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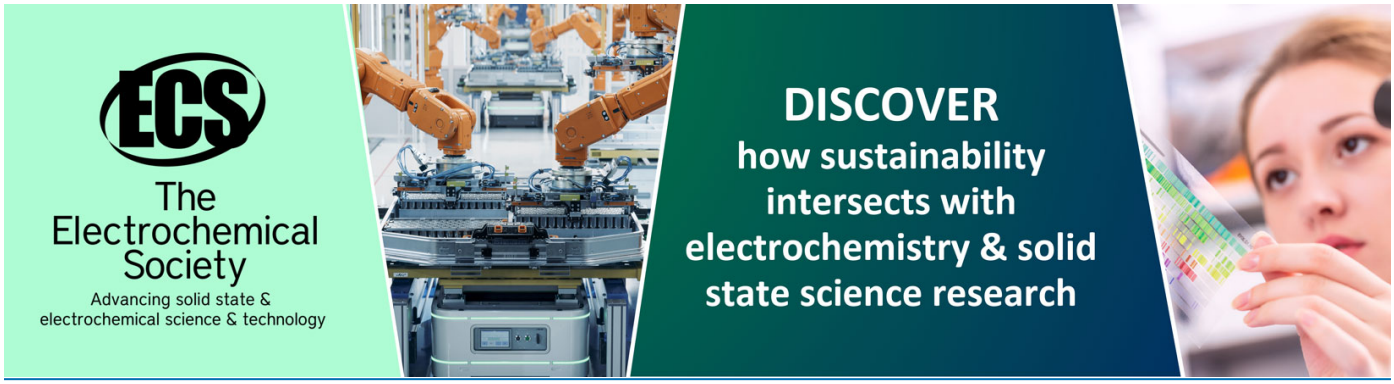

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Effect of different bleaching reagents and process sequences on the properties of steam-exploded empty fruit bunch (EFB) fiber

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Abstract. Bleaching reagent process is essential for the production of cellulose, pulp, and paper to increase the appearance and quality of the final products. Empty fruit bunch (EFB) is an agricultural waste with a lignocellulosic constituent. Recently, the conversion of EFB towards pulp for food packaging and paper manufacturing or cellulose-based materials has been actively developed in lab and pilot scales. However, obtaining efficient extraction and bleaching processes is still the main challenge. In this paper, the effect of different bleaching reagent at different sequences towards the brightness properties of the EFB fiber is observed. The EFB fiber was pre-treated using steam explosion process, hot water treatment, and alkaline treatment before undergoing the bleaching process. Four systems of bleaching process were set using NaOCl and NaO_2Cl as the bleaching reagents. Two sets of the system are using single reagent and the other two sets are using mixed reagent. In the single reagent bleaching system, four stages of bleaching process were used for each NaO_2Cl (C) and NaOCl (H) solution respectively. Meanwhile, the mixed bleaching reagent was conducted with two stages of processes with different sequence of reagents (CH and HC). The bleached cellulose obtained via these two systems were characterized in terms of brightness, chemical composition, thermal characteristic, and degree of crystallinity. The mixed bleaching reagent system the HC has produced the best quality of cellulose with brightness at 77.68%. The chemical and thermal characteristic of the bleached pulp fiber did not change when the bleaching method was applied to the fibers. Even though a single bleaching reagent system with sodium chlorite shows almost similar brightness at 78.66%, the quantity of solvent used is higher compared to the mixed bleaching reagent system. As a conclusion, mixed bleaching reagent system is an efficient system to produce a better quality of cellulose and paper from EFB.

1. Introduction

Researchers in the ASEAN countries, such as Indonesia, Malaysia, and Thailand, are focusing their study on the oil palm industrial waste due to a large amount of waste produced annually. Many studies have been done on the production of cellulose and paper from the lignocellulosic waste of the oil palm industries such as empty fruit bunch (EFB), fronds, mesocarp fiber and trunk [1–5]. Cellulose is the intermediate product for the production of food packaging, paper, pharmaceutical, biomedical, nutritional, cosmetic, polymer, and composites [6]; and paper is a material that can be used for various application such as writing, printing, packaging, hygiene and in industries [7]. Higher quality and grades of cellulose are required to meet the standard of papermaking or high-end application material [8, 9]. A principal goal in producing high-quality paper from lignocellulosic material is to remove lignin and hemicellulose without damaging the cellulose structure. Lignin is a cross-linked hydroxylated, and methoxylated aromatic macromolecule that gives color, resistance to biological

attack and structural rigidity to the material cell wall, and resistance to chemical due to its different units are linked by series of ether and carbon-carbon linkages [9, 10].

