Research Article

Producing Dietary Fibers from Sugarcane Bagasse Using Various Chemical Treatments and Evaluation of their Physicochemical, Structural, and Functional Properties

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Abstract
Sugarcane bagasse (SB) like other lignocellulosic materials contains high levels of insoluble dietary fibers (IDF) that can be extracted using various treatments. Moreover, the extracted IDF properties were found to be dependent on the implemented treatment. Thus, this study set out to evaluate the impact of five treatments (NaOH, NaOH+H_2O_2, NaOH+H_2SO_4, PAA (peracetic acid) and NaOH+PAA) and the subsequent bleaching treatment on the physicochemical, structural, and functional properties of SB fiber. In addition, the effect of particle size reduction on the physicochemical and functional properties was investigated. Lignin content, holocellulose content, XRD, FT-IR, and whiteness index were used to characterize the extracted fibers and to evaluate their structural modifications. The experiments confirmed that NaOH+PAA treatment extracted fibers that had the lowest lignin content (1.65%) and highest holocellulose content (93.07%) and exhibited the highest whiteness index (83.37). The high crystallinity index of NaOH+PAA extracted fibers in addition to the disappearance of spectral bands at 1512, 1595, 1620 and 1730 cm^{-1} of NaOH+PAA FT-IR spectrum confirms the preceding outcomes. The water holding capacity (WHC) and oil binding capacity (OBC) of NaOH+PAA extracted fiber and other extracted fibers were improved as a result of bleaching treatment. Reducing the particle size of treated bleached samples to > 500 µm significantly decreased their WHC and OBC whereas increased their α-amylase inhibitory activity. The obtained results indicate that NaOH+PAA is a promising method for the extraction of fibers from SB under moderate conditions.

Keywords: Dietary fiber, Functional properties, Particle size reduction, Physicochemical properties, Sugarcane bagasse

1 Introduction

Sugarcane is a worldwide cultivated agricultural crop. Its global production in 2021 was 1859.39 million tons [1]. Sugarcane represents the raw material for the sugar manufacturing industry that generates high volume residue known as bagasse at the rate of 240 kg for every utilized ton of sugarcane [2]. Sugarcane bagasse (SB) is usually burned to generate a part of the energy required for sugar manufacturing, also it is utilized as a feed for ruminant animals and a fertilizer [3]. Moreover, SB can be used to produce ethanol, methanol, polypropylene composites, particleboard and generate electricity [4]. SB mainly consists of cellulose, hemicellulose and lignin which are arranged in fine fibers bound together by lignin and hemicellulose [5], [6]. The high lignin content of SB in addition to its low hydration characteristic limits its utilization [7]. Thus, several studies have been performed to delignify SB to produce purified dietary fibers (DF) [7]–[9].

The ability of DF to lower the dangers of various diseases has been proven [10]. Indeed, DF characterized

by high water swelling capacity (WSC) and water holding capacity (WHC) can improve satiety, increase the volume of fecal and decrease the excretion time leading to low risks of obesity and constipation as well as colon cancer [11]. Thus, foods containing high levels of DF have gained attraction from many consumers who search for healthy foods [10]. Sangnark and Noomhorm [12] reported that the recommended daily consumption of DF ranges from 30 to 45 g. These aforementioned benefits of DF in addition to highly recommended daily consumptions encouraged many researchers to investigate the ability of incorporating DF in several products [10], [12], [13]. Afrazeh *et al*., [8] reported that the quality of products enriched with lignocellulosic materials is highly relied on the physicochemical and functional properties of these materials, such as water holding capacity (WHC) and oil binding capacity (OBC).

The functional properties of fibers as determinant factors for their utilization were found to be highly dependent on their physicochemical characteristics. Therefore, several studies have been conducted to evaluate the physicochemical properties of new fiber sources or to assess the effect of treatments on these properties [14]. Different delignification treatments, such as dilute acid hydrolysis, alkaline treatment, steam explosion, ionic liquid treatments, liquid hot water, and wet oxidation were investigated to modify physicochemical characteristics of various lignocellulosic materials. These treatments were implemented to remove lignin and hemicellulose, which enhance the digestibility and functional properties of studied lignocellulosic materials [15], [16].

The majority of fibers in plants are characterized as insoluble dietary fibers (IDF). These IDF comprise several functional groups, such as carboxylic acids, phenolics, ketones, aldehydes and ether linkages, which have a potent affinity to bind either oils or water as well as metal ions [11]. To expose these functional groups and increase their binding capacity, DF was subjected to various delignification treatments as previously mentioned. However, the effects of these treatments on chemical constituents of DF and consequently their physicochemical and functional properties were found to be varied [17]. Luo *et al*. [7], Afrazeh *et al*. [8], and Sangnark and Noomhorm [12] found that alkaline hydrogen peroxide treatment improved WHC and OBC of SB and the treated fibers exhibited higher water retentions (9.6–13.2%) than oil retentions (2.73–10.1%). In contrast, Gil-López *et al*., [9] found that either NaOH treated SB or H_2O_2 treated SB exhibited higher oil retentions (15.9 and 12.4%, respectively) than water retentions (12.1 and 9.8, respectively).

The physicochemical characteristics and consequently functional properties of DF are highly influenced by their particle size. Peerajit *et al*., [18] mentioned that a decrease in the size of fiber particles might cause an alteration in the structure of the fiber matrix, which in turn, leads to an increase in surface area and a fracture of fiber matrix pores, which might influence the hydration properties. Zheng and Li [11] found that the reduction of defatted coconut dietary fiber particle size increased their WHC. In contrast, OBC, glucose dialysis retardation index and α-amylase inhibition activity of these low sized fiber particles were decreased as their particle size decreased. Sangnark and Noomhorm [19] found that the WHC and OBC of sugarcane bagasse fiber increased as their particle size decreased. In contrast, the same authors [19] found that the WHC and OBC of rice straw fiber decreased as their particle size decreased. Thus, the main aim of the current study is to evaluate the efficiency of various chemical treatments in removing lignin and modifying the functional properties of the treated fiber. In addition, the effects of size reduction of treated fiber on its physicochemical and functional properties were evaluated.

