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Production of cellulose and microcellulose from pineapple leaf fibre by chemical-mechanical treatment

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Abstract. Pineapple leaf fibre (PALF) is major waste from pineapple cultivation with high cellulose content that exhibits superior mechanical properties. In this study, chemical-mechanical treatments were conducted to produce cellulose and microcellulose. For alkali and steam treatment, PALF treated with 5 wt% sodium hydroxide solution and steamed in an autoclave at temperature of 121 °C and pressure of 21 psi. Next, the PALF was bleached with 5 wt% sodium chlorite. Continued with acid hydrolysis with 3.5 M and 7.5 M hydrochloric acid to produce cellulose. For, the mechanical treatment which involved homogenization and ultrasonication to produce the microcellulose, the ultrasonication was varied for 30 and 60 min. The samples were analysed by Scanning Electron Microscopy, Thermal Gravimetric Analysis and Fourier Transform Infrared Spectroscopy (FTIR) to study surface morphology, thermal stability and functional group respectively. The results showed that ABAHU60 with alkali treatment, bleaching, acid hydrolysis, homogenization and ultrasonication of 60 min exhibits excellent thermal stability and surface morphology, where the maximum degradation temperature occurs at 349 °C, which is a 5% improvement compare to untreated fibre. Its surface is smoother without impurities, with a loose structure and reduce diameter of fibre.

1. Introduction

Pineapple leaf is a renewable agricultural residue, available in abundance, which is used very rarely and is of limited value at present. The utilization of pineapple leaf fibre can minimize the environmental pollution and are able to generate profits from the waste. In Malaysia, 1.2 million tonnes of agricultural waste are disposed of into landfills annually [1]. It is estimated that 15% of the total waste generated in Asia is agro waste, with agricultural waste generation in Malaysia at approximately 0.122 (kg/cap/day) in 2009 which is projected to reach 0.210 (kg/cap/day) by 2025 [2].

According to the Fruit Crop Statistic provided by the Department of Agriculture (2017), pineapple (*Ananas Comosus*) is a major fruit crop in Malaysia, with the second highest production of 340,772 metric tonnes and plantation area of 12,898.44 hectares. Therefore, in the context of Malaysia, with increasing pineapple production, pineapple wastes are also increasing proportionally. Therefore, agricultural waste management from the agricultural sectors in Malaysia is a critical issue. This kind of waste is usually eliminated by burning or decomposing and this may lead to the arising of some environmental issues. Turning pineapple leaf into wealth not only makes good environmental sense, but also regarded as resources for economy development [3]. Based on this concern, this research aimed to utilize this agricultural waste in order to produce natural biopolymer.

In Malaysia, several initiatives have been done in order to extract fibre from pineapple leaf and convert into commercial products. However, this process is still new for Malaysia compare to other countries. The proposed technology involved in sustainable pineapple leaf fibres (PALF) productions practicing the process that will reduce the environment pollution, minimize the waste and conserved natural resources. This sustainable manufacturing will maximize the productions of pineapple leaf fibres and develop green environment as well as boost the economy growth of the country [3].

Cellulose is an insoluble substance which is the main constituent of plant cell walls and vegetable fibres. Therefore, cellulose is unquestionably the most abundant naturally occurring reproducible organic compound [4]. It has been used as a manufacturing material for several commodities in the food and pharmaceutical industries, as well as in paint and textiles industry for years. In recent years, a wider application of cellulose has been proposed for developing various biocompatible products as well as a variety of commercial cellulose derivatives [5]. Typically, the cell wall of fibres comprises of repeated crystalline structure subsequent from the aggregation of cellulose chains. These cellulose bundles are surrounded by an amorphous matrix of hemicelluloses and lignin. The removal of these component and defibrillation of cellulose bundle gives rise to a new class of fibres, which is microcellulose with improved performance [6]. Moreover, the microcellulose is purified, partially depolymerized cellulose having the formula $(C_6H_{10}O_5)_n$. It is an insoluble derivative of cellulose which currently marketed in powder form, used as a thickener in foods and drugs [7].

The objective of this research is to produce the cellulose and microcellulose with effective pretretament or surface modification method using chemical and mechanical tratement. Basically, cellulose and microcellulose can be obtained through chemical treatments such as alkali treatment/ bleaching, peroxide, enzymatic hydrolysis, acetylation and, mercerisation and/or mechanical treatments such as ultrasonication, high pressure homogenisation, microfluidisation, cryocrushing, grinding [8]. Comparatively, ultrasonication have been reported to be a convenient technique for defibrillating cellulose fibre bundle into micro fibre with excellent mechanical properties [9]. Therefore, the scopes that involved in this research are conducting the study on the effect of steam and alkali treatment as the first treatment on the preparation of cellulose, the effect of acid hydrolysis as the third treatment on the preparation of cellulose, the effect of ultrasonication time on the preparation of the microcellulose. Moreover, it involved characterization of cellulose and microcellulose produced from pineapple leaf fibres by using Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM) and Thermal Gravimetric Analysis (TGA).

2. Methodology

2.1. Materials
The materials that were used in the experiment is PALF collected from Pekan Pina Sdn.Bhd, Pekan, PAhang as a raw material of cellulose. The chemicals used were purchased from Sigma-Aldrich and R&M Chemicals. The chemicals used were NaOH (sodium hydroxide) with 96% purity for alkali treatment, HCl (hydrochloric acid) with purity of 37% used for acid hydrolysis and $NaClO_2$ (sodium chlorite) with purity of 80% as a bleaching agent.

