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Hydrothermal Extraction of Cellulose from Sugarcane Bagasse for Production of Biodegradable Food Containers

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Abstract: Sugarcane bagasse (SCB), an organic waste generated during sugar and ethanol production, is a potential biomass source with a high cellulose content. In this study, cellulose was extracted from SCB using a hydrothermal method with various types of solvents, following which the extracted materials were used for food container production. An alkali solvent—sodium hydroxide (NaOH)—and organic acids—citric acid and formic acid—were included as extractive solvents at two different concentrations (0.25 M and 2.0 M). Hydrothermal extraction with the alkali solvent demonstrated higher cellulose extraction abilities (67.7–74.0%) than those with the acids (52.5–57.3%). Using a low alkali concentration in the hydrothermal extraction (H-NaOH_low) demonstrated a cellulose extraction ability near that when using a high alkali concentration in the conventional boiling method (B-NaOH_high): 67.7% and 70.5%, respectively. Moreover, cellulose extracted with H-NaOH_low had better mechanical properties than that from B-NaOH_high, likely due to fewer defective fibers in the former. A high alkali concentration led to vigorous reactions that damaged the cellulose fibers. Thus, hydrothermal extraction has the benefit of using fewer chemicals, leading to a lower environmental impact. In addition, H-NaOH_low fibers were employed for food container production, and it was found that the obtained product has excellent properties, comparable to those of commercial containers.

Keywords: cellulose; extraction; food container; hydrothermal

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1. Introduction

The recycling of residues from agricultural and industrial sectors is attractive due to increasing environmental concerns. In general, most residues from the agricultural sector are lignocellulosic materials, which are considered the future of non-food materials. They mainly consist of three natural polymers, including lignin, cellulose, and hemicellulose. Among these, cellulose is the most abundant in the form of microfibrils and is well-known for its turnover capacity, biocompatibility, and biodegradability [1]. Therefore, cellulose has been extracted from a variety of residual biomasses for various applications, including paper, building materials, automotive materials, textiles, food, and packaging industries [2].

Cellulose extraction methods include chemical [3], biological [4], hydrothermal [5], and microwave (MW)-assisted methods [6,7], all aiming to remove hemicelluloses and lignin from biomass feedstocks. The method of extracting cellulose from these materials should also conform to environmental standards to align with environmental concerns regarding the use of natural materials (agricultural residues).

The hydrothermal method is recognized as an environmentally friendly process; thus, it is appropriate to be used for the abovementioned purpose. It uses an aqueous solution

as a reaction system within a closed vessel and then creates special high-temperature and high-pressure conditions [8]. As a result, no or fewer chemicals are used in the hydrothermal process.

Sugarcane bagasse (SCB)—a fibrous waste material with a high cellulose content (40–50%)—is usually used for the production of cellulose [7]. Thailand is the fourth-largest producer of sugarcane in the world, with an annual production of up to 134.9 million tons (2017/18 milling season) [9]. Therefore, a high amount of sugarcane bagasse is generated annually and may cause an environmental impact if inappropriately managed, such as by burning, which leads to dust pollution, groundwater contamination, and greenhouse gas emissions. Therefore, the hydrothermal extraction of cellulose from SCB may be an attractive method for SCB waste management due to its lower environmental impact and cost-effectiveness compared with other environmentally friendly methods such as steam explosion [10] and CO_2 impregnation [11]. Nevertheless, the efficiency of hydrothermal extraction should be considered, as it has a lower extraction capability compared with conventional chemical methods. Hence, the improvement of its efficiency by introducing a small amount of chemicals or using non-hazardous active substances should be investigated and elucidated.

Considering the benefits of using an environmentally friendly method for cellulose extraction with fewer chemicals, hydrothermally extracted cellulose could be used in applications that require health safety standards, such as food containers and packaging. Some reports in the literature have employed hydrothermal methods for cellulose extraction from biomass with various extractive solvents, including fresh water [12], seawater [13], hydrochloric acid [14], and an alkali [15,16]. However, no study has focused on employing various types of solvents in a hydrothermal method and, consequently, using the extracted materials for food container production.

Therefore, in this study, hydrothermal extraction was carried out to obtain cellulose from SCB using organic acids (i.e., formic acid and citric acid), compared with the use of an alkali agent (NaOH). The extracted cellulose was then employed for food container production. The properties of the cellulose and the finished products were analyzed using appropriate techniques and instruments.

2. Results and Discussion

2.1. Extraction Ability

The composition of the SCB was first determined to compare the extraction ability of each process. The compositions of the obtained samples were determined as shown in Table 1. The SCB was composed of 47.8% cellulose, 26.4% hemicelluloses, 18.1% lignin, and other components in trace amounts. These values were close to those reported in the reviewed literature [17,18]. All samples from the various extraction methods showed higher cellulose contents than SCB but lower hemicellulose and lignin contents. This suggests that all the methods have the ability to extract cellulose from the materials by removing hemicelluloses and lignin.