The production method in producing paper and cellulose are almost similar, which consist of pretreatment method (defibrillation of fiber, hot water, and alkaline treatment) and bleaching process [11]. The bleaching process is one of the treatments to enhance the physical and optical qualities of the product by removing lignin or fiber decolorization. This process is crucial as it removes residual lignin remained and enhanced the brightness of pulp to the satisfactory level. There are two types of chemical bleaching of pulp [12]. The first technique is a brightening process where it uses a selective bleaching reagent or chemical that will destroy the chromatographic group but does not remove the lignin. The second technique is called true bleaching as the aim of this technique is to almost totally remove the residual lignin by adding an oxidizing chemical to the pulp in varying combination of sequence depending on the application of the product. It creates a longer-lasting whiteness, but it weakened the structure and strength of the fiber [11].

Each naturally produced fiber comes with a different color. Thus difference method in bleaching the fiber is needed to achieve the desired optical and physical quality [11]. Some of the bleaching reagents used are sodium hypochlorite, sodium chlorite, and hydrogen peroxide. Sodium hypochlorite is one of the common bleaching reagents employed as it is capable of oxidizing, brighten, and importantly is the ability to solubilize the lignin structure within the lignocellulosic structure. The advantage of sodium hypochlorite is that it is easy to handle and use. However, if it is not used correctly in a suitable condition, it will cause the fiber to lose strength and degrade. Another reagent that commonly used for the bleaching process is chlorine dioxide. It can be produced from sodium chlorite. It has a similar function with sodium hypochlorite, but it has an additional function where it can avoid the degradation of fiber due to chlorine released during the bleaching process. This reagent also may give a higher level of brightness compared to another bleaching reagent. The only disadvantages of this process are the release of hazardous waste and gas that is chlorine gas [9]. This chlorine gas can be managed by using proper personal protective equipment (PPE) such as respirator and goggle. For big industries, the implementations of a gas scrubber on the bleaching equipment can also become one of the solutions to this problem.

Thus, this study aims to combine sodium hypochlorite and sodium chlorite as bleaching reagent on the pulp produced from empty fruit bunch to achieve a better quality of cellulose and paper products while reducing the solvent consumption. Four systems of bleaching process were set using NaOCl and NaO_2Cl as the bleaching reagents. Two sets of the system are using single reagent, and the other two sets are using mixed reagent. The brightness, thermal stability, chemical composition, and crystallinity determination of the fiber were carried out to determine the performance of the bleaching method.

2. Experimental

2.1. Materials

The raw EFB were supplied by the LCSB Oil Palm Mill located in Pahang, Malaysia and all the chemical reagents such as 99% sodium hydroxide pallet (NaOH), 20% sodium hypochlorite solution (NaOCl), 80% sodium chlorite powder (NaO_2Cl) and 99% acetic acid (CH_2COOH) used in this study were supplied by the R&M Chemical.

2.2. Methods

2.2.1. Sample preparation

The EFB obtained from the oil palm mill are dried in the oven at 60°C for 12 hours. Then the dried EFB were ground using the TST industrial bottle crusher equipped with a 3mm mesh.

2.2.2. Pulp preparation

There are a few treatments needed to produce pulp. Firstly, the grounded EFB fibers were treated using steam explosion pre-treatment at 212°C for 10 minutes. Then the exploded fibers were washed using DI water and undergo the hot water treatment at 80°C for 1 hour. The hot water treated fibers

were then treated using 10% (w/v) alkaline (NaOH) solution at 80°C for 1 hour. The produced pulp was then washed using DI water until the pH reached 7±0.5 (neutral) and then dried in an oven at 60°C for 12 hours.

2.2.3. Bleaching process

Four systems of bleaching process were set using NaOCl and NaO_2Cl as the bleaching reagents. Table 1 shows the process sequences, conditions, and chemical changes.

Table 1. Bleaching sequence details.