2.2. Pineapple leaf fibres preparation
PALF were washed with tap water to remove dirt on the leaves and cut into size of 1 to 2 cm for storage purposes. The pineapple leaves were dried in an oven at 70 °C to prevent the growth of fungus on the leaves. The dried samples were kept in airtight sealed bag for experimental use.

2.3. Production of cellulose from pineapple leaf fibres
The PALF were treated with alkali and steam treatment respectively to compare the effectiveness of the treatment. Both samples were continued with bleaching treatment. Then, the fibres were further treated with and without acid hydrolysis for production of cellulose. For production of microcellulose, the cellulose was treated with mechanical treatment of homogenization and ultrasonication.

2.3.1. Alkali treatment of pineapple leaf fibres. PALF was treated with 5 wt% sodium hydroxide solution for 180 min at 90 °C at ambient pressure [10]. The ratio of the solution and fibre is in 20:1. Then, it was washed with distilled water until pH 7 and was dried in drying oven at 60 °C [11].

2.3.2. Steam treatment of pineapple leaf fibres. Steam at a temperature of 121 °C and a pressure of 21psi was set to treat pineapple leaf fibre for 60 min by using an autoclave to partially remove hemicellulose. After steam treatment, treated fibre was washed in distilled water and dried overnight at a temperature of 60 °C.

2.3.3. Bleaching of pineapple leaf fibre. The next step of the process is bleaching. The pineapple leaf fibre was mixed with 5 wt% sodium chlorite and pH adjusted to 4-5 [12, 13]. The treatment was conducted in water bath at 70 °C for 90 min. Then, the fiber were washed with distilled water until pH 7 and was dried in drying oven at 60 °C.

2.3.4. Acid hydrolysis. For acid hydrolysis, the suspension was hydrolysed with 3.5 M HCl for 12 h at 70 °C and then it was washed until pH 7. The next hydrolysis involves 7.5 M HCl left for overnight at temperature 70 °C [11]. It was washed until pH 7. The other samples were prepared by skipping the step of acid hydrolysis to study the role of acid hydrolysis in the production of the microcellulose.

2.4. Production of microcellulose by mechanical treatment of homogenization and ultrasonication
The cellulose produced was treated with mechanical treatment which involved high shear homogenization and ultrasonication. 8 samples were prepared on different treatment time of ultrasonication of 30 min and 60 min. For high shear homogenization, Eurostar 20 High Speed Digital Homogenizer from IKA, China was used. A 5% suspension was dispersed in 100 ml distilled water and then homogenized at 6000 rpm at room temperature for 20 min. For ultrasonication, it was performed with Crest Ultrasonic Cleaner from The Crest Group, United States for 30 min and 60 min respectively at 60 °C.

2.5. Characterization of cellulose and microcellulose

2.5.1. Fourier transform infrared spectroscopy (FTIR). All samples were dried in a drying oven at 60 °C until a constant weight was achieved [14]. The FTIR was conducted with Thermo Scientific iD7 ATR FTIR Spectrometer scanned in a range of $300 - 4000$ cm^{-1}.

2.5.2. Scanning electron microscopy (SEM). Scanning electron microscopy was conducted using Hitachi Tabletop TM3030 Plus Scanning Electron Microscope. The cellulose was observed at different magnification ($100\times$, $500\times$, $1000\times$, $2000\times$ and $5000\times$). To improve the scanning of the sample under the microscope, the samples are coated with platinum using sputtering techniques.

2.5.3. Thermal gravimetric analysis (TGA). The mass of the samples was measured over time as the temperature changes in the analysis. TGA was conducted with Hitachi STA 7000 Thermogravimetric Analyzer. About $6 - 10$ mg sample was analysed using nitrogen atmosphere at a rate of 100 mL/min by constant heating at a heating rate of 10 °/min, with ramping temperature, ranging from 30 to 700 °C.

3. Results and Discussion

3.1. Characterization of cellulose

3.1.1. Physical appearance of untreated fibre and treated fibre for production of cellulose. The physical appearance of untreated and treated fibres after each treatment was observed based on the colour and visible surface structure. Table 1 depicts the identification and the treatment involved for each fibre

while Figure 1 illustrates the physical appearance of untreated fibre and treated fibre for production of cellulose.

Table 1. The identification and treatment involved in each sample.

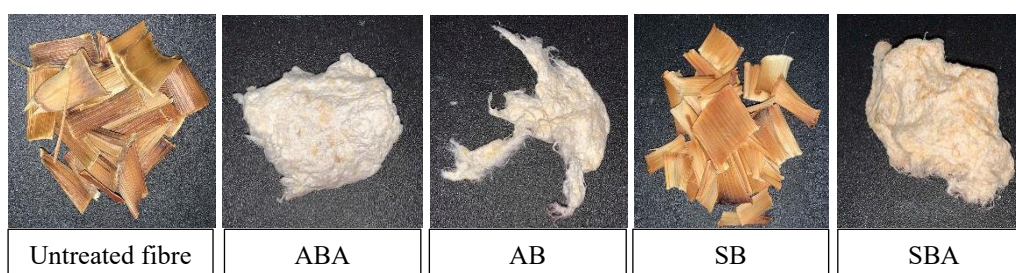| Sample	Treatment involved
Sample 1 (ABA)	Alkali Treatment + Bleaching +Acid Hydrolysis
Sample 2 (AB)	Alkali Treatment + Bleaching
Sample 3 (SB)	Steam Treatment + Bleaching
Sample 4 (SBA)	Steam Treatment + Bleaching +Acid Hydrolysis

| Untreated fibre | ABA | AB | SB | SBA |

Figure 1. Physical appearance of untreated fibre and treated fibre.