The B-NaOH_low and B-NaOH_high samples underwent conventional extraction methods involving boiling the materials with two different concentrations of alkali solvent (0.25 and 2 M NaOH). In general, the hydroxide ions (-OH) of an alkali can weaken the hydrogen bonds between cellulose and hemicellulose, as well as disrupt the ester linkages between hemicelluloses and lignin. In addition, they can break the glycosidic bonds between the monosaccharide groups inside hemicelluloses. This increases the solubilization of both lignin and hemicellulose fragments in the alkali solution, and boiling can enhance this solubilization by increasing the energy supplied to the system [19,20]. The higher NaOH concentration caused a higher extraction ability, with extraction abilities of

70.5% and 60.4% for B-NaOH_high and B-NaOH_low, respectively. This was due to the higher amount of hydroxide ions in the system. The amounts of hemicelluloses and lignin were also lower with a higher alkali concentration.

Table 1. Chemical compositions of samples extracted with various methods.

Sample	Extraction			Composition (wt%)		
	Method	Substance	Concentration (M)	Cellulose	Hemicelluloses	Lignin
SCB	-	-	-	47.8	26.4	18.1
B-NaOH_low	Boiling	NaOH	0.25	60.4	16.1	11.0
B-NaOH_high		NaOH	2	70.5	13.1	8.3
H-NaOH_low	Hydrothermal	NaOH	0.25	67.7	17.3	11.2
H-NaOH_high		NaOH	2	74.0	14.8	8.0
H-Citric_low		Citric acid	0.25	54.2	20.2	13.4
H-Citric_high		Citric acid	2	57.3	18.2	12.9
H-Formic_low		Formic acid	0.25	52.5	19.3	13.2
H-Formic_high		Formic acid	2	57.1	18.5	12.7
H-Water		Water	-	50.1	20.5	14.3

A hydrothermal process was used for comparison to increase the extraction ability even with a low alkali concentration, as seen in H-NaOH_low and H-NaOH_high. The hydrothermal method increased the extraction ability for both NaOH concentrations, as previously observed [21]. A significant increase of approximately 10% from 60.4 to 67.7 was found at the low NaOH concentration (0.25 M). This suggests that the hydrothermal conditions had a greater influence at the lower NaOH concentration. Thus, this indicates the benefit of the hydrothermal method using fewer chemicals compared with the conventional method.

Less-toxic acids (i.e., citric acid and formic acid) were also employed in the hydrothermal extractions, resulting in the H-Citric_low, H-Citric_high, H-Formic_low, and H-Formic_high samples. The roles of acids in cellulose extraction are the disruption of the lignocellulosic matrix by cleaving glucosidic bonds and then the transformation of polysaccharides into sugars with smaller molecules (mono- and oligosaccharides). The acids also hydrolyze hemicelluloses and lignin, increasing their solubilization [22–24]. Citric acid and formic acid (weak organic acids) usually have lower activity than strong acids such as HCl and H_2SO_4. However, the results show that both acids had a relatively high extraction ability, ranging from 52.4 to 57.0%. This reveals that hydrothermal methods can enhance the extraction activity of weak acids and, therefore, increase the chance of using less-toxic acids instead of highly toxic acids in cellulose extraction, thus lessening the environmental impact. Increasing the acid concentration only slightly increased the extraction ability for both citric acid and formic acid. Therefore, a low acid concentration can be used for cellulose extraction with the hydrothermal method.

In the H-water sample, only water was used as the extractive solvent in the hydrothermal method, and it demonstrated a cellulose extraction ability of up to 50.1%. Under hydrothermal conditions, water turns into vapor, which can disrupt the cellulose structure, hydrolyze the hemicellulose component into smaller sugar molecules, and depolymerize the lignin component into phenolic compounds [25]. This indicates the efficiency of the hydrothermal system, which can convert water into a capable solvent for cellulose extraction.

The hydrothermal treatment of cellulose, particularly under acidic conditions, can result in depolymerization, forming various soluble byproducts, including carboxylic acids, α-carbonyl aldehydes, furanic compounds, and carbocyclic compounds [26]. This leads to lower cellulose yields with the hydrothermal extractions. In addition, these byproducts

might remain in the extracted cellulose; thus, several washings with water were performed to remove these water-soluble compounds.

2.2. Cellulose Characterization

Cellulose samples (i.e., H-Formic_high, H-Citric_high, and H_NaOH_low) were selected to be applied in food container production due to their high cellulose concentrations and low toxicity remaining after hydrothermal extraction. For comparison, B-NaOH_high—representing the cellulose sample extracted using the conventional method—was also selected for the application. All the selected samples were investigated for their cellulose properties compared with raw sugarcane bagasse (SCB) and commercial pure cellulose (cellulose).

The functional groups of the samples were determined with FTIR, and all obtained FTIR spectra are shown in Figure 1. All sample spectra exhibited cellulose's characteristic peaks, including O–H stretching between 3600 and 3200 cm^{-1}, C–H stretching between 3000 and 2800 cm^{-1}, C–O–C stretching at 1159 cm^{-1} and 897 cm^{-1}, and C–O stretching vibration at 1025 cm^{-1}. Nevertheless, those peaks were not clearly distinct for SCB and H-Formic_high due to their lower cellulose contents. The characteristic peaks of hemicelluloses and lignin were observed in SCB, H-Formic_high, and H-Citric_high; namely, the C=O stretching vibration between 1765 and 1715 cm^{-1} of hemicelluloses, and the C–O–C stretching vibration between 1250 and 1200 cm^{-1} and C=C stretching vibration between 1600 and 1500 cm^{-1} of lignin [27–29]. This suggests that the samples extracted with formic acid and citric acid still had high amounts of hemicelluloses and lignin, while the samples extracted with NaOH had small amounts, such that they could not be observed via FTIR. These results agree with the data in Table 2.

