Regenerated cellulose from high alpha cellulose pulp of steam-exploded sugarcane bagasse

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Abstract

The need for biodegradable films for packaging, absorbents, and fibers has encouraged the development of novel biodegradable films made from natural sources, especially agricultural byproducts. The present investigation involved preparation of alpha cellulose and regenerated cellulose film, in view of the use of sugarcane bagasse, the cellulose-rich waste from the sugar industry. In order to prepare a cellulose pulp, the bagasse was exploded separately by saturated steam at temperatures of 195 °C and 205 °C for 5 min, washed, oven-dried, and submitted to an alkali pulping and bleaching process. The chemical compositions consisted of alpha cellulose, holocellulose, lignin, and the extractives of the bagasse and its pulp were analyzed. The results showed that the pulp contained high levels of alpha cellulose and low lignin. The cellulose pulp was being successfully regenerated as cellulonic films in an acid coagulation bath at different coagulation times. The characteristics of the steam exploded bagasse, cellulose pulp, and regenerated cellulose were investigated by SEM, XRD, FTIR, TGA, tensile test, contact angle, and water retention measurement. The results of the XRD, FTIR and TGA all indicated that high alpha cellulose with low lignin pulp could successfully be made from steam-exploded sugarcane bagasse. The SEM images, contact angles, and water retention values also revealed that the regenerated films coagulated in an acid bath for 15 min were more hydrophilic than those that had coagulated for 30 min. The tensile test indicated that the regenerated cellulose films coagulated for 30 min were stronger than those coagulated for 15 min.

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Introduction

The decreasing in non-renewable natural resources and the rapidly increasing in pollution problems motivate utilization of natural polymers to create interesting new materials [1,2]. Some of petroleum-based products, at present, are gradually being replaced by bio-based products made from cultivated plants or lignocellulosic materials such as agricultural residues. They are widely available, do not take long to substitute, and can rapidly decompose in nature. It has been estimated that the agricultural byproducts from major commodity crops such as sugarcane, rice, and soybean, created...
worldwide will reach over two thousand million tons or more per year [3]. This makes these byproducts ideal resources for natural cellulose products. The use of the cellulose, the most abundant natural polymer compound found in various parts of plants, as a feedstock to replace petroleum-based products, such as plastic film, packaging, medical materials, membranes, absorbents, fibers, and in the future, more will be technically possible.

Sugarcane bagasse is cellulose-rich waste from the sugar industry, generally used as the main fuel source for sugar mills, a feed source for beef cattle, a fertilizer, and landfill materials. Sugarcane can be a potential feedstock because it can be grown and be harvested year round, and after the juice is removed, the bagasse itself can be stacked and stored for a long time [4]. Since the bagasse contains approximately 38–50% cellulose [4-6] and after a proper purification process the amount of lignin can be reduced from 1.62% to 0.68%, making it an ideal feedstock for the production of cellulose-based products such as cellulose pulp and its derivatives [7].

Regenerated cellulose is one of the end-uses of cellulose dissolving pulp, a chemical pulp with a high content of alpha cellulose and relatively low hemicelluloses and lignin content [8]. Regenerated cellulose materials have increasingly gained attention since they are biocompatible, biodegradable, thermal and chemical stable, and low cost. They can be prepared through three-step process, dissolution, shaping and regeneration, which will eventually transform cellulose to useful materials in various forms such as films, beads, fibers, etc. [9]. To meet different type of demands, regenerated cellulose can be used to produce a variety of products, such as fibers for textiles and clothing, disposable medical materials and artificial membranes [10,11]. Recently, novel functional materials have been designed and fabricated via applied chemical and physical treatments during regeneration of cellulose solution such as cellulose/biopolymer composite hydrogels, porous membranes, and inorganic/cellulose hybrids [9,12]. Therefore, regenerated cellulose material will become a promising candidate in utilization of cellulose.

In order to achieve high pulp quality the steam explosion should be introduced prior to the chemical pulping process [13,14]. This method is widely applied to any lignocellulosic biomass due to its potential for disrupting the crystallinity of cellulose, delignification [15,16] and easy hydrolysis of the hemicelluloses [15,17]. In other words, steam explosion can make it easier to separate the cellulose from the bagasse in the further stages, making use of fewer chemicals, less time, and less money in the terms of the overall processes.

