

Effect of Steam and Bleaching Treatment on the Characteristics of Pineapple Leaves Fibre Derived Cellulose

Surenthiran Gnanasekaran, Siti Nur Najihah Muslih, Jun Haslinda Shariffuddin, and Noor Ida Amalina Ahamad Nordin*

Faculty of Chemical and Process Engineering Technology, Universiti Malaysia Pahang, Lebuhraya Tun Razak, 26300 Gambang, Pahang, Malaysia

ABSTRACT

Pineapple leaf fibres (PALF) is one of the abundant residues generated from pineapple plantation. The residues are left on the plantation for nutrient cycling or burning, and this circumstance leads to environmental issues. PALF has high cellulose content among other natural fibres. Cellulose is a reinforcing element that exists as whisker-like microfibrils and has a long-chain structure. In this study, cellulose produced from PALF was treated by steam and chemical treatment. The fibre was treated with steam at 121°C, a pressure of 21 psi for 30 or 60 min. Next, the steam-treated fibre was treated with 5 wt% sodium chlorite (NaClO₂) solution with pH adjusted between 4 and 5 for 90 min. The condition was varied with three different temperatures, which were room temperature, 50, and 70°C. Then, the bleached fibre was treated with 5 wt% sodium hydroxide (NaOH) at room temperature for 3 h. After the treatments, the fibre was analysed for its thermal stability, morphology, and chemical composition. Cellulose obtained from the treatment condition of steam for 60 min, bleaching at 70°C, and alkali treatment at room temperature expressed the highest degradation temperature of 276°C at 20% weight loss, percentage of cellulose of 86% and lowest moisture content (8%) compared to others sample. It also had an excellent surface morphology with finest fibril disintegration. It showed longer steam treatment (60 min) degrading more hemicellulose; and bleaching treatment at high temperature (70°C), increasing the rate of oxidative delignification. In conclusion, the suggested treatment provides a simple but efficient method to isolate cellulose that can be used for various types of applications.

ARTICLE INFO

Article history:

Received: 10 February 2020

Accepted: 13 November 2020

Published: 31 December 2020

DOI: <https://doi.org/10.47836/pjst.28.S2.11>

E-mail addresses:

surenthirangnanasekaran@gmail.com (Surenthiran Gnanasekaran)

snajihah95@gmail.com (Siti Nur Najihah Muslih)

junhaslinda@ump.edu.my (Jun Haslinda Shariffuddin)

idamalina@ump.edu.my (Noor Ida Amalina Ahamad Nordin)

* Corresponding author

Keywords: Cellulose, chemical treatment, pineapple leaf fibre (PALF), steam treatment, thermal degradation

INTRODUCTION

Pineapple is a perennial herbaceous plant with 0.75 – 1.5 m height and 0.9 – 1.2 m spread. It has a short stem with dark green colour. This tropical plant originated from Southeast America and was cultivated in Tanah Melayu in 1922 (Najeeb et al., 2020). In 2015, pineapple plantation grown in Malaysia covered an area of 10 847 hectares with an estimated fruit production of 272 570 metric tons (Abd-Halim, 2016). Several agencies are responsible for the success of this industry such as the Federal Agricultural Marketing Authority (FAMA), Malaysian Pineapple Industry Board (MPIB), and some local universities (MPIB, 2010).

While Malaysia is known as one of the largest pineapple producers in Asia, at the same time, it creates a large quantity of waste from this industry (Najeeb et al., 2020; Shafie et al., 2012). Pineapple waste is not fully utilised and usually burnt, and this circumstance may lead to air pollution. Pineapples are mostly consumed for nutritional purposes either as a fresh product or processed fruit. Only 20% of the pineapple is canned for nutrition usage and the rest, which includes leaves, peeled skin, core, base, and crown, is discarded as waste (Shafie et al., 2012).

Pineapple leaf fibre (PALF) is one of the abundantly available waste materials generated from the pineapple industry (Sena Neto et al., 2013; Wan Nadirah et al., 2012). Usually, PALF is left on the plantation for nutrient cycling (Ahmed et al., 2000). Attempts have been made to utilise PALF for feedstock and energy production (Asim et al., 2015). Besides, the use of natural fibres for composites, as alternatives to traditional reinforcement materials offers numerous advantages such as low cost, high specific properties, low energy consumption, low density, biodegradability, flexibility and availability of a wide variety of fibres around the globe (Benyahia et al., 2014). Components of natural fibre consist of cellulose, hemicellulose and lignin (Satha et al., 2020). Cellulose has relatively high strength, high stiffness, low density and good thermal stability (Mahardika et al., 2018). Higher content of cellulose (80%) in PALF compared to other natural fibres makes it promising candidate for reinforcing biocomposite (Cherian et al., 2010; Huda et al., 2008). The application of cellulose includes household items (Lavanya et al., 2011), wall insulation (Ashik et al., 2015), packaging (Sena Neto et al., 2013) and others. Numerous studies have shown the potential of PALF as reinforced material in a polymer matrix (Garcia et al., 2016).