System	C	H	HC	CH
Sequence	Chlorite Bleaching only	Hypochlorite Bleaching only	Hypochlorite Bleaching followed by Chlorite Bleaching	Chlorite Bleaching + Hypochlorite Bleaching
Bleaching Stage	4 time	4 time	2 time	2 time
Reagent	4% (w/v) NaO_2Cl	4% (w/v) NaOCl	4% (w/v) NaO_2Cl 4% (w/v) NaOCl	4% (w/v) NaOCl 4% (w/v) NaO_2Cl
Time (min)	60	60	60	60
Temperature (°C)	80	80	80	80

In the C and H systems, the bleaching process was done for four times in series using single bleaching reagent with 4% (w/v) concentration. The HC and CH systems were designed to reduce the usage of reagent. Both bleaching reagents were combined with a different sequence of addition to the exploded EFB fiber. Similar bleaching reagent concentration is still used; 4% (w/v) sodium hypochlorite solution and 4% (w/v) sodium chlorite solution for each sequence and the pH was adjusted using acetic acid until the pH reaches 3. For each system, the process was carried out at 80 °C for 60 minutes.

2.3. Characterization

2.3.1. Brightness test

The bleached pulp fibers hand sheets were prepared according to the TAPPI standard T 205 sp-02 with some modifications. The preparation starts with the preparation of stock solution by dispersing the fiber at 1.2% consistency in a 2L DI water and then diluted to 0.3% consistency in 8L. Then, the sheet was formed using the sheet machine. The sheet making process starts with filling the cylinder with 0.4L of the prepared stock and later adding water until it was 35 cm above the wire. Then, the solution was stirred using a perforated stirrer for 6 to 7 times, and the solution was let to be motionless for 5 to 6 seconds. The drainage was opened, and two blotting papers were placed on both sides of the wet sheet. The wet sheet was couch rolled for 10 seconds. Finally, the blotting papers were removed, and the sheet was dried using a hot press plate for 10 to 15 min.

The brightness was measured using the standard MS ISO 2470-1, where the spectrometer quantifies the actual percentage of the light reflected from the sample at the wavelength of 457 nm. Technidyne Colour Touch 2 Model ISO spectrophotometer was used to measure the brightness of the hand sheet. The brightness of the produced hand sheet was taken three times, and the average value was calculated.

2.3.2. Fourier transform infrared (FTIR)

Infrared spectroscopy of the bleached pulp fiber of each bleaching system was carried out using Thermo Scientific Nicolet iS5 FTIR Spectrometer equipment. The bands were recorded within the region from 4000 to 500 cm^{-1} to identify the changes in chemical properties for each fiber. The spectrometer was calibrated before characterizing the samples.

2.3.3. Thermogravimetric analysis (TGA)
TGA using the TA instrument TGA Q500 was performed to determine the thermal characteristic of bleached pulp fiber for each system. TG and DTG curves are obtained under a dynamic atmosphere of nitrogen gas (flow rate of 35 mL per minutes) heating rate of 10°C per minutes from 30°C to 900°C.

2.3.4. X-ray diffraction (XRD)
The crystallinity of the fiber before and after the bleaching process was monitored using Broker D8 Advance X-ray diffractometer with Ni-filtered CuKα radiation. The fibers were scanned within a 2θ angle range from 5° to 60° at 2 per minute. The crystallinity index Crl value was calculated using the following formula (2.1) [13, 14]:

$$Crl(\%) = \frac{I_{002} - I_{am}}{I_{002}} \times 100\% \quad (2.1)$$

Where I_{002} is the peaks intensity of crystalline fraction and I_{am} is the intensity of amorphous fraction. Average crystallite size for the fiber was calculated according to the following equation, the Scherrer formula (2.2) [13, 14]:

$$D_{002} = \frac{k\lambda}{\beta_{002} cos\theta} \quad (2.2)$$

Where k is Scherrer constant at 0.84, λ is x-ray wavelength at 1.54 nm, β for in radians is the full width at half of the peak of D_{002} peak and θ is corresponding Bragg angle.

3. Results and discussion

3.1. Optical brightness
Figure 1 and figure 2 show the effect of several bleaching stages and a combination of bleaching system on the optical quality of the fiber, which in this case, is the brightness of the bleached pulp fiber. The results of the brightness of the bleached pulp fibers were compared with a control, which is a commercial paper.