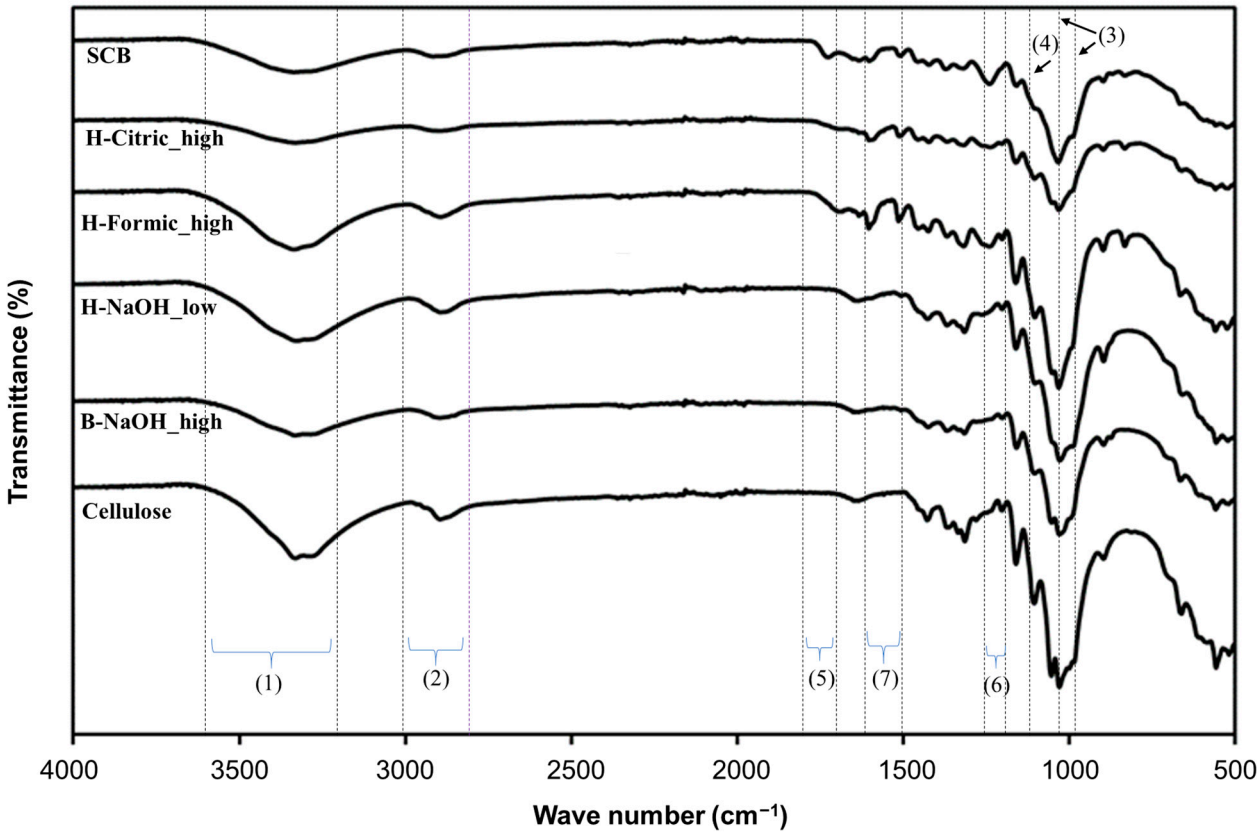


Figure 1. FTIR spectra of the samples: (**1**) O–H, (**2**) C–H, (**3**) C–O–C, (**4**) C–O, (**5**) C=O, (**6**) C–O–C, and (**7**) C=C stretching vibrations.

Table 2. Mechanical properties of sheets obtained from sugarcane bagasse-extracted cellulose.

Sample	Tensile Strength (MPa)	Elongation at Break (%)	Tear Strength (N)
B-NaOH_high	16.3 ± 2.0	4.0 ± 0.7	21.4 ± 2.6
H-NaOH_low	22.1 ± 2.4	4.1± 1.0	26.3 ± 2.1

The thermal stability of the cellulose samples was evaluated with TGA measurements and a DTG analysis, which measures the weight loss rate of a material as it is heated. The DTG peaks of the samples shown in Figure 2 indicate that a specific thermal event occurred during the heating process. All samples except cellulose had three DTG peaks but at different degrees for each sample. This indicates that there were three ranges of thermal decomposition during heating. In general, the thermal decomposition between 245 and 290 °C was the decomposition of hemicelluloses, that between 290 and 350 °C was the decomposition of cellulose, and that between 350 and 500 °C was the decomposition of lignin [30]. For SCB, all three peaks were not clearly separated due to a high number of other components. The cellulose peaks were seen for all extracted samples, with slight shifts from that of pure cellulose. These shifts could be derived from the change in the nature of the cellulose, probably due to MW and crystallinity. A lower MW can cause the DTG peak to shift to lower temperatures [31], attributed to the increased mobility and volatility of smaller molecules. In this study, the DTG peaks for cellulose were lower in H-Citric_high and H-Formic_low (both at 353 °C) compared with those in B-NaOH_high and H-NaOH_low (at 376 °C and 360 °C). This suggests that the hydrothermally treated samples with acids had a lower MW, presumably resulting from some degree of depolymerization of the cellulose. The lignin and hemicellulose peaks were clearly observed for H-Formic_high and H_Citric_high, but they were slightly observed for H-NaOH_low and B-NaOH_high due to their low contents.