2 Materials and Methods

2.1 *Raw material and chemicals*

The sugarcane bagasse (SB) was obtained from Kom Ombo Sugar Factory, Aswan, Egypt. It was washed using tap water and dried in a forced air dryer (Shel-lab, USA) at 60 °C for 24 h. The dried SB was pulverized into a fine powder (Cole-Parmer, USA), sieved through an 18-diameter mesh, and then stored in airtight plastic bags. All used chemicals were analytical grade. Sodium hydroxide and hydrogen peroxide were purchased from El Nasr pharmaceutical company, Egypt. Sulfuric acid and acetic acid glacial were purchased from PioChem company, Egypt. Sodium chlorite was purchased from LOBA chemie Pvt. Ltd, India.

2.2 *Lignin and holocellulose determination*

Acid insoluble lignin content was assessed as outlined in the NREL standardized procedure [20] wherein 0.3 g of the sample was mixed with 3 mL H_2SO_4 (72%). The mixture was maintained at 30 °C for 1 h and regularly stirred at intervals of 10 min. Afterward, the mixture was diluted with 84 mL water, which was then kept at 120 °C for 1 h. Finally, the residues were filtered through sintered glass crucibles and dried at 105 °C for 12 h. The lignin content was estimated as the percentage of the difference between the solid weight before and after burning at 650 °C to the sample weight.

The holocellulose of various samples was determined according to the method described by Viera *et al*. [21]. In a 250 mL beaker, 100 mL of distilled water was mixed with the sample (5 g) then concentrated acetic acid (0.5 mL) and sodium chlorite (0.75 g) were added and stirred until the chlorite was completely dissolved. Then, the beaker was put in a waterbath at 75 °C and covered with a watch glass for 1 h with irregular mixing. The samples were digested for three hours wherein at the beginning of the second and third hours the same quantity of reagents was added. At the end of the digestion period, the beaker temperature was lowered to ambient temperature and its content was filtered through a sintered glass crucible. The residue was washed several times with water until neutral pH, dried in an oven at 105 °C for 6 h and weighed to quantify the holocellulose.

2.3 *Treatment methods*

For all the following treatments, the SB to liquid ratio was kept at 1:20 and the obtained treated SB residue was dried at 105 °C until it reached a constant weight. The treatment parameters were chosen according to earlier works in the literature and preliminary trials.

2.3.1 NaOH

SB sample was treated with diluted NaOH (1%, w/v) for 1 h at a temperature of 100 °C [22].

2.3.2 NaOH + H_2O_2

SB sample was consecutively treated with diluted NaOH (1%, w/v) for 1 h and H_2O_2 solution (10%, v/v) for 1 h at 100 °C and 80 °C, respectively.

2.3.3 H_2SO_4 + NaOH

SB sample was consecutively treated with diluted H_2SO_4 (1% v/v in water) and NaOH solutions (2.0% w/v), where each step of them was performed at 120 °C for 40 min. At the end of each step, SB treated residue was filtrated and excessively washed until it reached a neutral pH [23].

2.3.4 Peracetic acid (PAA)

PAA was prepared as described by Zhao *et al*., [24] as follows: The acetic acid was reacted with hydrogen peroxide (30%) at a volume ratio of 2:1, respectively, for 72 h at room temperature and sulfuric acid was added at the concentration of 3% w/w as a catalyst. The treatment was carried out at 80 °C for 2 h, where SB sample (3 g) was added to 60 mL PAA solution in a glass flask which was intermittently stirred to maintain the homogeneity of the system as possible. Finally, the residue was filtered and washed with an ample amount of distilled water.

2.3.5 NaOH + PAA

It was carried out in two steps [25]. Firstly, the SB sample was treated with 10% NaOH solution at 90 °C for 1.5 h. At the end of this step, the SB residue was filtered through a sintered glass crucible and washed with an ample amount of distilled water to a neutral pH. Secondly, the SB residue was further treated with PAA solution, which represented 50% of the initial bagasse weight, at 75 °C for 2.5 h. Finally, the residue was filtered and washed with an ample amount of distilled water.

2.4 *Bleaching procedure*

In order to bleach various SB samples, sodium chlorite solution (1.4% w/v) was mixed with the SB sample at a ratio of 1:20 (SB:$NaClO_2$ solution) and 0.5 ml glacial acetic acid to adjust the reaction pH to 4. The reaction was performed for 1 h at 75 °C [10].

2.5 *Fourier Transform infrared (FTIR)*

Various samples were subjected to an ATR-FTIR spectrometer (VERTEX 80, Bruker, Germany) combined with Platinum Diamond ATR at the mid IR region of 4000–400 cm^{-1} with a resolution of 4 cm^{-1}.

2.6 *X-Ray Diffraction (XRD)*

Various samples were subjected to an X-ray diffractometer (XRD-6000, Shimadzu, Japan) at an electric current of 15 mA, and voltage of 30.0 kV. Copper Kα radiation was implemented under a scanning range of 4.0 to 70.0 degrees and a scanning speed of 2° (2θ) min^{-1}. The CI was calculated using Equation (1)

$$CI\ (\%) = \frac{(I_{002} - I_{am})}{I_{002}} \times 100 \quad (1)$$

Where I_{002} and I_{am} are the maximum intensity of the 002 peak (around 2θ of 22°) and the minimal depression of the amorphous structure (around 2θ of 18°), respectively.