Figure 1 shows the bleaching system using system H recorded the brightness of the fiber at 18.44% with 1 time. Then, the fiber brightness increases from the 1 time to 2-time (+15.13%), but at 3-time the brightness of the fiber decreases slightly (-3.06%) and continues to increase at 4-time (+19.48%) reaching 50.50% of brightness. It is shown that the final brightness of the fiber bleached using system H is still 37.39% behind the control. For the second bleaching system C, the brightness of the bleached pulp fiber was 57.10% at 1 time, which is already showing a higher brightness than the final 4-time. Then, the brightness of the bleached pulp fiber gradually increases from 2-time (+13.94%) to 3-time (+7.64%), reaching 78.66% of brightness than that of at 4-time the brightness of the bleached pulp fiber decreases (-4.88%) to 73.78%. The final brightness of the bleached pulp fiber of the C also does not achieve the same brightness of the control. The brightness of the pulp was compared in table 2.

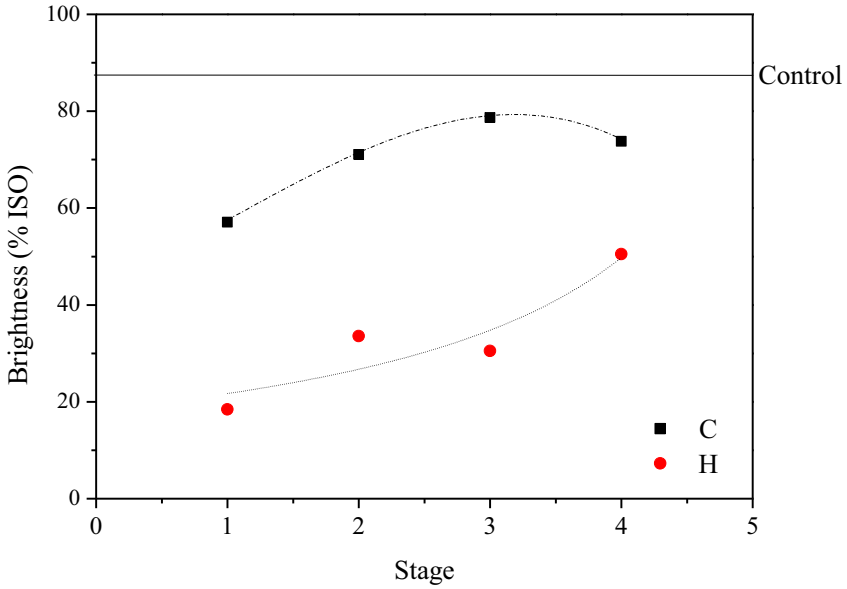

Figure 1. Pulp brightness with a single reagent.

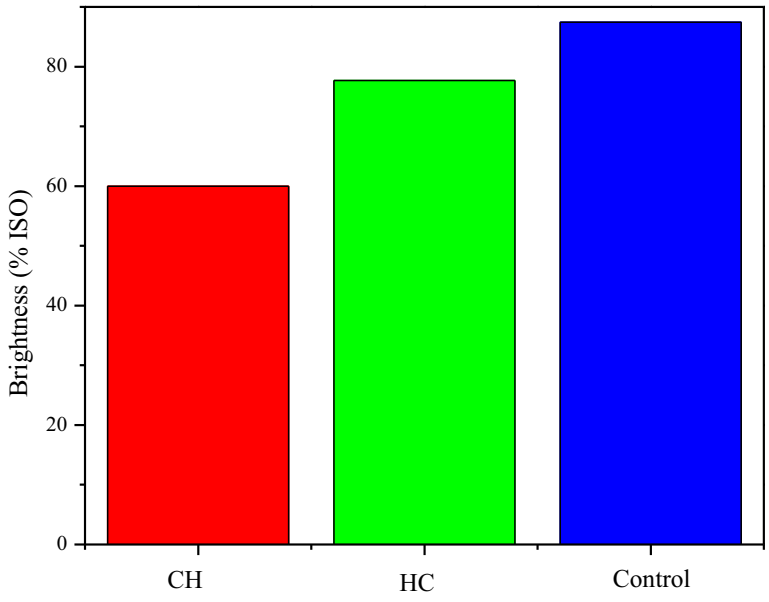

Figure 2. Pulp brightness with mixed reagent.