The crystal structures of the cellulose samples were evaluated with an XRD analysis. The characteristic XRD peaks of cellulose generally appear at 2θ = 16.5°, 22.5°, and 34.3°, which correspond to the crystallographic planes of (110), (200), and (040), respectively [30,32]. The XRD patterns of all the cellulose samples are shown in Figure 3. All the samples exhibited XRD peaks that correlated with the characteristic peaks of cellulose. The cellulose crystallinity could be calculated from the XRD peaks, as shown in the figure. Pure cellulose exhibited the highest crystallinity of 78.9%, in agreement with previously reported values [33]. All the extracted samples had a higher cellulose crystallinity (62.4–72.7%) than the SCB (60.7%). This was due to the raw biomass comprising high contents of other components, including hemicelluloses and lignin, which disturb the crystallization process of the cellulose [34,35]. Moreover, the higher cellulose crystallinity of the extracted samples came from the combined recrystallization and hornification of the cellulose after the extraction [36,37]. The higher fractions of lignin and hemicelluloses in H-Formic_high and H-Citric_high, which correspond to the amorphous parts of the material, led to their lower crystallinity compared with the alkali-extracted samples.

The morphologies of the cellulose samples were determined using an SEM analysis. The SEM images of all the samples at the same two magnification setups, 500 and 20 μm, are shown in Figure 4. SCB showed a compact morphology, while the extracted samples exhibited a loose morphology with more exposed fibers. The alkali-extracted samples exhibited more segregated fibers compared with the organic acid-extracted samples (H-Formic_high and H-Citric_high). The latter samples still had high amounts of hemicelluloses, which help to hold the cellulose fibers together, as well as lignin, which keeps the fiber structure rigid and binds the hemicelluloses to the cellulose. These agglomerated fibers might not be suitable for food container production due to their potentially decreased strength and

increased porosity. When comparing two alkali-extracted samples, it can be seen that B-NaOH_high provided more damaged fibers than H-NaOH_low. The higher NaOH concentration led to vigorous reactions and, consequently, deformed the fibers' structures.