According to Figure 1, the colour of ABA and AB is lighter compared to SB and SBA. ABA and AB with alkali treatment is more effective in the production of cellulose as it showed a whitish fibre which is closer to the appearance of cellulose. For SB with treatment of steam treatment without acid hydrolysis, the fibre remains its leaf structure. The lighter colour of the fibre indicates that more components are removed from the fibre during the treatment. Fibres that are high in lignin are generally brown colour [15]. Thus, the brownish colour in SB and SBA is due to the lignin content in the fibre. This indicates that further treatment is needed for production of higher purity of cellulose.

3.1.2. Fourier transform infrared spectroscopy (FTIR) of cellulose. Figure 2 shows the comparison of FTIR results of untreated fibre and fibre treated with alkali or steam treatment with and without acid hydrolysis. As shown in Figure 2, the FTIR spectra of untreated pineapple leaf fibres and the 4 samples treated were compared. The absorption bands were observed in two wave number regions of 3500 - 2800 cm^{-1} and 1800 - 800 cm^{-1}. The observed peaks in the wave number range of 3500 - 2800 cm^{-1} is the characteristic for stretching vibration of O-H and C-H bonds in polysaccharides [16]. The broad peak at 3330 cm^{-1} is characteristic for stretching vibration of the hydroxyl group in polysaccharides [17]. This peak includes also intermolecular and intramolecular hydrogen bond vibrations in cellulose [18]. The band at 2900 cm^{-1} is attributed to C-H stretching vibration of all hydrocarbon constituent in polysaccharides [17].

Typical bands assigned to cellulose were observed in the region of 1800 - 800 cm^{-1} which belong to stretching and bending vibrations of -CH_2 and -CH, -OH and C-O bonds in cellulose [19]. C=C stretching that attribute to vibration of benzene and aromatic lignin of the skeletal vibration were observed in the sample at peak around 1050 cm^{-1} wavelength [20]. At peak around 600 cm^{-1}, the peak signifies the present of C-H bond from glucose ring of the cellulose were observed [21].
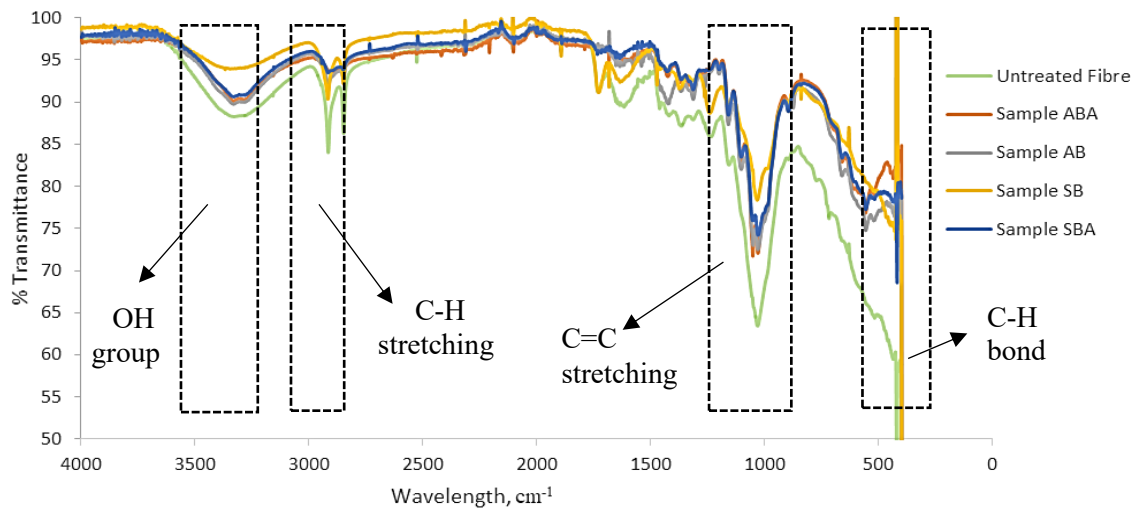
Figure 2. Comparison of FTIR results of untreated fibre and fibre treated with alkali or steam treatment with and without acid hydrolysis.

Based on Figure 2, untreated fibre has the broadest peak when compare to other sample and this indicates that some interaction such as hydrogen bonding interaction has occurred. This is supported by the theory of the natural fibres are constitutes of cellulose and lignin; these celluloses consist of many fibrils, associated with hydrogen bond to provide strength and flexibility [22]. Generally, the treated sample shows a similar trend of decreased intensity. This decreased intensity indicates that the treated sample has lesser bond existing in the component [23]. Thus, the chemical treatment and steam treatment are both effective on the removal of lignin from the pineapple leaf fibre as the intensity of the bonding decrease at wavelength around 1050 cm^{-1}.