2.7 *Color*

Various samples were subjected to a Chroma meter CR-410 (Konica Minolta, Japan) to determine their color in terms of L* (lightness/darkness, a* (redness/greenness) and b* (yellowness/blueness). To estimate the whiteness index (WI), the subsequent Equation (2) was utilized [10]:

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

2.8 *Water holding capacity (WHC)*

The WHC of various samples was evaluated according to the method of Zheng and Li [11]. One gram of the sample and 30 mL of distilled water were vigorously blended at ambient temperature for 2 h. Subsequently, the wet sample was separated on a muslin cloth to drain out the excessive water. The retained portion over the muslin cloth was accurately transferred to a petri dish and weighed (initial wet weight), then dried at 105 °C to a constant final dry weight. WHC was calculated as Equation (3):

$$WHC\ (g/g) = \frac{(initial\ wet\ weight - final\ dry\ weight)}{final\ dry\ weight} \quad (3)$$

2.9 *Oil binding capacity (OBC)*

The OBC of various samples was determined according to the method of Zheng and Li [11], where 1 g of SB sample (initial weight) and 20 mL of corn oil were blended together and kept at ambient temperature for 1 h. Afterward, the blend was centrifuged for 10 min at 1500×g. The supernatant was poured out and the sediment was obtained by filtering over a muslin cloth and weighted (sediment weight). OBC was calculated as outlined in Equation (4)

$$OBC\ (g/g) = \frac{(sediment\ weight - initial\ weight)}{initial\ weight} \quad (4)$$

2.10 *Reduction of fiber particle size*

In order to reduce the conventional particle size of the obtained fiber samples, a ball milling process (PQ-N_2 Planetary Ball Mill, Across International, USA) was implemented. Sample powder (25 g) was ball milled at 4000 rpm in a 200 mL agate vessel containing 130 numbers of zirconia beads (75 beads 0.5 mm diameter, 30 beads 1.0 mm diameter and 25 beads 1.5 mm diameter). Different samples were milled for 90 min to obtain fine powder which was then sieved through 80 diameter mesh (<500 μm).

2.11 *Dialysis of glucose*

The effect of various samples on the dialysis of glucose was determined according to the method of Ou *et al*., [26] with slight modifications. In a dialysis bag with a membrane of 12000 cut-off molecular weight, 0.5 gm of the sample was added to 25 mL glucose solution (50 mmol/L) and dialyzed versus distilled water (80 mL) at 35 °C. The dialysate glucose content was estimated using glucose assay kits after an incubation interval of 60 min. A dialysis bag without a sample was used as a control test.

2.12 *α-Amylase inhibition activity*

The α-Amylase inhibition activity of various samples was determined using Biovision enzymatic kits No.

K482-100 (Bivision, Milipita, CA, USA). The sample was mixed with various volumes of distilled water to prepare different concentration mixtures which were used to assess the sample concentration that inhibited 50% of α-amylase activity (IC50). In a similar way, different concentrations of acarbose were prepared to determine the concentration that inhibits 50% of α-Amylase activity (IC50).

2.13 *Statistical analysis*

All results were expressed as mean ± standard deviation values. XLSTAT software version 2019.2.2.59614 (Addinsoft, USA) was used to perform one-way ANOVA followed by Tukey's test (p-value < 0.05) and Pearson's correlation to analyze the obtained data.

3 Results and Discussion

3.1 *Holocellulose yield, lignin and holocellulose percentages*

The effect of various treatments before and after bleaching treatment on holocellulose yield, lignin and holocellulose content was investigated and the obtained results are shown in Figure 1. The holocellulose is the total polysaccharide fraction (hemicellulose and cellulose) that remains after removing lignin from lignocellulosic materials [27]. The holocellulose yield for various treatments significantly (p-value < 0.05) differed. The highest significant holocellulose yield (78.67 ± 2.20%) was obtained when NaOH treatment was implemented alone. On the other hand, H_2SO_4+NaOH treatment exhibited the lowest significant holocellulose yield (35.97 ± 0.62%). In their work on delignifying sugarcane bagasse, Laluce *et al*., [22] Nath *et al*., [28] found that holocellulose yield for NaOH treatment (1% w/v) at 100 °C for 1 h and at 50 °C for 2 h was 60.17 and 72.05%, respectively. Rocha *et al*., [29] found that the holocellulose (cellulose and hemicellulose) yield for sulfuric acid treatment (1% w/v H_2SO_4, 120 °C, 10 min) followed by NaOH treatment (1.5% w/v NaOH, 100 °C, 1 h) was 37.77%. In addition, under treatment conditions of 1% w/v H_2SO_4 at 121 °C for 80 min sulfuric acid treatment reduced the holocellulose yield of treated SB to 36.46%, which was further reduced to 20.30% after NaOH treatment (0.5% w/v NaOH, 80 °C, 90 min)