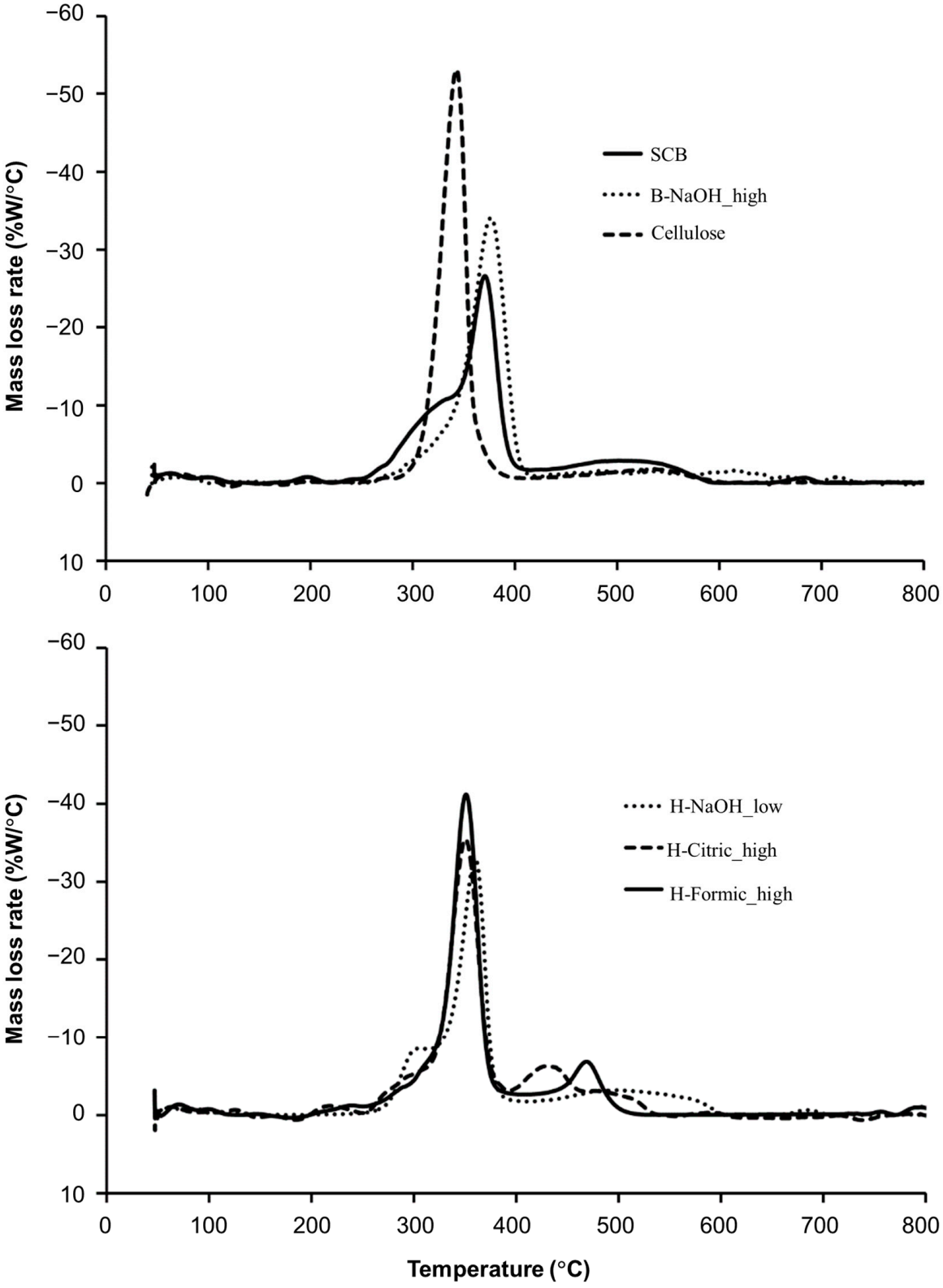


Figure 2. DTG curves of the samples.

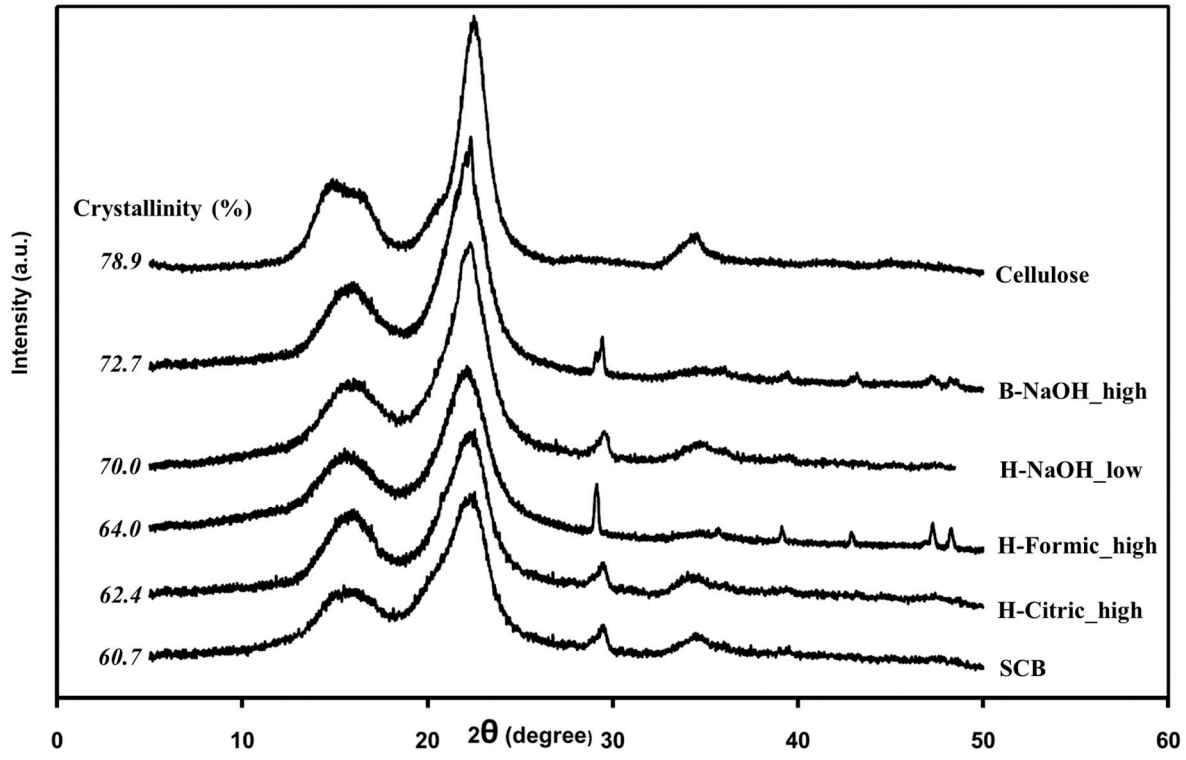


Figure 3. XRD patterns of the samples.