This research intends to make high alpha cellulose pulp and regenerated cellulose film from agricultural waste from sugar mill industry. The bagasse is exploded with steam before it reacts with alkali and bleached to separate the cellulose from the lignin and hemicellulose. Steam explosion can effectively reduce the amount of hemicelluloses and acid-soluble lignin of bagasse fibers, while acid-insoluble lignin is removed throughout alkaline washing [18]. The cellulose is then made into the form of viscose and regenerated back to solid-formed cellulose using conventional viscose process. Finally, the regenerated cellulose should be analyzed for its strength, chemical and physical characteristic, and thermal stability.

2. Experimental

Sugarcane cultivar, K84-200, from the eastern region of Thailand, grown commercially for the sugar industry, was used in this study. The bagasse was cleaned, soaked in water for 24 h, dried, and kept in plastic bags to be used throughout the study. The chemical composition (%w/w) of the bagasse is α-cellulose 41.42%, holo-cellulose 69.53%, lignin 19.03%, and extractives 4.16% on a dry weight basis, shown in Table 1. All of the chemicals used were of analytical grade and used as received without any further purification. They all were obtained from either Sigma-Aldrich or local suppliers.

2.1. Steam explosion

A sugarcane bagasse sample (150 g dry weight basis), 2–3 cm in length, was loaded into a 3-L stainless steel reactor and treated by saturated steam at a temperature of 195 °C for 5 min. In due time, slurry was collected from the reactor. The steam exploded solid, hereinafter referred to as steam exploded bagasse, was separated by filtration, thoroughly washed with clean water, centrifuged to eliminate the fluid, then dried in an oven at 60 °C, and finally, the dry samples were stored in a plastic bag for the experimental reasons. The steam explosion treatment was repeated on another 150 g of the same sample using saturated steam temperature at 205 °C for 5 min. The dry weight of the steam exploded samples was calculated as the dry weight of the solid remaining after treatment referred to 100 g of the sugarcane bagasse introduced into the reactor. The chemical compositions, including alpha cellulose, holocellulose, lignin, and extractives of the steam exploded bagasse (solid remaining) were analyzed according to TAPPI-T203-om-93, Acid chloride Method of Browning [19], TAPPI-T222-om-98, TAPPI-T204-om-97, TAPPI-T264-om-97 and TAPPI-T207-om-93.

2.2. Pulping and bleaching processes

The steam exploded bagasse was submitted to alkali pulping process. The sample was reacted with 20% (w/w) NaOH using a liquid to solid ratio of 20:1 at 80–90 °C for 2 h. At the end of the alkali process, the cellulose pulp, which was brown in color, was separated from the black liquor, rinsed through a sieve [mesh size number 50] with clean water until the water ran clear, oven dried at 60 °C, weighed to calculate percentage of the remaining solid (referred to 100 g of the steam exploded bagasse), and then exposed to the bleaching process. In the bleaching stage, the brown pulp underwent three cycles of bleaching. For the first cycle of bleaching, the pulp reacted with 4% (w/w) NaClO2, adjusted to pH 3–4, using acetic acid. The liquor to dry fiber ratio, cooking temperature, and time were kept at 20:1, 80–90 °C, and two hours, respectively. The treated pulp was then washed until the pH of the washing water became neutral. After that, they were treated again with 2% (w/w) NaOH, using a liquid to solid ratio of 20:1 at 80–90 °C for 30 min. The bleached pulp was then washed.
thoroughly with clean water and given a final rinse with distilled water at the end of the last cycle. The second and third cycles were conducted in a similar fashion to the first cycle, except that the concentration NaClO₂ required 3% (w/w) in the second and 1.5% (w/w) in the third bleaching. The final pulp (the third bleaching), hereinafter referred to as cellulose pulp, was oven dried at 60 °C, weighed to calculate the percentage of the remaining solid (referred to 100 g of the steam exploded bagasse) and stored in plastic bags for analytical and regeneration processes. The chemical compositions included alpha cellulose, holocellulose and lignin of the pulp were analyzed according to the methods as above stated. The pulp was also checked its degree of polymerization (DP) and viscosity according to TAPPI T230 Om-94.