Table 2. The brightness of pulp bleached via four systems and its control

Sample	Brightness (%)			
	1 time	2 time	3 time	4 time

H	18.44	33.57		30.51	50.50
C	57.10	71.04		78.66	73.78
CH			59.97		
HC			77.68		
Control			87.89		

Figure 2 and table 2 show the results for the mixed systems. It shows that the bleached pulp fiber, for the CH was 59.97% and HC was 77.68%, which was higher single reagent in 4-time H and 4-time C. All the bleached pulp fiber do not reach the same or higher brightness than the control due to the control was not originating from the same source of raw material, which may affect the result of the bleaching process [11]. The reason for the darkening of the bleached pulp fiber at 3-time in system H and 4-time in system C is due to the darkening reaction lead to color reversion and degradation of the fiber [15]. The brightness of the paper would be lower if the number of bleaching stage and holding time were extensively having a negative effect on both brightness [11].

Based on the obtained result, it can be concluded that C has a higher brightness index than the H by a considerable margin. For the mixed reagent system, it was shown that HC excels better than CH, suggesting that combining both bleaching reagents will bring a higher brightness index with less chemical, energy and, time in which will greatly decrease the cost of the process and will be less harmful to the environment.

3.2. Chemical properties

Figure 3 and figure 4 show the FTIR spectroscopy both single and combined reagent for the bleaching process. The trends for both systems are almost the same. However, it can be seen that as the time of bleaching increase, the intensity of the absorbance peaks decreases. The chemical properties of each peak can be divided into two major regions that consist of low wavenumber also called as fingerprint part (400-1800 $cm^{-1)}$ which consist of the and at high wavenumber which is -OH and -CH stretching vibration (2700-4000 cm^{-1}) [16, 17]. The low wavenumber region consists of the C=O stretching of the acetyl and ester bond [2, 18]; 1612 cm^{-1} to 1508 cm^{-1} are referring to the C=C aromatic ring and C=H vibration of lignin[2]; 1375 cm^{-1} are to the C-H deformation of cellulose and lignin [2, 19]; 1234 cm^{-1} corresponds to the axial asymmetric strain of =C-O- in ether, ester, and phenol group of lignin [14]; and lastly the 1108 cm^{-1} and 1033 cm^{-1} are attributed to C-O stretching and C-H rocking vibration of cellulose [2, 19, 20]. Most of the peaks in the fingerprint region are attributed to the linkages and bond between the hemicellulose and lignin matrix to the lignocellulosic matrix.

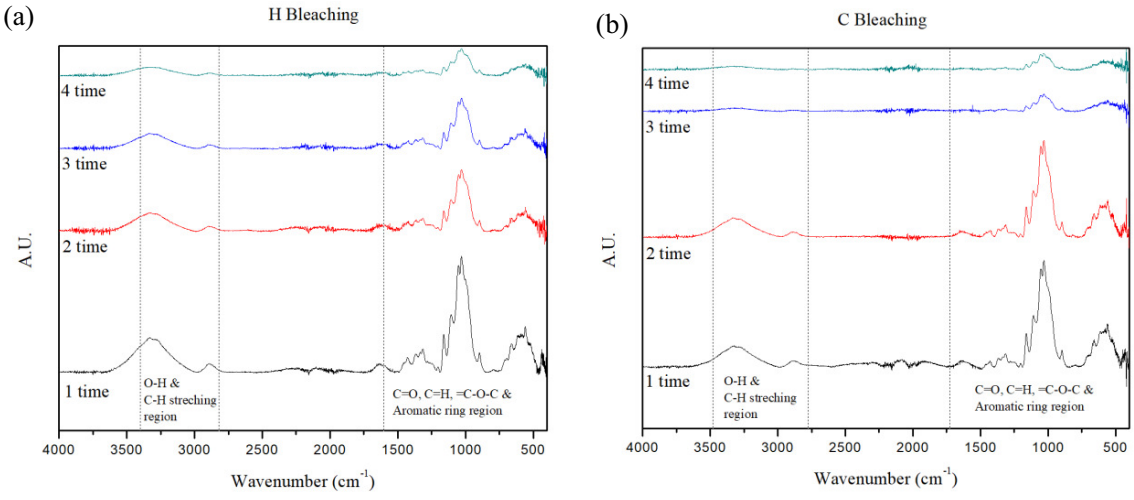

Figure 3. Infrared spectra pulp single reagent for (a) H and (b) C bleaching system.