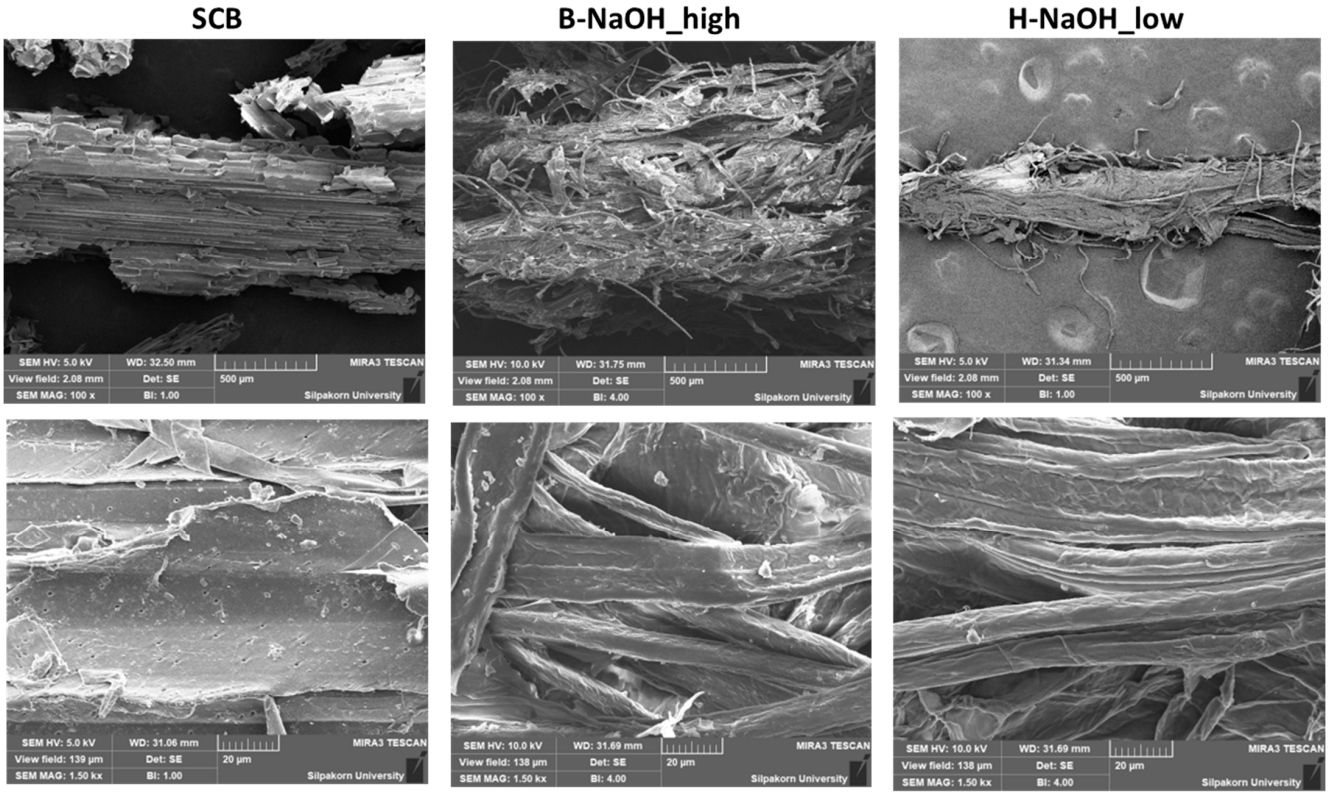


Figure 4. *Cont.*

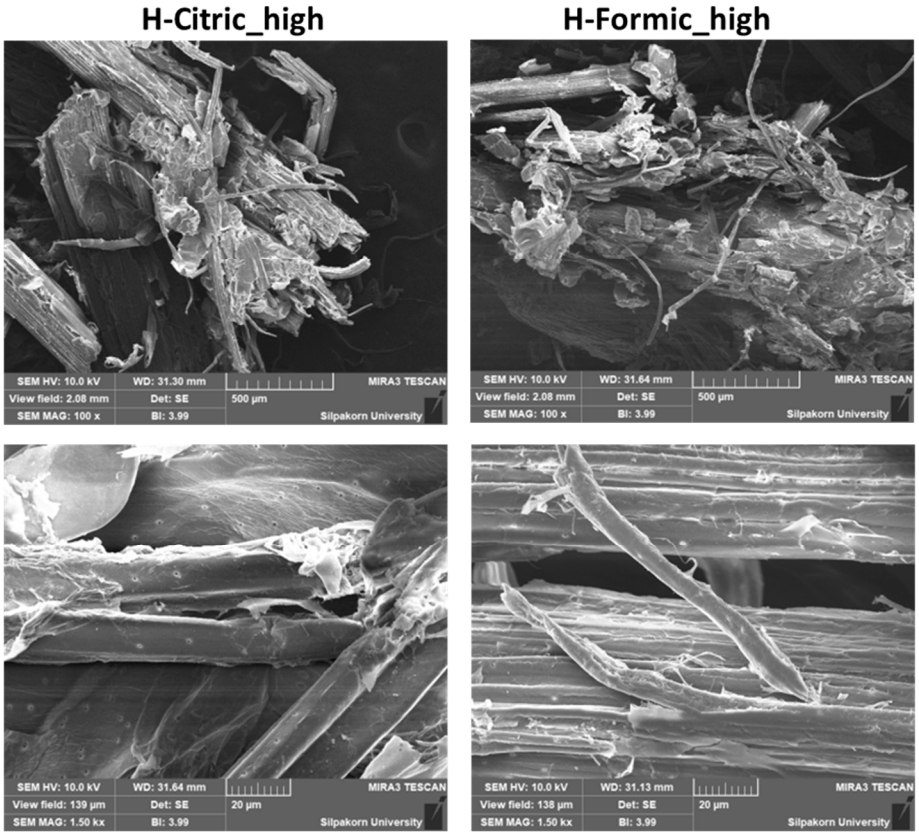


Figure 4. SEM images of the samples.