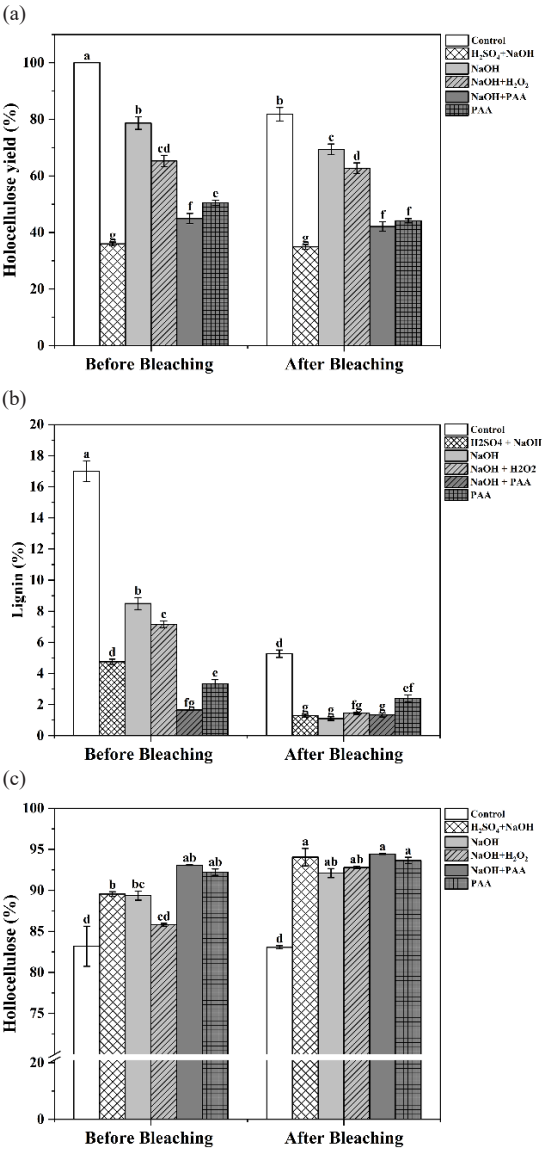

Figure 1: Effect of various treatments on (a) holocellulose yield (%), (b) lignin (%) and (c) holocellulose (%).

[30]. This decrease in the holocellulose yield associated with H_2SO_4 + NaOH could be attributed to the severity of treatment conditions (high temperature and longtime) compared to other treatments. Moreover, the data in Figure 1(a) reveal that the bleaching treatment led to a further significant decrease in the holocellulose yield of bleached samples; however, the yield of samples after the bleaching treatment followed an identical pattern to that observed after various treatments.

Data in Figure 1(b) show that the lignin content of untreated SB (control) was 16.99 ± 0.66%, which confirms previously published data related to the lignin content of SB [15]. All treatments and bleaching treatment significantly resulted in a decrease in lignin content. Indeed, NaOH+PAA treatment exhibited the most significant decrease in lignin content (1.65 ± 0.006%) with the highest delignification degree of 90.29%. These results are in agreement with those of Han *et al*., [25] who found that NaOH+PAA treatment decreased the lignin content from 24 ± 1.20 in raw SB to 1.15 ± 0.08 with a delignification degree of 95.21%. They ascribed the high delignification degree associated with NaOH+PAA treatment to the formation of HO+ (hydroxonium ions) from PAA under an acidic medium. As electron sites of lignin react with these ions causing electrophilic substitution with oxidative demethylation, displacement of side chains, ring hydroxylation, oxidative ring opening, cleavage of β-aryl ether bonds and epoxidation of olefin structure.

As shown in Figure 1(b), it is apparent that bleaching of treated and untreated SB samples significantly reduced their lignin content. The lignin content of the untreated sample dropped from 16.99 ± 0.66% to 5.27 ± 1.18% with delignification degree of 68.98%. Similarly, the lignin contents of treated samples were further reduced as a consequence of bleaching treatment and their delignification degree varied between 85.93% and 93.52%. These results agree with those of Rocha *et al*., [4] and Sompugdee *et al*., [10] who found that the bleaching treatment of previously alkaline treated SB fibers further decreased their lignin content. The effectiveness of bleaching treatment in removing lignin could be attributed to chlorine dioxide (ClO_2) that was generated from the $NaClO_2$ bleaching agent under acidic conditions. As, chlorine dioxide (ClO_2) caused lignin depolymerization, ring-opening and oxidizing ring-conjugated structures [31]. Moreover, looking at Figure 3(b), it is apparent that there were insignificant differences in lignin content between bleached samples treated by NaOH only or in sequential treatments. This phenomenon could be ascribed to the swelling of cellulose as a consequence of alkali treatment, which increase the porosity of the biomass and improve the elimination of lignin and hemicellulose [9].

In contrast to lignin content, the holocellulose content of various samples was significantly increased after the implementation of various treatments (Figure 1(c)). The holocellulose of the control sample was 83.18 ± 2.45%. This finding is consistent with that of Mobarak and Fahmy [32] who found that holocellulose content of raw SB was 82.4%. Guimarães *et al*., [33] reported that the holocellulose content of SB ranged between 50–80%. The same data show that NaOH+PAA treatment exhibited the highest retention of holocellulose contents (93.07 ± 0.05%) and the lowest lignin content (1.65 ± 0.006%). Furthermore, except control sample, bleaching treatment significantly increased the retention of holocellulose content for all treated samples. Data in Figure 1(b) and (c) show that the increase in holocellulose content was associated with a decrease in lignin content. The correlation between holocellulose and lignin contents was significant (p-value < 0.0001) and negative (r = –0.8113), which further supports the preceding association between lignin and holocellulose.

3.2 *Fourier Transform Infrared Spectra (FTIR)*

Various samples were subjected to FTIR spectroscopy to investigate their chemical structure and the obtained spectra are shown in Figure 2. The obtained spectra are almost comparable and exhibit the ordinary attributes of SB absorption peaks. The untreated SB sample shows the following spectral bands: 3170–3490 cm^{-1} for O-H stretching hydrogen bonds, 2850–2970 cm^{-1} for C-H stretching, 1730 cm^{-1} for C-O stretching vibration for the acetyl and ester linkages in lignin, hemicellulose, pectin etc., 1620–1649, 1512 cm^{-1} associated with the aromatic ring present in lignin and absorbed water, 1250 cm^{-1} are C–O out of plane stretching due to the aryl group in lignin [34] and 832 cm^{-1} for lignin C-H stretching. In addition, the same sample spectrum shows sharp bands at the range of 850–1200 cm^{-1} which are related to the structure of cellulose and hemicellulose [16].