3.1.3. Surface morphology of cellulose. Figure 3 depicts the SEM image of Cellulose ABA which involved in alkali treatment, bleaching and acid hydrolysis in the production of cellulose while Figure 4 depicts the SEM image of Cellulose SBA which involved in steam treatment, bleaching and acid hydrolysis in the production of cellulose.
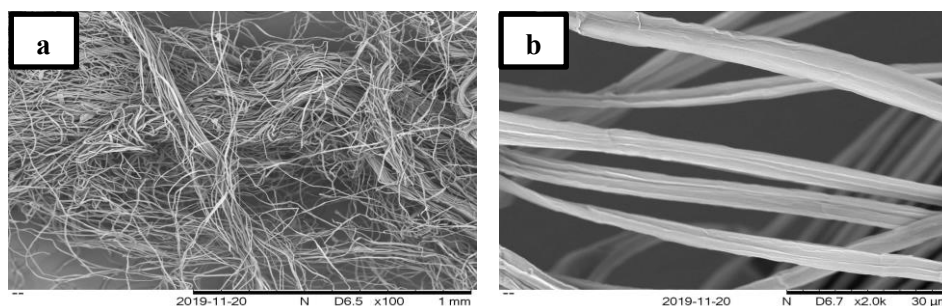

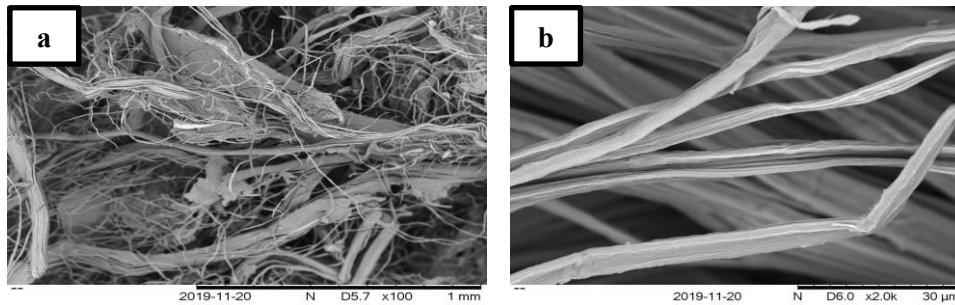**Figure 3.** SEM image of Cellulose ABA under different magnifications (a) 100× and; (b) 2000×.

Figure 4. SEM image of cellulose SBA under different magnifications (a) 100× and; (b) 2000×.

According to the Figure 3 and Figure 4, the SEM of Cellulose ABA shows a more uniform and smooth fibrils in the magnification of 100× while for Cellulose SBA the cellulose produced is not uniform as some of it still in bundle form and a part of it had become fibrils. Whereas SEM image of ABA and SBA in 2000× show clear defibrillation of fibre with clear fibril surface. This is because Cellulose ABA with the treatment of alkali treatment had enable the hemicellulose to hydrolyse and become water soluble. The steam treatment at high pressure is conducted for the same purpose but there is residue of hemicellulose as shown in Figure 4 [24]. Bleaching plays a role for the removal of the lignin present in the pineapple leaf fibre which will allows further defibrillation. Lignin was oxidized by the sodium chlorite and formic acid buffer in the bleaching process. The oxidation of the lignin leads to lignin degradation and leads to the formation of hydroxyl, carbonyl and carboxylic groups, which facilitate the lignin solubilization in an alkaline medium. Through the bleaching process, the lignin and hemicellulose has been broken due to the removal of the amorphous content resulting in microfibril bundles [25].

3.1.4. Thermal stability analysis of cellulose. The Thermogravimetric Analysis (TGA) and Derivative Thermogravimetry (DTG) reading curve of the sample with different treatment was compared. The maximum of the weight loss curves, and the charred residues is compared to evaluate the thermal properties of cellulose. Figure 5 which is the TGA graph shows the comparison of weight percent while Figure 6 is the DTG graph which illustrates the comparison of derivative weight of the fibre with different treatment.
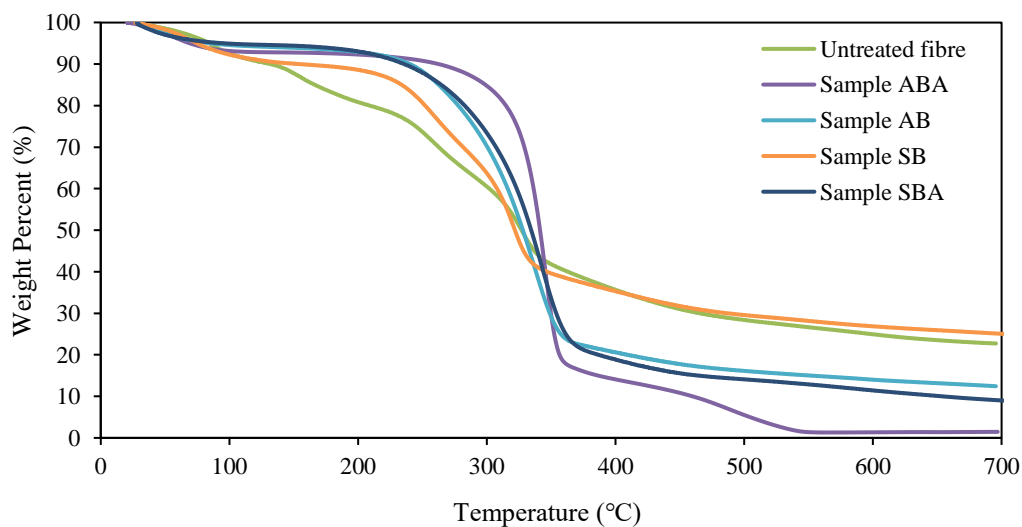

Figure 5. Thermogravimetric analysis (TGA) graph.