2.3. Food Container Production

The selected extracted samples were molded into sheets for further use in food container production by first mixing them with starch and passing them through a compression mold. Only H-NaOH_low and B-NaOH_high could be constructed into a sheet. The cellulose fibers of H-Citric_high and H-Formic_high did not form a sheet; instead, they were fragile and unable to withstand stretching during the process. This was related to the cellulose's purity and the fibers' structural characteristics, as can be observed from the SEM images.

The sheets obtained from H-NaOH_low and B-NaOH_high were tested for their mechanical properties, and the results are shown in Table 2. All the measured values indicate that the sheet produced from H-NaOH_low had better mechanical properties than that from B-NaOH_high, which was due to the less-damaged fibers of H-NaOH_low under the hydrothermal treatment. This indicates the benefit of the hydrothermal method, which provides extracted cellulose fibers of high quality and with fewer chemicals.

The sheet derived from H-NaOH_low was then chosen to produce a food container (i.e., a 7-inch plate) using a specific mold. The mechanical properties of the produced plate were determined and compared with two commercial plates (sugarcane bagasse plates). The results are shown in Table 3. Most of the mechanical properties of the produced plate were comparable to those of the commercial ones. The slightly lower tensile strength value of the produced plate may be due to the absence of additives or a coating process, which enhances the strength of commercial products. The increased stretchability (elongation at break) and resistance to tearing (tear strength values) of the produced plate, compared with commercial ones, were probably due to the good quality of the fibers obtained using the hydrothermal methods.

Table 3. Mechanical properties of food container produced from sugarcane bagasse-extracted cellulose compared with commercial food containers.

Food Container	Tensile Strength (MPa)	Elongation at Break (%)	Tear Strength (N)
Commercial 1	31.7 ± 2.3	3.5 ± 0.4	18.1 ± 4.6
Commercial 2	8.3 ± 1.2	1.00 ± 0.2	9.0 ± 1.1
Produced plate (H-NaOH_low)	23.1 ± 3.3	4.0 ± 1.1	26.8 ± 3.1

The products obtained for the manufacturing of food containers must conform to the standards for food containers, including the maximum content allowed for toxic substances. The hydrothermal treatment of cellulose could result in several byproducts that might be toxic; thus, a further toxic substance assessment should be carried out to ensure the safety of the hydrothermally extracted cellulose for practical use.

3. Materials and Methods

3.1. Material

Sugarcane bagasse was collected from Raimaijon Co., Ltd., Ratchaburi Province, Ban Pong, Thailand. It was cut into 3–4 cm long pieces and then boiled in hot water for 30 min (2–3 times) until the Brix value of the solution reached zero, indicating no sugar was left in the bagasse. After that, the bagasse was dried under sunlight for 3 days, subsequently dried in an oven at 80 °C for 24 h, and then kept in an airtight container for further use. Analytical-grade chemicals, including citric acid, formic acid, sodium hydroxide, and ethanol, were obtained from Merck, Bangkok, Thailand.

3.2. Cellulose Extraction

3.2.1. Chemical Method

The conventional chemical method, which uses chemicals for extraction, was conducted here for comparison with the environmentally friendly hydrothermal method. First, the bagasse was immersed in a stirring solution of NaOH (0.25 and 2 M) for 2 h at 90 °C with a fiber–solution ratio = 1:20 by weight. After that, the bagasse was removed from the solution, washed several times with tap water, and neutralized by immersing it in an acetate buffer (1 mol/L; pH 5.5) for 2 h. The bagasse was washed again with deionized water until the solution's pH was near 7, filtered, dried at 65 °C for 1 day in an air-circulating oven, and kept in a desiccator for further use.

3.2.2. Hydrothermal Method

The hydrothermal extractions were carried out with a 2 L stainless-steel reactor chamber, featuring an inner Teflon liner. A solution of water, organic acid (i.e., formic and citric acids; 0.25 and 2 M), or NaOH (0.25 and 2 M) was introduced into the reactor for each experimental run. The bagasse with the fiber–solution ratio = 1:20 was filled into the reactor and placed in an oven at 90 °C for 2 h. After that, the bagasse was treated following the same procedure as in the conventional method.

3.3. Food Container Molding

The obtained bagasse fibers (cellulose) were used for food container production using a self-designed mold with a compression machine. Firstly, 50 g of cellulose was mixed with a colloidal solution of starch (5 g of corn flour in 500 g of water). The slurry mixture was sieved, dried, and spread in the mold. Then, the mold was placed in the compression machine at 60 °C and 50 kg/cm^3 for 5 min. The obtained product was removed from the mold and kept for further testing.