IR spectra of treated samples (Figure 2(a)) show a reduction of spectral bands at 1250 cm^{-1} and did not show spectral bands at 832, 1458, 1512, 1595, 1620 cm^{-1} that characterize the functional groups of lignin [21]. On the other hand, the same spectra showed an increase in spectral bands at 897, 993, 1031, 1050, 1104, 1160, 1200, 1317 and 1371 cm^{-1} that could be related to cellulose [16], [35]. These results confirm the ability of used treatments to remove lignin and

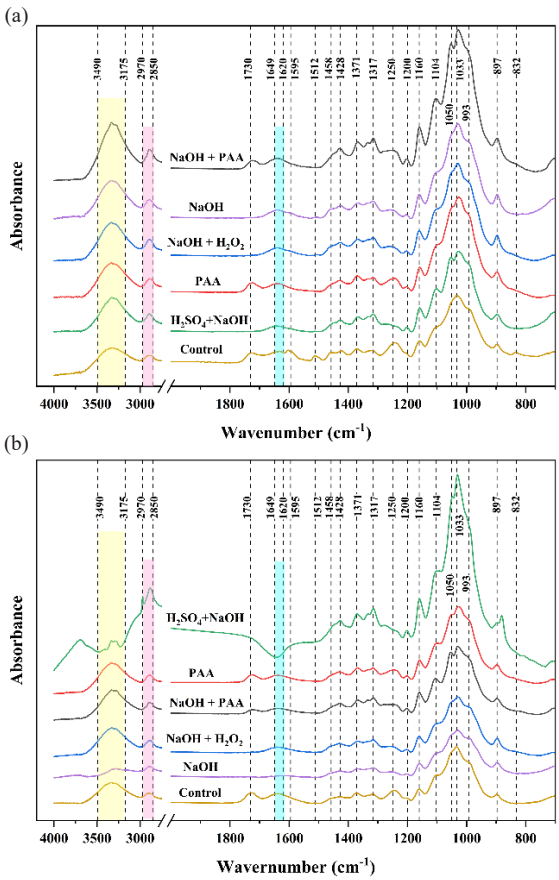

Figure 2: FT-IR spectra of (a) treated and control SB samples (b) treated and control SB samples after bleaching.

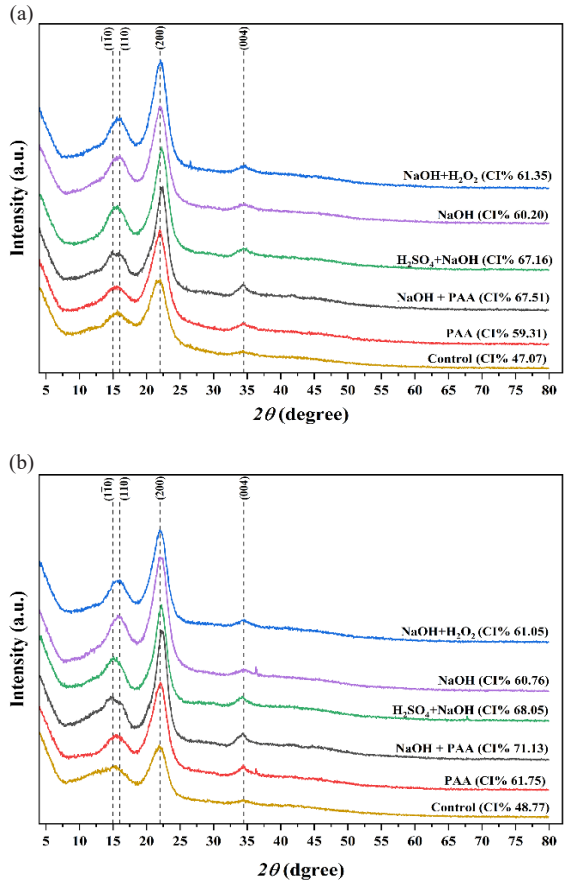

Figure 3: X-ray diffractograms of (a) treated and control SB samples (b) treated and control SB samples after bleaching (Crystallinity Index percentage (CI%).

purify the obtained fibers. Except the IR spectra of PAA and NaOH+PAA, IR spectra of other treatments did not exhibit bands at 1730 cm^{-1}, which indicate their abilities to break the linkages between lignin and hemicellulose [16].

Data in Figure 2(b) show that after bleaching treatment, the untreated sample (control) exhibited a reduced spectral band at 1250 cm^{-1} while the spectral bands at 832 and 1512 cm^{-1} were not found. These results show the effectiveness of sodium chlorite in removing lignin. Meanwhile, there are no observed changes in the IR spectra of treated samples except at 3175–3490 cm^{-1} for H_2SO_4+NaOH and NaOH IR treated samples spectra. The flattened bands at this range reveal alteration in O–H bonds [36]. Gil-López *et al*., [9] observed a similar flattened band in the IR spectrum of SB treated by H_2O_2 at the same wavenumber range. They attributed the existence of these bands to the formation of free radicles that attack macromolecules leading to this alteration.

3.3 *X-ray diffraction (XRD)*

To assess the effect of various treatments on the crystallinity and polymorph of cellulose, treated and untreated as well as bleached samples were subjected to X-ray diffraction (XRD) analysis and the obtained diffractograms are shown in Figure 3. All samples showed similar and identical X-ray diffractograms to that of cellulose I crystal lattice. These XRD patterns display pronounced peaks around 2θ angles of 15°, 16°, 22.5° and 34.6°, which are assigned to crystallographic