The objective of this study was to produce cellulose from PALF using steam and chemical treatment. Recently much work has been done on pre-treatment to extract the cellulose. Nevertheless, little has been done about the physiochemical treatment. These may increase the accessibility of cellulose using the mild treatment. Therefore, in this study, PALF was treated with the different retention time of steam treatment and followed by the different temperature of bleaching treatment to maximize the removal of hemicellulose and lignin with less degradation of cellulose.

MATERIAL AND METHODS

Materials

Pineapple leaves were collected from Pekan Pina Sdn. Bhd., Pekan, Pahang, Malaysia. Sodium hydroxide, sodium chlorite and sulphuric acid were obtained from Sigma Aldrich Malaysia.

Methods

Fibre Preparation. Pineapple leaves were washed with tap water to remove dirt. Cleaned leaves were cut into 1 to 2 cm for storage purposes. The fibre was dried in oven at 70°C for 24 h to prevent fungal growth and contamination. Samples were stored in sealed plastic bags at room temperature until further use (Prado & Spinacé, 2019).

Steam Treatment of Fibre. PALF was treated with steam at 121°C and 21 psi for 30 or 60 min using Hirayama HVE-50 Autoclave Sterilizer (Cherian et al., 2010). After steam treatment, the fibre was washed with distilled water and dried overnight at 70°C (Santos et al., 2013).

Fibre Bleaching. Steam-treated fibre (1 g fibre:50 mL solution) was bleached using 5 wt% sodium chlorite solution with pH adjusted between 4 and 5 using sulphuric acid for 90 min under three different conditions: room temperature, 50, and 70°C in Jeio Tech BS-06 Water Bath. After bleaching, the treated fibre was washed with distilled water and dried overnight at 70°C (Santos et al., 2013).

Alkaline Treatment of Fibre. Bleached fibre (1g fibre:50 mL solution) was treated using 5 wt% sodium hydroxide (NaOH) solution at room temperature for 3h (Prado & Spinacé, 2019). After that, the treated fibre was washed with distilled water and dried overnight at 70°C (Santos et al., 2013). The dried samples were ground using Retsch Ultra Centrifugal Mill ZM 200 for analysis purposes.

Overall treatment process was summarized in Figure 1.

Thermal Stability Analysis. The samples were tested using Hitachi STA7200 Thermal Analysis System for thermogravimetric analysis (TGA). The measurements were conducted in a nitrogen atmosphere at a rate of 100 mL/min by constant heating at 10°C/min with ramping temperature ranging from 30 to 600°C. About 4–7 mg of the sample was placed in a ceramic crucible and tested (Abraham et al., 2011). The moisture content of the sample was obtained from TG analysis at temperature of 150°C.

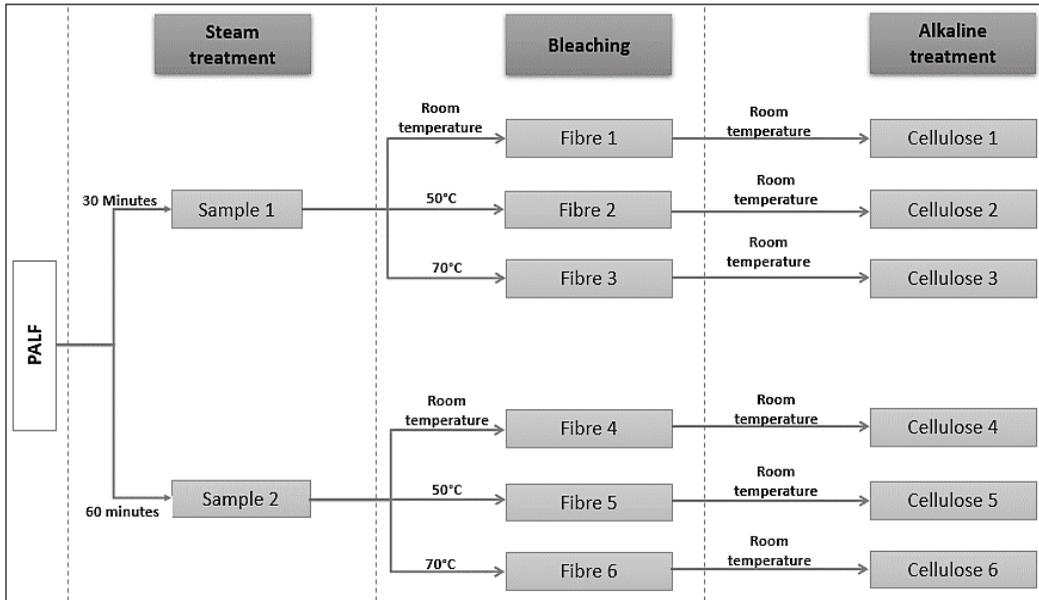


Figure 1. Summarized treatment on PALF by steam, bleaching and alkaline treatment to isolate cellulose

Morphology Analysis. Hitachi TM3030 Plus Tabletop Microscope was used for scanning electron microscopy (SEM) to obtain the surface morphology of cellulose. Prior to SEM analysis, samples were coated with platinum using sputtering techniques (Cherian et al., 2010).

