%) has been investigated and according to the whiteness index of the present research product (69.38%), it can be used to all mention industry [33,34].

CONCLUSIONS

In this study, α-celluloses with optimized properties of 69.834% of the whiteness index, 1.02 of the Kappa number, and 23.158% of the cellulose content (yield) were extracted from SBP using the soda-anthraquinone pulping method and bleaching process. Also, it was observed that yield of extracted α-cellulose decreases in severe conditions, which despite the higher whiteness index and lower Kappa number, indicates a loss of some cellulose. This observation revealed that the optimal combination of NaClO concentration, time, and the temperature was required to obtain α-cellulose products with desired quality. The optimal bleaching conditions for SBP were determined as 3.28% $^w/_w$ NaClO at 25 ℃ for 45 minutes. The FT-IR showed that the disappearance of some peaks could be caused by the removal of esters linked to the hemicellulose or lignin during the pulping and bleaching process. The SEM images confirm these results and indicate the purity and quality of the extracted cellulose were increased after bleaching. The result shows that the value-added products with sufficient whiteness index and low Kappa number can be obtained for use in various industries of cellulose paper from SBP of the sugar factory.

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