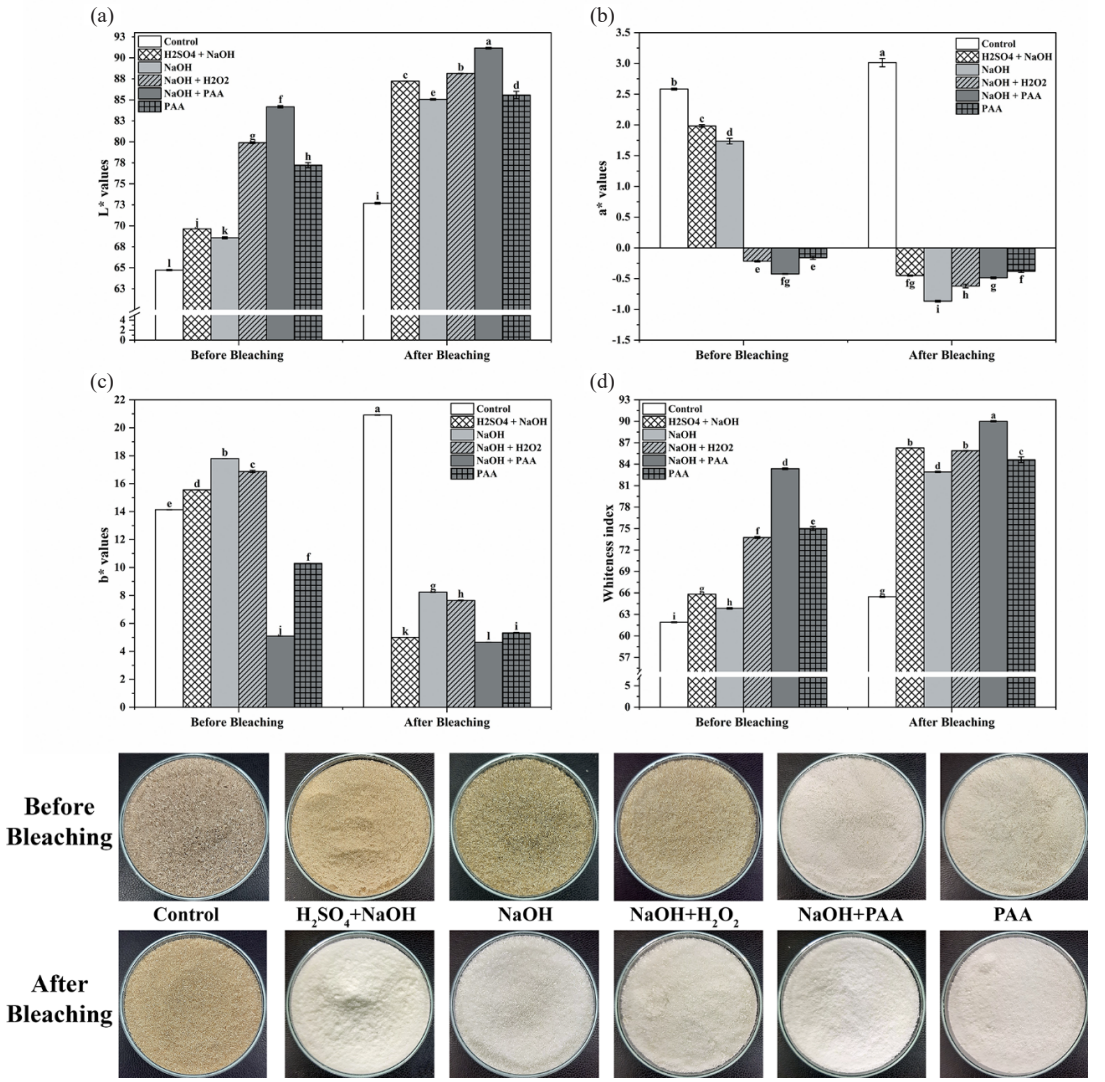

Figure 4: Color parameters of various treated samples: lightness (L* values) (a), redness/greenness (a* values) (b), yellows/blueness (b* values) (c) and whiteness index [d] and their photographs.

planes of $1\bar{1}0$, 110, 200 and 004, respectively [25], [34], [37].

These results reveal that the treatments and subsequent bleaching treatment did not alter the cellulose polymorph. In comparison to untreated samples, all treated samples exhibit higher crystallinity indexes (CI%). This increase in CI% could be ascribed to the elimination of the amorphous portion (hemicellulose and lignin) [25], [34], [37], [38]. On the other hand, bleaching of either treated or untreated (control) samples had little effect on the CI%.

3.4 *Color*

The color of cellulose fibers is highly correlated to their impurities content and it is a determinant factor for their final utilization in various food products [39]. Figure 4 illustrates the photographs of various samples and their color parameters in terms of L*, a* and b* values as well as their whiteness index (WI). All treatments significantly (p-value < 0.05) increased the lightness (L* values) of various samples and the bleaching step also led to a further significant (p-value < 0.05)

increase in the lightness. Despite the control sample that exhibited positive a* values (redness) either before or after the bleaching step, all treated samples significantly differed from control ones and showed negative a* values (greenness) after the bleaching step. In comparison to the control sample, samples treated with NaOH+PAA and PAA alone exhibited significant (p-value < 0.05) low b* (yellowness) values, whereas other treatments led to a significant increase in sample yellowness. On the other hand, further treatments of treated samples with sodium chlorite resulted in an additional decrease in sample yellowness.

All the aforementioned changes in color parameters of treated and subsequent bleached samples are obviously highlighted in the WI of various samples. Clearly, as shown in Figure 5, all treatments led to a significant (p-value < 0.05) increase in WI and the subsequent bleaching step further increased WI of various samples. Indeed, the sample treated with NaOH + PAA exhibited the highest significant (p-value < 0.05) WI either before or after bleaching treatment. The correlation between WI and lignin content of various samples was significant (p-value < 0.0001) and negative (r = –0.7830). Thus, it could be attributed this increase in WI to the ability of various treatments to remove lignin which was dramatically decreased after bleaching treatment. These results agree with the findings of other studies, in which WI of various lignocellulosic materials such as SB [1], [40], Oil palm Mesocarp [41], and rice husks [42] increased after consecutive treatment and bleaching. Throughout these studies, the increase in WI was ascribed to the ability of these treatments and the bleaching process to remove lignin as well as hemicellulose and other impurities.