Generally, there are 4 successive phases in the thermal degradation process of pineapple leaf fibre. The initial weight loss of the fibres in the thermal range of 50 °C to 100 °C occurred due to the removal of moisture and some volatile components in the fibres [26].
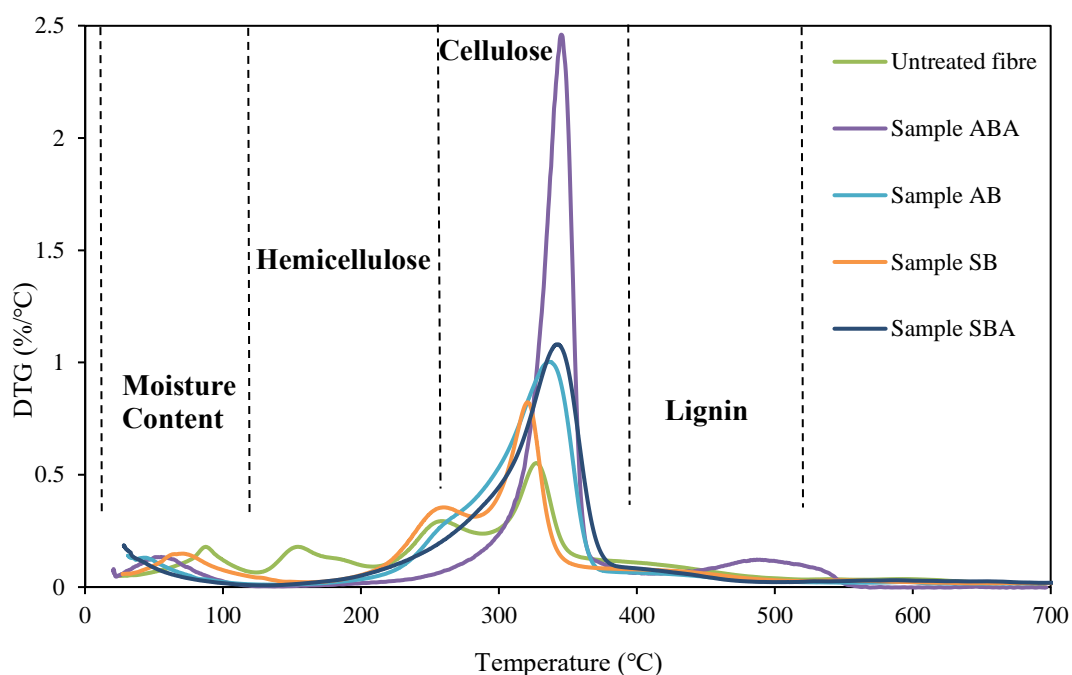

Figure 6. Derivative thermogravimetry (DTG) graph.

The small peak in the temperature around 100 °C is associated with the vaporization of water in the cellulose that is due to the hydrophilic nature of functional group, OH, in the cellulose [27]. The weight loss due to water evaporation was remarkably higher in untreated fibres than treated fibres. This indicates that there is higher moisture content in the untreated fibres as there are more hydrophilic tendency in the raw fibre [17]. For the second phases, the second weight loss (160 to 250 °C) was associated with degradation of amorphous hemicellulose components and glycosidic linkages in cellulose. Hemicellulose shows the lowest thermal stability in the fibres and had a rapid degradation temperature range of 200–350 °C [28]. On the other hand, cellulose is more thermally stable, compared with hemicellulose, whereas for lignin with the aromatic structure exhibit the highest thermal stability. Its thermal decomposition mainly takes place between 160–900 °C and decompose fastest between 310–420 °C [29].

All the fibres except cellulose ABA and cellulose SBA show few steps decompositions at the range of temperature of 100 °C to 350 °C with the highest decomposition in one step ranging from 300 °C to 350 °C. The Cellulose SBA (Steam + bleached +acid hydrolysis) showed one step weight loss with a maximum at 343 °C whereas for Cellulose ABA (Alkali + bleached + acid hydrolysis) showed one step weight loss with a maximum at 344.9 °C. This result show that the alkali treated fibre had higher thermal stability than steam treated fibre. According to the Figure 5 and Figure 6, the treated fibre will have higher thermal stability compare to the untreated fibre. The untreated fibre started its decomposition easily with the weight loss mainly happened at 100–340 °C. Table 2 shows the summary of degradation temperature of untreated fibre, steam treated fibre and alkali treated fibre.

Table 2. Summary of degradation temperature of different fibre.