2.3. Cellulose dissolution and regeneration

The cellulose pulp (1 g, dry weight basis) was contacted with 18% (w/v) NaOH for 4 h. After that, the solution was drained and the pulp was pressed to remove excess NaOH. The weight of the pressed pulp was three times that of the start pulp. The pressed pulp referred to as alkali cellulose was shredded and left at an ambient temperature for sixteen hours. Then, the alkali cellulose reacted with carbon disulfide in a proportion of about 2 g of carbon disulfide per 3 g of alkali cellulose, at an ambient temperature of 30–35 °C. The reaction was completed in a three to four hours period of time. After this reaction, the cellulose xanthate was slurried with 25 g of 4% (w/v) NaOH to form viscose. The viscose then was filtered and deaerated. The viscose solution was converted into a cellulose film by casting on an acrylic tray. The viscose in the tray was left until irreversible gelation took place and was then immersed to make a completely regenerated cellulose in an acid bath containing 150 g/L of sodium sulfate, Na₂SO₄, and 100 g/L of sulfuric acid, H₂SO₄, for 15 min. The regenerated cellulose in gel form in the tray was then thoroughly washed with clean water until the pH of the film was the same as that of water before use. The regenerated cellulose film was air dried and kept in plastic bags for characteristic tests. The test results of the regenerated cellulose film using an immersion time of 15 min were compared with those of the regenerated films immersed for 30 min.

2.4. Testing

2.4.1. Scanning electron microscopy (SEM)
The morphological property was investigated by using a scanning electron microscopy (SEM), JEOL JSM-6380 LV scanning electron microscope with a voltage of 15 kV. Prior to SEM measurements the samples were sputter-coated with a thin layer of gold by a SPI sputter coater.

2.4.2. Contact angle

The contact angles of the samples were determined using the optical method by attension optical tensiometer, Theta Lite. The films were cut down to 20 × 20 mm. and attached to the base of the equipment. A drop of deionized water was carefully deposited on the surface of the film using a graduated syringe. The contact angle was obtained by photographs and submitted to software analysis for ten seconds.

2.4.3. X-ray diffraction (XRD)
The X-ray diffraction technique was used to evaluate physical structures. X-ray diffractiongrams were obtained with a Philips Analytical X DFW3710 BASED diffractometer. Radiation was generated with an X-ray tube with Cu anode (Kα radiation, λ = 1.54 Å) at 40 kV and 30 mA. The 2θ range was 5.010–59.990°, and the resolution was 0.02° with 1 s average time per step.

2.4.4. Fourier transform infrared spectroscopy (FTIR)
The FTIR spectra were measured by transmission mode using a Perkin Elmer IR SPECTRUM ASCII FEDS 1.60 instrument with KBr pellets.

2.4.5. Thermo gravimetric analysis (TGA)
Thermo gravimetric analysis (TGA) graphs were verified on a TA Instruments Q500 in a nitrogen atmosphere with a heating rate of 10 °C/min. The thermal behavior of the samples was obtained by recording the TG curves in the range 30–700 °C.

2.4.6. Tensile test

The tensile tests (ASTM D882-95a) were conducted using a Hounsfield H10KM universal tester. The samples were cut into strips allowed to equilibrate to ambient conditions before testing commenced.

2.4.7. Water retention

A single gram of regenerated cellulose film was immersed in distilled water for one hour. The water-swollen film was filtered out, put on a small mesh screen, then inserted inside a centrifuge tube, capped, and placed in a centrifuge machine where it was spun and the water was separated from the film. The centrifuged film was accurately weighed after being centrifuged at 3000 rpm for 15 min, and then dried at 60 °C after eight hours, until it reached constant mass (dry weight), before