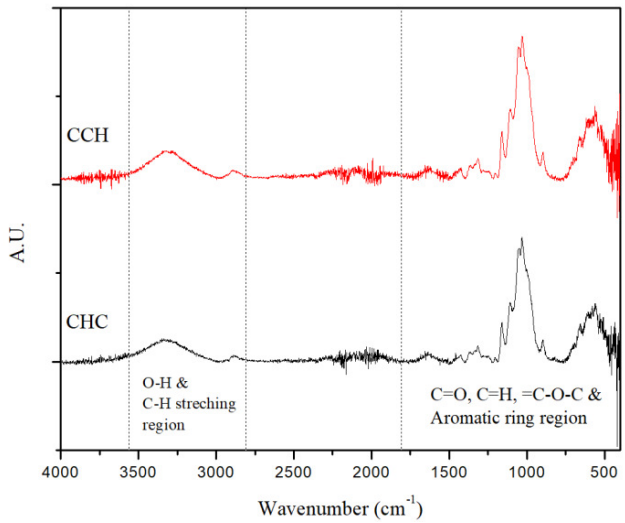

Figure 4. Infrared spectra pulp with mixed reagent.

The effect of bleaching process on the chemical properties of the fiber was observed and shown in figure 3 and figure 4, whereby this includes the effect of bleaching stages for both sodium chlorite and sodium hypochlorite bleaching; and the effect of combining both bleaching reagent on the chemical properties of the fiber. It can be seen at 2915 cm^{-1}, the decrease of the cellulose, hemicellulose, and lignin aliphatic C-H stretching vibration peak [2, 16, 21]. The peak might be resulted by the bleaching process which caused the breakage of bonds and the hydrolysis of the hemicellulose fraction and depolymerization of lignin components from the lignocellulosic structure of the EFB fiber [14, 18, 22]. The C is showing the highest amount of lignin removed during the bleaching process. We can conclude that both bleaching systems do not change the chemical properties of the fiber and have successfully decreased the lignin content of the fiber and the number of the lignin removed increased and, the number of bleaching stages increased.

3.3. Thermal Characteristic

The outcome of bleaching on the thermal characteristic of the fiber was shown in figure 5 and figure 6. The thermogravimetric (TG) curve of the bleached fiber was compared to study the effect of the process. The trend of the TG curve is shown in figure 5 and figure 6, shows that the degradation of these structures was divided into three stages due to the chemical structure of cellulose, hemicellulose, and lignin. Each of the lignocellulosic structure has its degrading temperature which varies from others, starting with hemicellulose (200-300˚C) followed by cellulose (250-400˚C) and lastly lignin (above 350˚C) [14, 23].

The TG curves (figure 5 and figure 6) show that the moisture within the fiber was evaporated before 120˚C as indicated by a small amount of weight loss. Figure 6 shows the TG curve for H and C. The TG curve shows all of the bleached pulp fibers that start to decompose at a high rate of 200˚C until 400˚C and followed by a low rate of decomposing until it reached 900˚C and produced char yield at 10.75%, 10.75%, 10.75% and 9.91% for the 1 time, 2-time, 3-time, and 4-time for system H, respectively. The calculated char yield for the 1 time, 2-time, 3-time, and 4-time for system C was 6.21%, 3.96%, 6.21%, and 6.21%, respectively. Figure 6 shows a combination of reagent for bleaching process, which starts decomposing at 200˚C, but HC finishes decomposing at 400˚C, and CH finishes at 570˚C. Both combinations show an almost similar trend and stability from 100˚C to 365˚C, but after 365˚C, the second combination further decomposes while the first combination remains unchanged. The char yield for both combinations are 5.38% and 2.34%. Based on these results, the combination of bleaching systems slightly changes the thermal characteristic of the fiber but produced less char yield.