Sample	Weight loss at 100 °C	Temp at 20% weight loss	Temp at 50% weight loss	Residue at 700 °C
Untreated Fibre	7.42	209.12	327.00	22.73
Sample SB	7.71	252.05	321.85	25.06
Sample SBA	5.06	282.83	334.50	9.01
Sample AB	6.85	288.93	328.04	12.43
Sample ABA	5.40	314.54	340.98	1.45

According to Table 2, the fibre with acid hydrolysis had lower value of moisture content with weight loss of 5.06% for Sample SBA and value of 5.40% for Sample ABA. The moisture content in the sample was indicated by the weight loss of the sample at 100 °C. Moreover, both sample with acid hydrolysis has the lowest residue at 700 °C as the amount of lignin in each sample is the lowest. This is because the residue of the cellulose may due to the accumulation of lignin [26]. The Sample ABA has the highest temperature for 50% weight loss which is 340.98 °C.

3.2. Characterization of microcellulose
3.2.1. Physical appearance of microcellulose. Table 3 depicts the identification and the treatment involved for each fibre. Figure 7 illustrates the physical appearance of alkali treated fibre for production of microcellulose while Figure 8 illustrates the physical appearance of steam treated fibre for production of microcellulose.

Table 3. The identification and treatment involved in each sample.

Sample	Treatment involved
Sample 5 (ABAHU30)	Alkali Treatment + Bleaching +Acid Hydrolysis+ Homogenization + Ultrasonication 30 min
Sample 6 (ABAHU60)	Alkali Treatment + Bleaching +Acid Hydrolysis+ Homogenization + Ultrasonication 60 min
Sample 7 (ABHU30)	Alkali Treatment + Bleaching + Homogenization + Ultrasonication 30 min
Sample 8 (ABHU60)	Alkali Treatment + Bleaching + Homogenization + Ultrasonication 60 min
Sample 9 (SBHU30)	Steam Treatment + Bleaching + Homogenization + Ultrasonication 30 min
Sample 10 (SBHU60)	Steam Treatment + Bleaching + Homogenization + Ultrasonication 60 min
Sample 11 (SBAHU30)	Steam Treatment + Bleaching +Acid Hydrolysis+ Homogenization + Ultrasonication 30 min
Sample 12 (SBAHU60)	Steam Treatment + Bleaching +Acid Hydrolysis+ Homogenization + Ultrasonication 60 min

| ABAHU30 | ABAHU60 | ABHU30 | ABHU60 |

| SBHU30 | SBHU60 | SBAHU30 | SBAHU60 |

Figure 7. Physical appearance of microcellulose after treatment.

According to Figure 7, the colour gradient of cellulose can be observed. The whitish colour of the microcellulose indicates that the cellulose had higher purity as more components are removed from the fibre. Moreover, the surface of the cellulose appears to smoother and in cottony structure compared to previous treatment.

3.2.2. Surface morphology of microcellulose. Figure 8 depicts the SEM image of Microcellulose ABAHU30 while Figure 9 depicts the SEM image of Microcellulose ABAHU60. The treatment time of ultrasonication was varied as for Microcellulose ABAHU30 is 30 min whereas for Microcellulose ABAHU60 is 60 min. As shown in Figure 10 and 11, the longer time of ultrasonication can produce a more uniform shape of fibrils [30].
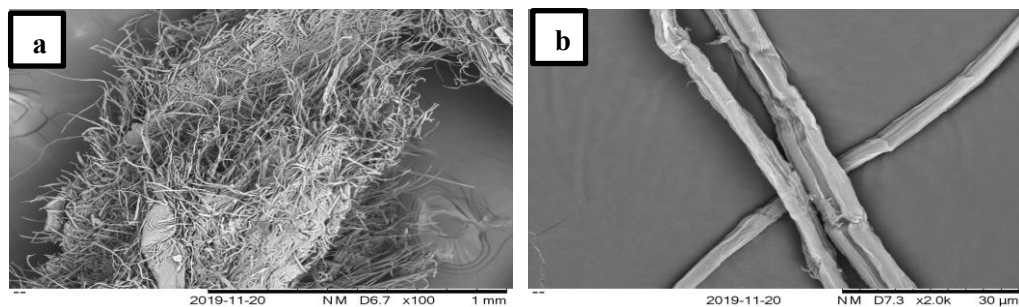

Figure 8. SEM image of Microcellulose ABAHU30 under different magnifications (a) 100× and; (b) 2000×.