3.5 *Water holding capacity (WHC) and oil binding capacity (OBC)*

Cellulose, hemicellulose, and lignin are the major constituents of SB that govern its ability to absorb either hydrophilic or hydrophobic substances. These constituents affect the nature of bagasse sites (hydrophilic and hydrophobic sites) that attract these substances. In addition, it was reported that the architectural system spaces of the bagasse could play an important role in trapping these substances [43]. Data in Figure 5(a) show that the samples treated with NaOH and NaOH + H_2O_2 exhibited a significant

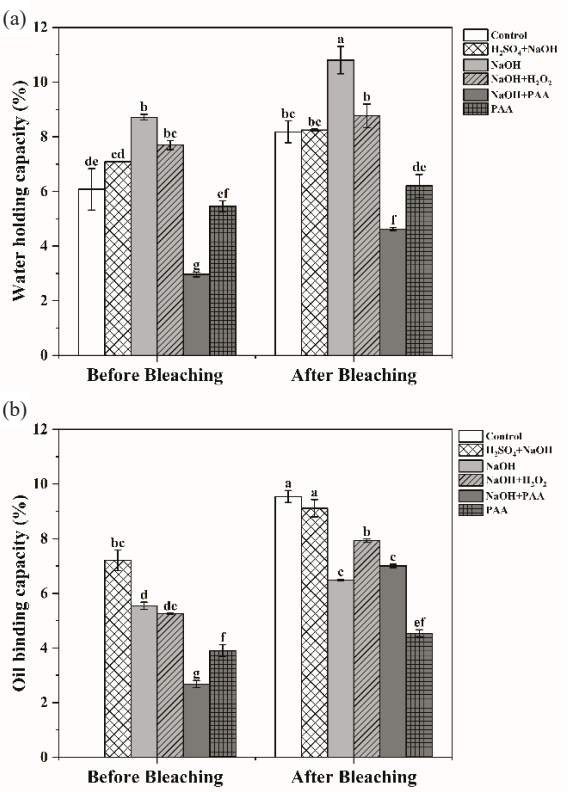

Figure 5: Effect of various treatments on the water holding capacity (%) (a) and oil binding capacity (%) (b).

(p-value < 0.05) increase in WHC in comparison to the control sample, whereas the NaOH+PAA treated sample showed a significant decrease in WHC. Regarding the WHC of the unmodified (control) sample, the further bleaching treatment significantly increased the WHC of treated samples, while the only NaOH treated sample showed a significant (p-value < 0.05) higher WHC than that of the bleached control sample. On the other hand, the PAA and NaOH+PAA bleached treated samples exhibited lower WHC than that of the bleached control sample. The reason behind the reduction of WHC associated with treatments involved PAA might be attributed to extensive structural changes/ breakdowns that occurred due to the strong oxidizing activities of PAA and its ability to generate free radicals [9], [44].

OBC can be loosely described as the amount of oil that could be absorbed by one gram of dry sample in the presence of oil under the influence of centrifugal force [9]. The relationship between OBC

and oil/fat absorption through the gastrointestinal tract and their subsequent excretion in the stool has been reported [8]. As shown in Figure 5(b), the unmodified (control) sample did not exhibit any ability to absorb oil whereas all treated samples exhibited various degrees of OBC. Furthermore, subsequent bleaching treatment led to a significant increase in the OBC of various samples. Indeed, the bleached control sample and H_2SO_4+NaOH bleached treated sample exhibited the highest significant OBC. These improvements in WHC and OBC could be attributed to the effect of treatments and bleaching step on the cellulose conformational structure causing the exposure of binding sites or functional groups to retain oil and water [10]. In their work, Kim *et al*., [13] found that alkaline treated SB fiber showed a reduced OBC and increased WHC. Moreover, Sangnark and Noomhorm [12], found that alkaline hydrogen peroxide treatment of SB increased its WHC and OBC. They ascribed this increase in OBC and WHC to the degradation of lignin that occurred during the treatment. In addition, Sompugdee *et al*., [10] mentioned that the increment in OBC after SB treatments could be attributed to the increase in crystallinity of cellulose (hydrophobic portion).

3.6 *The effect of size reduction on the physicochemical and functional properties*

Prior studies have found a direct relationship between insoluble dietary fiber particle size and their functional properties. However, there are discrepancies in these relationships, as some studies showed a positive relationship and others showed a negative relationship between OBC and WHC and low particle size fibers [45]. To explore the effect of size reduction on the physicochemical and functional properties of delignified fibers, the most appropriate samples were chosen based on the subsequent criteria: firstly, all samples exhibited lignin content higher than 2% were excluded. Secondly, the OBC and WHC of the remaining samples were inspected and the samples that showed values lower than 4% were also excluded. The samples that met the preceding criteria were NaOH, H_2SO_4 + NaOH, NaOH + H_2O_2 and NaOH + PAA treated bleached samples. Data listed in Table 1 reveal that pulverizing the samples significantly (p-value < 0.05) reduced their WHC and OBC.

Moreover, there were no significant (p-value > 0.05) differences in WHC and OBC between pulverized samples. These results are in line with those of Chen *et al*., [46] who found that the WHC and OBC of alkaline hydrogen peroxide treated rice straw fibers decreased as the fiber particle size decreased. They mentioned that these reductions in WHC could be attributed to the alteration of hydration characteristics of dietary fiber because of milling. Indeed, under certain circumstances, milling might alter and collapse the fiber matrix that absorbs water leading to low WHC.