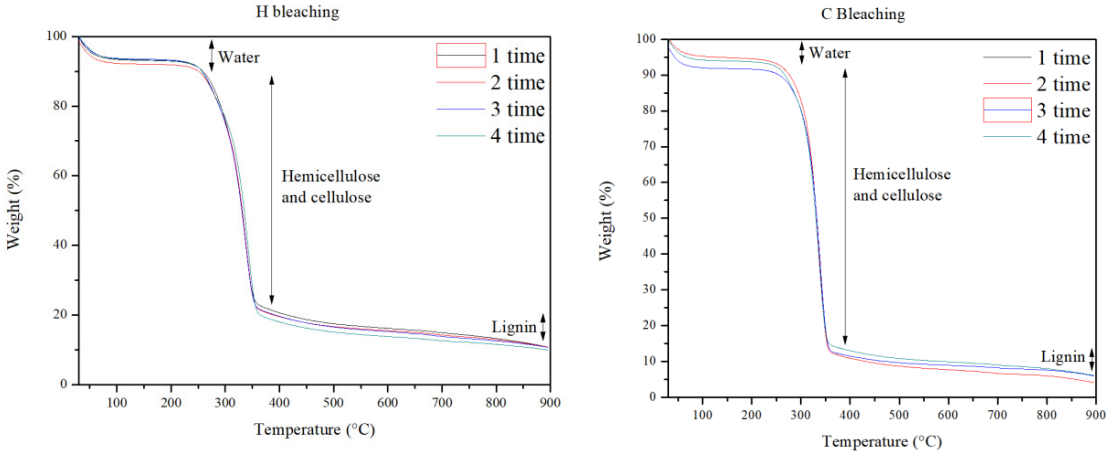

Figure 5. TG curve with a single reagent.

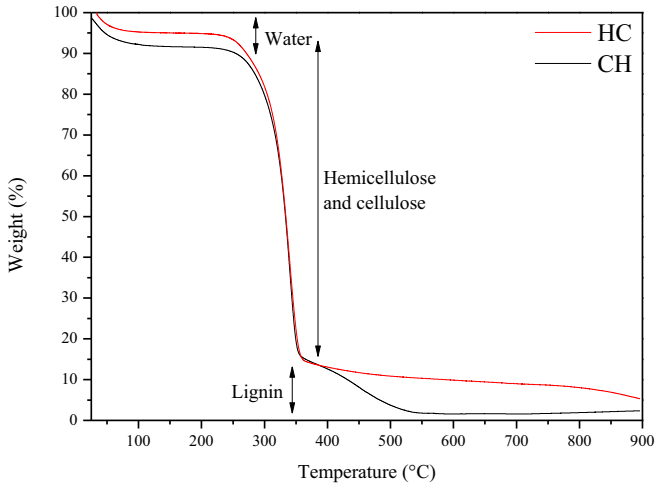

Figure 6. TG curve with mixed reagent.

The hemicellulose, cellulose and, lignin content within the fiber can be measured using the weight loss percentage during the degradation of the fiber. The lignocellulosic structure has its own degrading temperature. The thermal degradation (T_{on}) and temperature of maximum degradation (T_{max}); and the calculated, hemicellulose, cellulose, lignin and, char yield for both fibers are shown in table 3.

Single reagent in table 3 shows that the cellulose and hemicellulose content for H increases as the time of process increases, but for C the value fluctuated. The lignin content for H also decreases as the time of process increase and, the CH process also shows an in-consistence value. The HC in system 2 shows a higher hemicellulose and cellulose content and a lower lignin content compared to CH. The fluctuation content of the lignocellulosic structure was probably due to the degradation of the structure [15]. The char yield produced was due to the number of impurities after the bleaching process [14]. It can be concluded that several stages during the bleaching do not affect the thermal stability of the bleached pulp fiber and an extensive amount of bleaching will cause the lignocellulosic structure of the fiber to vary.

Table 3. Thermal properties bleached and delignified fiber.

Sample	Hemicellulose & Cellulose			Lignin			Char yield (%)
	T_{on} (°C)	T_{max} (°C)	Weight (%)	T_{on} (°C)	T_{max} (°C)	Weight (%)	
H							
1 time	200	355	69.87	355	400	12.53	10.75
2 time	200	355	70.25	355	400	11.01	10.75
3 time	200	355	71.17	355	400	11.39	10.75
4 time	200	360	72.91	360	400	10.33	9.91
C							
1 time	200	360	82.55	360	400	8.75	6.21
2 time	200	360	81.93	360	400	8.57	3.96
3 time	200	360	78.82	360	400	6.51	6.21
4 time	200	360	79.18	360	400	8.34	6.21
Mixed reagent							
HC	200	365	80.39	365	400	9.02	5.38
CH	200	365	76.57	365	400	12.46	2.34

3.4. Crystallinity

Besides of the changes in the brightness, chemical properties and thermal stability of the bleached pulp fiber, the crystallinity of the bleached pulp fiber was another significant parameter to be evaluated to study the effect of breaching process on the crystallinity of the bleached pulp fiber. The crystallinity of the bleached pulp fiber was studied to identify the effect of the process on the fiber. Figure 7 shows the XRD pattern of 4-time C and H, and CH and HC in order to observe the changes in the crystallinity index and crystallinity domain size before and after the bleaching process.