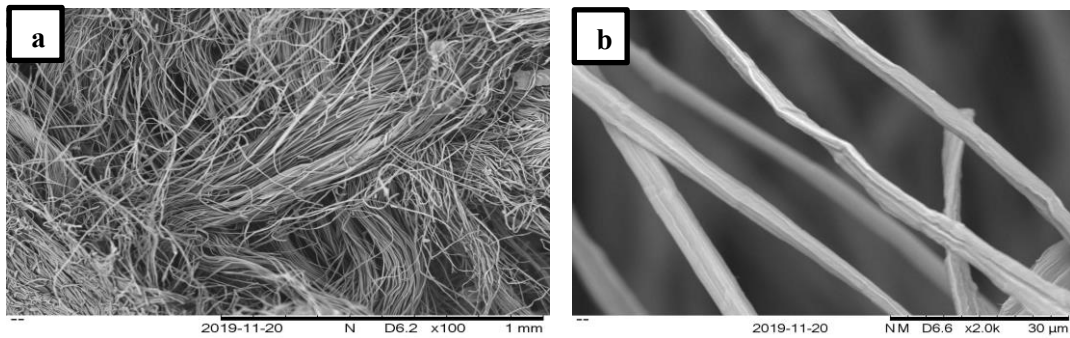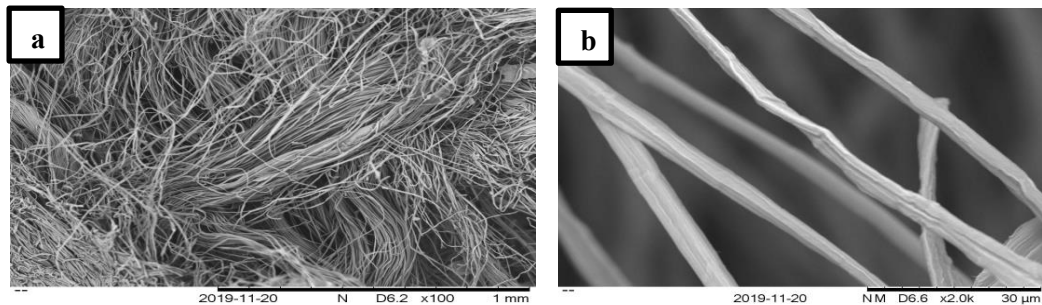**Figure 9.** SEM image of Microcellulose ABAHU60 under different magnifications (a) 100× and; (b) 2000×.

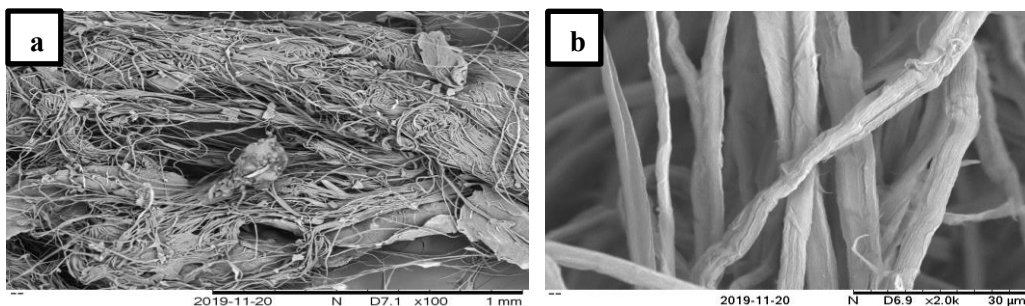**Figure 10.** SEM image of Microcellulose ABAHU60 under different magnifications (a) 100× and; (b) 2000×.

Figure 11. SEM image of Microcellulose ABHU60 under different magnifications (a) 100× and; (b) 2000×.

Microcellulose ABAHU60 and Microcellulose ABHU60 are microcellulose produced by alkali treatment with and without the step of acid hydrolysis. With the additional step of acid hydrolysis, the fibrils produced is more uniform and smooth compare to the cellulose without the step of acid hydrolysis as it helps to further disintegrate the fibrils [25].

Microcellulose SBHU60 and Microcellulose SBAHU60 are microcellulose produced by steam treatment with and without the step of acid hydrolysis. As shown in Figure 12 and 13, the fibrils produced is a silky and smooth appearance fibre and devoid of surface impurities with the additional step of acid hydrolysis. The fibre without the step of acid hydrolysis appeared rough, exists in a big bundle with a lot of impurities attached on the surface. Moreover, it possessed many indented surfaces structures due to defibrillation and washing way of some part of amorphous chemical components
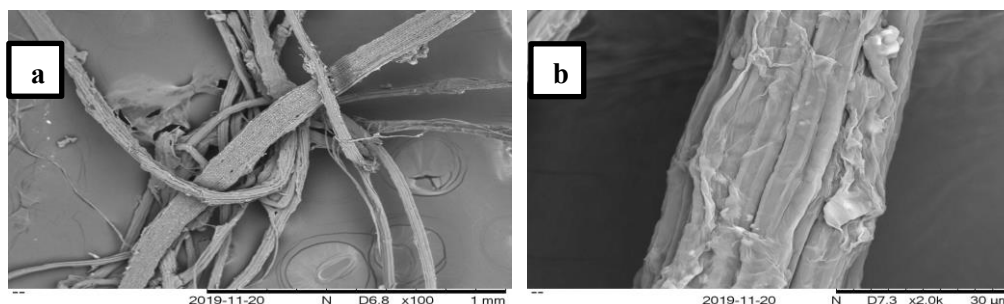
Figure 12. SEM image of Microcellulose SBHU60 under different magnifications (a) 100× and; (b) 2000×.

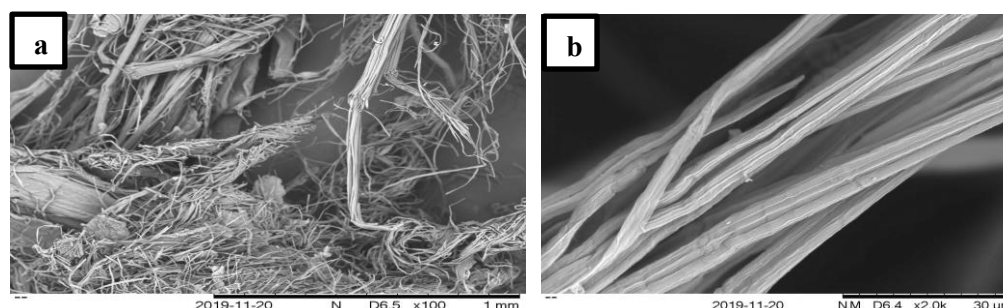**Figure 13.** SEM image of Microcellulose SBAHU60 under different magnifications (a) 100× and; (b) 2000×.