<table>
<thead>
<tr>
<th>Chemical content</th>
<th>Sugarcane bagasse (raw)</th>
<th>Steam exploded bagasse 195 °C</th>
<th>205 °C</th>
<th>Bleached cellulose pulp 195 °C</th>
<th>205 °C</th>
<th>Standard method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alpha-cellulose</td>
<td>41.42</td>
<td>53.70</td>
<td>56.47</td>
<td>90.88</td>
<td>89.33</td>
<td>TAPPI-T203-om-93</td>
</tr>
<tr>
<td>Holo-cellulose</td>
<td>69.53</td>
<td>61.49</td>
<td>65.46</td>
<td>98.93</td>
<td>98.43</td>
<td>Acid chlorite Method of Browning</td>
</tr>
<tr>
<td>Lignin</td>
<td>19.03</td>
<td>12.85</td>
<td>12.70</td>
<td>0.69</td>
<td>0.64</td>
<td>TAPPI-T222-om-98</td>
</tr>
<tr>
<td>Extractive</td>
<td>4.16</td>
<td>25.56</td>
<td>24.65</td>
<td>–</td>
<td>–</td>
<td>TAPPI-T204-om-97</td>
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<td></td>
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<td>TAPPI-T264-om-97</td>
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<td>TAPPI-T207-om-93</td>
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</table>

Table 1 – Chemical content of the steam exploded bagasse.
3. Results and discussions

The steam explosion destroyed the complex structure of the sugarcane bagasse, causing some components with small molecules to be easily washed off with water. The insoluble residue, remaining after the sugarcane bagasse explosion with steam at 195 °C and 205 °C, and were therefore at 56.44% and 51.61%, respectively. The pulps remained after the steam exploded and bagasse treated by the actions of alkaline were at 30.63% and 26.77% for those steamed at 195 °C and 205 °C, respectively. The final pulps remaining after the steam exploded bagasse was treated by bleach were 18.43% and 23.54% for those steamed at 195 °C and 205 °C, respectively. The results showed that by increasing the steam temperature the percentage of remaining residue decreased. This is because the fibril structure has been destroyed and the hemicelluloses can be separated more easily at higher temperatures. Lignin can also be partially depolymerized more easily when the fibers were treated with higher steam temperature, resulting in lower lignin content (Table 1) [18,22,23]. The degree of polymerization and the viscosity values of final pulps were 1517.63 and 9.69 mPa.s(cP) for those steamed at 195 °C and 1842.76 and 12.80 mPa.s(cP) for those steamed at 205 °C. The chemical compositions of the steam exploded bagasse samples and the final pulps obtained after alkali pulping and NaClO2 bleaching processes were summarized in Table 1. The results showed that the steam exploded bagasse samples treated with saturated steam at 195 °C and 205 °C, and then further treated with the alkaline of the selected conditions contain higher holocellulose and lower lignin contents when compared to the steam exploded samples. Therefore, the first treatment with saturated steam and the next treatment with alkaline and bleach of the selected conditions can be one of the effective methods for extracting cellulose from the sugarcane bagasse [24]. The extracted pulp showed a relatively high percent of alpha cellulose. It is roughly comparable to those of Borregaard pulp, Kraft hardwood pulp, Kraft softwood pulp and low grade linters which have 90.9%, 84.7%, 86.6%, and 92% alpha cellulose content, respectively [25,26]. The trends of pulp yields after alkaline action and after steam explosion were consistent. Meanwhile, the trends of pulp yields after bleaching, the DP and viscosity values were reversed. More weight reduction after alkaline and bleaching treatment may be due to the removal of lignin and hemicelluloses, more of which remains in the steam exploded bagasse at 195 °C. The heterogeneity of sample, the constancy of the procedure, and the severity of the condition may cause more damage to the cellulose structure leading to a reduction of pulp yield and polymer molecular weight for the final pulp, bleached cellulose pulp and the bagasse exploded with steam at 195 °C [27,28].

3.1. Scanning electron microscopy (SEM)

Fig. 1a–d includes a series of SEM images of a bagasse fiber after a steam explosion at temperature of 195 °C and 205 °C under various magnifications. The steam explosion isolated single fibers from fiber bundles and increased the surface area exposed to chemicals. Therefore, the reaction was carried out effectively [29].