3.4. Characterization

3.4.1. Chemical Composition

The compositions of the raw bagasse and the extracts were analyzed according to the procedures described by Mensaha et al. [38]. Each sample W_A (1 g) was boiled in 60 mL of acetone at 90 °C for 2 h. After that, the fiber sample was separated, washed with deionized water, and dried at 110 °C. The finished sample (W_B) was weighed, and then the percentage (by weight) of contaminants inside the sample was calculated using the following equation (Equation (1)):

$$\% \, Contaminant = \frac{W_A - W_B}{100} \quad (1)$$

After removing the contaminants, the compositions of the samples, i.e., hemicelluloses, lignin, and cellulose, were determined.

- The determination of hemicelluloses:

An amount of 1 g (W_B) of the sample (contaminant-free bagasse) was boiled in 150 mL of a 0.5 M NaOH solution at 80 °C for 3 h 30 min. Then, the sample was separated, washed with deionized water until neutral, and dried at 110 °C until reaching a constant weight. The final weight (W_C) was used to calculate the percentage of hemicelluloses using the following equation (Equation (2)):

$$\% \, Hemicelluloses = \frac{W_B - W_C}{100} \quad (2)$$

- The determination of lignin:

An amount of 1 g (W_A) of the sample (raw bagasse) was soaked in 30 mL of a sulfuric acid solution for 4 h and then boiled at 100 °C for 1 h. Then, the sample was separated and washed with deionized water until neutral, and the filtrate was titrated with an acid to ensure no sulfate ions were left in the system. The obtained sample was dried at 110 °C and weighed, and the obtained weight (W_D), i.e., the lignin weight, was then converted into a percentage using the following equation (Equation (3)).

$$\% \, Lignin = \frac{W_D}{100} \quad (3)$$

- The determination of cellulose:

The weight of cellulose was calculated by subtracting the weight of the raw bagasse from the total contaminants, hemicelluloses, and lignin, and was converted into a percentage according to the following equation (Equation (3)):

$$\% \, Cellulose = \frac{1 - (W_A + W_C + W_D)}{100} \quad (4)$$

3.4.2. Functional Group

Fourier transform infrared (FTIR) spectroscopy was used to determine the functional groups in the samples using a Thermo Scientific Nicolet iS5, Waltham, MA, USA. The FTIR spectra were collected in transmittance mode and recorded in the range of 400–4000 cm^{-1} at a 1 cm^{-1} resolution.

3.4.3. Thermal Stability

The thermal stability and decomposition behavior of the samples were analyzed with a Pyris I thermogravimetric analyzer (TGA), PerkinElmer, Inc, Waltham, MA, USA. Each

sample was heated from 50 °C to 800 °C at a heating rate of 10 °C/min under a nitrogen environment.

3.4.4. Crystal Structure

An X-ray diffractometer (XRD) analysis was employed to determine the crystal structures of the samples using PANalytical, Aeris, Almelo, the Netherlands. The scanning range was from 2θ of 0–60° at a speed of 2°/min, using Cu-K radiation with a wavelength of 1.54 Å. The XRD crystallinity indexes (% CrI) were calculated using Segal et al.'s method [39], according to the following equation:

$$\% \text{ CrI} = \left(\frac{I_{002} - I_{AM}}{I_{002}} \right) \times 100 \quad (5)$$

where I_{002} is the intensity of the 002 crystalline peak at 22°, and I_{AM} is the height of the minimum between the 002 and 101 peaks.

3.4.5. Morphology

A scanning electron microscope (SEM) was employed to investigate the surface morphologies of the samples using MIRA 3, Tescan, Brno – Kohoutovice, Czech Republic. The samples were mounted on stubs and sputter-coated with gold for 15–30 s before being examined.

3.4.6. Mechanical Properties

A universal testing machine (UTM) (QC-536M1-2L4, Cometech, Taichung City, Taiwan) was used to evaluate the mechanical properties of the samples, according to ASTM D828-16e1 [40] for tensile properties and elongation at break, and ASTM D1938-14 [41] for tear strength. The prepared samples were cut to the required size and shape according to the mentioned standards.

4. Conclusions

Cellulose was extracted from SCB via the hydrothermal method with various solvents, and the results were compared with those obtained from the conventional boiling method. It was found that the hydrothermal method with an alkali solvent was more efficient than that with organic acids (citric and formic). Using a low alkali concentration (0.25 M) in the hydrothermal extraction method (H-NaOH_low) exhibited a cellulose extraction ability comparable to that achieved using a high alkali concentration (2 M) in the conventional boiling method (B-NaOH_high). H-NaOH_low also produced fibers with a low-defect structure, resulting in better mechanical properties than those obtained via B-NaOH_high. In particular, the high alkali concentration led to vigorous reactions that damaged the cellulose fibers. Thus, hydrothermal extraction offers the benefit of using fewer chemicals and having a lower environmental impact. In addition, H-NaOH_low was employed for food container production, and it was found that the obtained product has excellent properties, comparable to those of commercial ones.

Author Contributions: Conceptualization, A.J. and E.C.; methodology, A.J.; formal analysis, A.J., T.S., K.S. and E.C.; investigation, A.J., T.S., K.S. and E.C.; writing—original draft preparation, A.J. and E.C.; writing—review and editing, A.J. and E.C.; funding acquisition, A.J. and E.C. All authors have read and agreed to the published version of the manuscript.

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