According to the SEM analysis result, it is clearly show that Microcellulose ABAHU60 has the best surface topography compared to the other samples. By referring to Figure 11, the cellulose is more exposed and has a looser structure with uniform fibrils. Moreover, the fibril surface is smoother as the surface impurities are mostly removed through the chemical treatment.

3.2.3. Thermal stability analysis of microcellulose. Table 4 shows the summary of degradation temperature of microcellulose produced compared with pure commercial cellulose. The TGA and DTG reading curve of the Sample 6 and Sample 12 was compared with pure commercial cellulose. Figure 14 which is the TGA graph shows the comparison of weight percent of the fibre with different treatment.

Table 4. Summary of degradation temperature of microcellulose.

Sample	Weight Loss at 100 °C	Temp at 20% Weight Loss	Temp at 50% Weight Loss	Residue at 700 °C
Sample 6 (ABAHU60)	6.98	316.68	349.84	1.47
Sample 12 (SBAHU60)	4.90	298.40	339.38	7.07
Pure Commercial Cellulose	8.52	313.98	339.42	3.03

According to Figure 14, the analysis shows that the microcellulose of Sample 6 (ABAHU60) have similar residual value at 700 °C with commercial cellulose. However, for sample 12, it has higher value of residual char. This may due to the presence of stable aromatic matrix component, lignin [29]. The decomposition of the microcellulose and commercial cellulose shows several stages, indicating the presence of different components that decompose at different temperatures. The degradation of Sample 6 has a higher value, 349.84 °C, than Sample 12 and pure commercial cellulose.
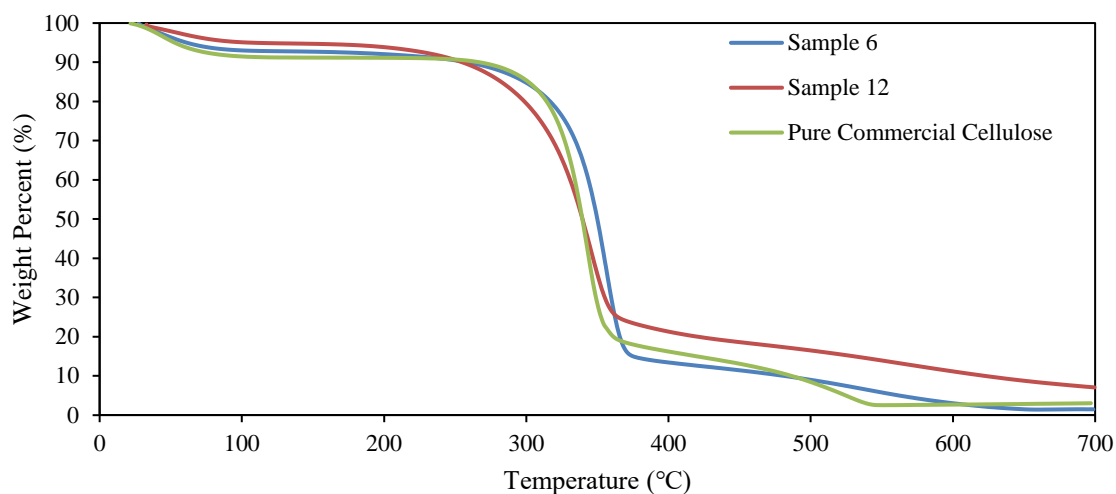
Figure 14. Thermogravimetric Analysis (TGA) graph.

4. Conclusion

In conclusion, the physical appearance, thermal stability, fibre size and surface morphology of cellulose produced from PALF were analysed in this research. The effect of the steam treatment, alkali treatment and acid hydrolysis were analysed through the characterization of cellulose and microcellulose. The alkali treated fibre produce a more uniform fibrils compared to the steam treated fibre. Thus, alkali treatment is more effective compared to steam treatment. Moreover, based on the results, the additional step of acid hydrolysis produces better quality of cellulose. In addition, the longer treatment time of ultrasonication improved the uniformness of the microcellulose and reduce the size of microcellulose produced. For the thermal stability analysis, the high degradation temperature indicates that the cellulose able to withstand high temperature, and contains less moisture, compared to the other samples. Based on the surface morphology analysis, it can be clearly shown that the treatment performed are able to reduce hemicellulose, lignin, and surface impurities of the fibre.

ABAHU60 showed the best result among all samples, where it has the highest degradation temperature of 349 °C. Moreover, it exhibits the excellent surface morphology and uniform fibrils structure. This microcellulose showed similar trend of TGA and DTG graph with pure commercial cellulose. Therefore, the chemical treatment of alkali treatment, bleaching and acid hydrolysis with the mechanical treatment of homogenization and 60 min ultrasonication can produce cellulose with good thermal stability and morphology.

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