The reduction in postprandial blood glucose associated with DF could be ascribed to their effect on retarding the diffusion of glucose in small intestinal and lowering the breakdown of starch into glucose units as a result of increasing small intestinal content viscosity and inhibition of α-amylase activity, respectively [47]. Data listed in Table 1 show that almost all tested samples either pulverized or did not exhibit significantly (p-value < 0.05) lower glucose levels in dialysate than that of the blank. These results indicate the ability of tested samples to delay the diffusion of glucose through the dialysis membrane. Moreover, the same results reveal that the pulverization of tested samples significantly (p-value < 0.05) decreased their ability to delay the diffusion of glucose. The correlation between glucose concentration in dialysate and WHC of various samples was significant (p-value < 0.0094) and negative (r = –0.8777). Therefore, the low glucose diffusion rates associated with unpulverized samples might be attributed to their incremental effect on solution viscosity [41].

The viscosity of any fiber depends on its water absorption capacity [48]. In contrast to the previous results, the pulverization of the tested samples significantly (p-value < 0.05) increased their inhibitory effect against α-amylase activity. Indeed, almost all pulverized samples exhibited higher inactivation activity (low IC_{50} values) against α-amylase in comparison to unpulverized ones. These results reflect those of Chen *et al*., [47] who found that reducing the particle size of DF increased their α-amylase inhibitory activity. They ascribed this increase in inhibitory activity against α-amylase to structural changes associated with a reduction in particle size. These changes lead to an increase in surface area and the number of fiber network pores which might not only embed

Table 1: Effect of the size reduction on the physicochemical and functional properties of treated bleached fibers

Sample	Glucose in Dialysate after 60 min (mg/dL)	α-Amylase Inhibition Activity (IC_{50} µg/ml)	WHC	OBC
	$140.16^{a} \pm 0.58^{\beta}$	$12.29^{g} \pm 0.67^{\zeta}$	-	-
NaOH	$93.85^{d} \pm 2.9$	$63.79^{f} \pm 5.06$	$10.81^{a} \pm 0.50$	$6.49^{e} \pm 0.03$
NaOH + H_2O_2	$87.29^{d} \pm 6.96$	$472.69^{a} \pm 14.28$	$8.77^{b} \pm 0.43$	$7.93^{b} \pm 0.06$
NaOH + PAA	$119.26^{b} \pm 4.06$	$191.70^{e} \pm 9.43$	$4.62^{e} \pm 0.06$	$7.01^{c} \pm 0.06$
H_2SO_4 + NaOH	$96.92^{cd} \pm 8.4$	$403.37^{b} \pm 13.65$	$8.25^{b} \pm 0.04$	$9.12^{a} \pm 0.32$
Reduced size fibers (<500 µm)				
NaOH	$110.24^{bc} \pm 1.16$	$48.20^{f} \pm 4.21$	$2.60^{d} \pm 0.11$	$1.71^{d} \pm 0.06$
NaOH + H_2O_2	$123.46^{b} \pm 0.14$	$297.58^{c} \pm 10.46$	$2.67^{d} \pm 0.04$	$1.86^{d} \pm 0.25$
NaOH + PAA	$124.08^{b} \pm 1.02$	$234.40^{d} \pm 9.04$	$2.41^{d} \pm 0.10$	$1.91^{d} \pm 0.13$
H_2SO_4 + NaOH	$143.44^{a} \pm 1.16$	$211.41^{de} \pm 9.76$	$1.87^{d} \pm 0.12$	$1.95^{d} \pm 0.04$

Means followed by different lowercase letters in the same columns significantly differ based on Tucky`s test at p-value < 0.05
$^{\beta}$ Glucose in dialysate after 60 min for blank
$^{\zeta}$ α-Amylase inhibition activity in terms of IC_{50} for Acarbose Standard

starch and enzyme into these pores but also expose α-amylase to inhibiting substances on the extended surface of the fiber causing a decrease in the α-amylase activity.

4 Conclusions

The current study has examined the impact of NaOH, NaOH + H_2O_2, NaOH + H_2SO_4, PAA and NaOH + PAA treatments and the subsequent bleaching treatment on the physicochemical, structural, and functional properties of sugarcane fiber. Among the used treatments, NaOH + PAA treatment exhibited superior efficiency in removing lignin. NaOH + PAA treated sample contained the lowest lignin content and highest holocellulose content. In addition, the same treatment produced fiber that had the highest WI value. XRD analysis showed that X-ray diffractograms of all samples were identical to that of cellulose I and CI% of NaOH + PAA treated sample was the highest compared to other samples confirming the preceding changes in constituent's content. FTIR spectra of all treated and bleached samples did not show any spectral bands at 832, 1512, 1595, and 1620 cm^{-1} that related to functional groups of lignin indicating the effectiveness of used treatments and sodium chlorite in removing lignin. NaOH and H_2SO_4 + NaOH treated samples showed the highest WHC and OBC, respectively; however, NaOH + PAA treated samples showed the lowest WHC and OBC. Moreover, the effect of fiber particle size reduction on the physicochemical and functional properties of treated bleached samples was investigated. The reduction of fiber particle size decreased its WHC and OBC and increased the glucose diffusivity through the dialysis membrane and inhibitory activity against α-amylase. In general, therefore, it seems that the physicochemical and functional properties of SB fiber are influenced by implemented treatment which could be taken into account during the development of foods with specific functions. The current research has only examined the effect of used treatments on the physicochemical and functional properties of DF in a comparative manner at fixed treatment parameters. Thus, further studies need to be carried out to investigate the effect of treatment parameters manipulation on the properties of DF, especially NaOH + PAA treatment.

Author Contributions

G.S.M.A.: conceptualization, data curation, formal analysis, investigation, methodology, writing – original draft, writing –review & editing; A.M.A.E.: conceptualization, formal analysis, methodology, supervision, writing –review & editing; M.K.S.M.: conceptualization, formal analysis, methodology, supervision, writing –review & editing; E.A.A.: conceptualization, data curation, formal analysis, methodology, visualization, writing –original draft, writing –review & editing.

Conflicts of Interest

The authors declare no conflict of interest.

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