The SEM images can also reveal a clean surface and a reduction of the diameter of the fiber by the end of the pulp-making process, Fig. 2 [30]. Fig. 3a–d includes images of clean and well-separated bagasse cellulose fiber after pulping process. It clearly shows that the shape of the macrofibris, which have smaller diameters when compared with those after steam explosion.

3.2. Fourier transform infrared spectroscopy (FTIR)

FTIR spectroscopy is a non-destructive method used to obtain important information about the chemical structure and the crystallinity of a cellulose fiber. The spectra are normally separated into two regions, 4000–2700 cm⁻¹ and 1800–800 cm⁻¹. The 1800–800 cm⁻¹ is called the finger printed region, where the band at 1642 cm⁻¹ is related with adsorbed water in cellulose and some hemicelluloses [31]. The band around 1426, 1364, 1335, and 1314 cm⁻¹ are attributed to CH2 bending, CH bending, OH bending, CH2 rocking vibration, respectively [31–33]. Figs. 4 and 5 shows the FTIR spectra of steam exploded bagasse samples, bleached pulps, and regenerated cellulose films. The steam exploded bagasse samples show the adsorption bands at 1702, 1604, 1514 and 1235 cm⁻¹ which are assigned, respectively, to C=O carbonyl group of carboxylic acid in lignin and ester group in hemicellulose, C=C aromatic ring of lignin, aromatic ring of lignin, and C-O stretching of lignin and hemicellulose [34,35]. The bands at 1702, 1604 and 1514 cm⁻¹ disappeared and the band at 1235 cm⁻¹ was significantly reduced for the cellulose pulps after being treated with alkaline and bleach.

The change of a cellulose crystal structure leads to a simplification of the spectral contour through a reduction in intensity or even the disappearance of the bands characteristic of the crystalline domains [36]. The results show less energy absorbed due to CH2 bending at the band 1426 cm⁻¹ of the regenerated film spectrum. Since the FTIR absorption band at 1426 cm⁻¹, assigned to a symmetric CH2 bending vibration, decreases and is also known as the “crystallinity band”. Therefore, a decrease in its intensity will reflect the reduction in the degree of crystallinity of the regenerated film. The FTIR absorption band at around 894 cm⁻¹, assigned to C–O–C bridge stretching, is designed as an “amorphous” absorption band, an increase in its intensity occurring in the amorphous samples, compared to the initial ones [37].

3.3. X-ray diffraction (XRD)

The diffraction patterns of steam exploded bagasse fibers, alkaline and bleach treated pulps and regenerated cellulose films were shown in Fig. 6. The sharp peaks can be attributed to the scattering of crystallinity, while their diffuse backgrounds were attributed to disordered regions. Therefore, the steam
exploded bagasse and the pulps should be considered to be semicrystalline [38]. The diffraction patterns of bagasse fibers and bagasse pulps show cellulose I structures, with sharp peak at 22.6° and a wide peak between 14.4° and 16.7° [30,38,39]. After dissolution and coagulation process, the regenerated film was casted. The cellulosic regenerated films showed typical cellulose II structure with peak at 11.9°, 20.2° and 21.9° [39]. The results were clearly shown that the crystal structure of bagasse pulp was completely transformed from cellulose I to cellulose II which is the crystalline characteristic of regenerated cellulose product after dissolution and regeneration process.

3.4. Contact angle

The contact angle can indicate the hydrophobic and hydrophilic properties of a certain substrate. The measurement of a contact angle usually defined wettability of the film surface. Small contact angles correspond to high wettability, while large contact angle correspond to low wettability. Fig. 7 shows the contact angle of regenerated cellulose films made from bagasse pretreated with steam explosion process. The results showed that the film became less hydrophilic as the coagulation time increases. The incomplete dissolution of cellulose pulp, the acid concentration in coagulation bath,
Fig. 3 – SEM images of cellulose fibers after pulping process: bagasse fibers, steam explosion at 195 °C under different magnification: (a) 350× and (b) 1000×; bagasse fibers, steam explosion at 205 °C, and individual fiber: (c) 350× and (d) 1000×.

Fig. 4 – FTIR spectra for (a) steam exploded 195 °C, (b) bleached steam exploded 195 °C, (c) regenerated cellulose 195 °C.