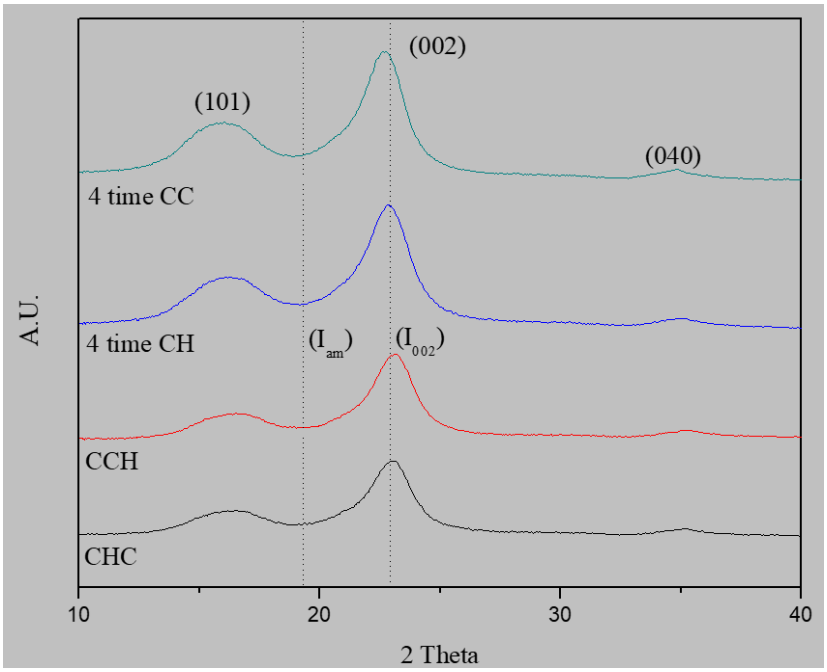

Figure 7. XRD of 4-time C and H; and CH and HC.

XRD pattern in figure 7 shows two peaks of diffraction intensity around the 2θ = 15.6 and 22.6, indicating that all the fiber was cellulose I polymorph [2],[14]. The crystallinity index and crystallinity

domain size calculated using Equation (2.1) and Equation (2.2) were shown in table 4. The crystallinity index of the bleached and delignified fiber in for 4-time with system H and C are the highest which were 88.90% and 89.73%, but for both HC and CH which were 61.02% and 58.76%. The crystallinity domain size of 4 time with system H and C were 3.76 nm, 3.75 nm; and HC and CH were 3. 67 nm and 4.16 nm, respectively. The higher crystallinity index for both 4-time with system H and C was possible due to the higher number of the bleaching process and, during the bleaching process, the chemical reagent had penetrated the amorphous region of the cellulose causing hydrolytic cleavage of glycosidic bonds and releasing individual crystallites [2]. The removal of the amorphous region had caused the crystalline region within the bleached and delignified fiber to realign and change the crystalline domain size [20].

Table 4. Degree of crystallinity index and crystalline domain size of bleached pulp fibers.

Sample	Crystallinity index (%)	Crystallinity domain size (nm)
H (4 time)	88.90	3.76
C (4 time)	89.73	3.75
HC	61.02	3.67
CH	58.76	4.16

4. Conclusions

Pulp produced from the EFB was successfully bleached using single reagent that is H and C. The best chemical reagent for the bleaching process of the EFB pulp was the C which gives the highest brightness value that was 78.66%. However, the combination of both reagent, HC show a promising brightness result of 77.68%, which is only 1.98% behind the highest obtained brightness and, it consists only 2-time of the bleaching process, which greatly reduces the chemical usage. Furthermore, the chemical composition characterization also shows that the lignin and hemicellulose were removed during the bleaching process. The bleaching process also does not affect the thermal characteristic of the fibers. The crystallinity of the fiber also varies if different bleaching system were applied to the fiber. Additional studies on the bleaching of EFB pulp are needed in term of the physical quality of the bleached pulp fiber such as the strength of the fiber to produce higher quality cellulose and paper.

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