...and the contact time between film surface and the acid solution may possibly influence the homogeneity of regenerated structure, which can affect how the surface of film interacts with liquid. With increased coagulation time, the cellulose molecular chains have more time to organize themselves into an ordered structure resulting in denser structure and better uniformity of the surface features, Fig. 8. It indicated that the complete of regeneration of the regenerated cellulose in
coagulation bath may result in different surface morphology and accessible contact areas leading to different wettability and mechanical strength [37,38].

3.5. **Thermo gravimetric analysis (TGA)**

The thermal stability of all of the samples was carried out using thermogravimetric analysis. The TG and DTG curves of steam exploded sugarcane, bleached pulp and regenerated film, are shown in Figs. 9 and 10. All of the TGA curves of the pyrolysis process could be divided into two major weight loss regions, corresponding to, first, slow pyrolysis (30–150 °C) mainly due to the volatilization of water from the cellulosic samples. Second, was fast pyrolysis (280–390 °C) due to its dehydration and depolymerization of cellulose chain [40,41]. In comparison, the thermal stability of the steam exploded sugarcane bagasse was lower than that of bleached cellulose pulp and regenerated cellulose films since the onset of thermal degradation of bagasse was about at 200 °C. The increased thermal stability of the bleached pulp is due to the removal of lignin and hemicellulose residues after the alkaline and bleach treatment [30].
Fig. 7 – (a–d) Static contact angle of a water droplet on a solid surface of regenerated cellulose films, coagulated at 15 and 30 min, made from bagasse pretreated with a different steam explosion process (a) 195 °C 15 min, (b) 195 °C 30 min, (c) 205 °C 15 min, (d) 205 °C 30 min.

Fig. 8 – (a–d) The morphologies of the surfaces of regenerated films with different steam temperature and coagulation time (a) 195 °C 15 min, (b) 195 °C 30 min, (c) 205 °C 15 min, (d) 205 °C 30 min.

3.6. Tensile test

Table 3 shows the value of the breaking stress of the regenerated films with different coagulation times. The elastic modulus, maximum stress and breaking stress of the regenerated film increased with longer coagulation time. This may be because the cellulose molecular chains have more time to organize themselves into an ordered structure so that the maximum stress of the films increase [42]. Moreover, in comparison with conventional polymer films such as LDPE, HDPE, and PP, which have the value of maximum stress equal 8.3–32.1 MPa, 22.1–31.4 MPa, and 31–41.9 MPa respectively [43], the maximum stress of the regenerated cellulose films are comparable with those of conventional films.
The bagasse regenerated ability to retain cellulose was tested after being steam exploded at 195 °C, its lignin decreased by 33.26% and its hemicellulose decreased by 11.56%. The FTIR spectra confirmed that lignin and hemicellulose were effectively removed from the bagasse after being exploded with saturated steam. The alpha cellulose was significantly increased after the bagasse was exploded, especially at 205 °C. Cellulose pulp made from bagasse contained a high percentage of alpha-cellulose and a low percentage of lignin. Regenerated film from bagasse can successfully be made, confirmed by FTIR and XRD results.

The regenerated films show hydrophilic properties which may be useful for some applications. However, this property can be enhanced or diminished through a coating process [45,46]. This study suggests that the steam exploded bagasse may have a high potential for producing alpha cellulose pulp result in opening up promising applications for regenerated cellulose products.

4. Conclusion

Sugarcane bagasse pretreated with a steam explosion can be a good resource for natural cellulosic products. The steam explosion can significantly help to defibrillate the cellulose bundles, and these results in a better accessibility of cellulose for alkali treatment. After the bagasse was steam exploded at 195 °C, its lignin decreased by 33.26% and its hemicellulose decreased by 11.56%. The FTIR spectra confirmed that lignin and hemicellulose were effectively removed from the bagasse after being exploded with saturated steam. The alpha cellulose was significantly increased after the bagasse was exploded, especially at 205 °C. Cellulose pulp made from bagasse contained a high percentage of alpha-cellulose and a low percentage of lignin. Regenerated film from bagasse can successfully be made, confirmed by FTIR and XRD results.

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Conflicts of interest

The authors declare no conflicts of interest.

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