Chemical Composition of Cellulose. Cellulose samples were analysed for its chemical composition. The isolated cellulose was bleached using 5 wt% sodium chlorite solution with pH adjusted to 4–5 using sulphuric acid at 70°C to obtain the percentage of lignin. The bleached fibre was washed with distilled water and dried overnight at temperature of 70°C. Next, the bleached sample was immersed in 5 wt% sodium hydroxide solutions at room temperature for 24 h to obtain the percentage of hemicellulose. After that, the treated fibre was washed with distilled water and dried overnight at 70°C. The residue left contained only cellulose (Nordin et al., 2017). Equations 1 to 3 were used to calculate the percentage of chemical composition in the samples.

$$Lignin \% = \frac{Initial\ weight\ (g)_{dry} - Weight\ after\ extraction,\ NaClO_2\ (g)_{dry}}{Initial\ weight\ (g)_{dry}} \times 100\% \quad [Eq. 1]$$

$$Hemicellulose \% = \frac{Weight\ after\ extraction,\ NaClO_2\ (g)_{dry} - Weight\ after\ extraction,\ NaOH\ (g)_{dry}}{Initial\ weight\ (g)_{dry}} \times 100\% \quad [Eq. 2]$$

$$\text{Cellulose \%} = \frac{\text{The residue of NaOH extraction (g)}_{\text{dry}}}{\text{Initial weight (g)}_{\text{dry}}} \times 100\% \quad [\text{Eq. 3}]$$

RESULTS AND DISCUSSION

Physical Appearance of Treated Fibre

Physical Appearance of Steam-treated PALF. Figure 2 shows the physical appearances of the steam-treated fibre where (a) was 30 min and (b) was 60 min of steam treatment. As portrayed in Figure 2, PALF treated with steam for 60 min (sample b) had a darker brown colour compared to 30 min sample (sample a). Hemicellulose has low thermal degradation, which makes it degrade easier than cellulose and lignin.

Hydrolysis of hemicellulose takes place during steam treatment, by breaking down into monosugar which consists of numerous amounts of hydroxyl group. However, under the high temperature of steam treatment, the total number of hydroxyl groups consisting of O and H decreases due to degradation of monosugar. This mechanism also known as caramelization of monosugar (Stelte, 2013). Hence, PALF that undergoes steam treatment for 60 min is darker than 30 min treated PALF, due to longer residence time of steam treatment causes high degradation of hemicellulose by eliminating hydroxyl group and increasing of carbon content.

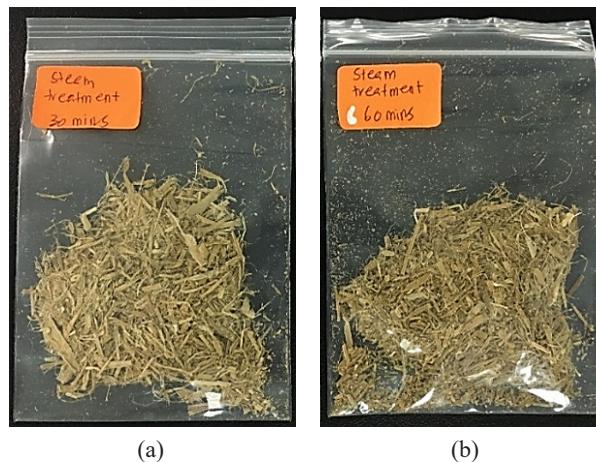


Figure 2. Physical appearance of fibre after steam treatment for (a) 30 min and (b) 60 min

Physical Appearance of Steamed_Bleached PALF. Figure 3 shows the physical appearance of fibre after steam treatment followed by bleaching treatment. Bleaching temperature was varied at room temperature (sample 1 and 4), 50°C (sample 2 and 5) and 70°C (sample 3 and sample 6). As seen in Figure 3, fibre with bleaching temperature of 70°C has the lightest colour compared to other samples either the samples were treated with steam for 30 or 60 min. NaClO₂ act as an oxidative agent in delignification process

by oxidizing aromatic ring or benzoquinone structure of lignin and hemicellulose to change its solubility (Wu et al., 2019). It also may react with the side chain carbon-carbon double bond and carbonyl bond of lignin for further oxidative degradation, leading to lignin removal (Hubbell & Ragauskas, 2010; Lee et al., 2014). According to Listiyani et al. (2012) temperature of bleaching treatment gives an significant impact on oxidative delignification of PALF. When the temperature of bleaching treatment is increased, the rate of oxidative delignification will increase which causes more lignin and hemicellulose to solubilize (Hefti, 1960). Hence, the lighter colour of fibre demonstrates that more lignin and hemicellulose were oxidized and removed during the treatment. The fibre surface was still rough after undergoing the treatments, indicating that in order to produce cellulose, further treatment is needed to improve the fibre surface and size.



Figure 3. Physical appearance of fibre after steam and bleaching treatment; (a) steam treatment 30 min with bleaching treatment at (1) room temperature; (2) 50°C; (3) 70°C (b) steam treatment 60 min with bleaching treatment at (4) room temperature; (5) 50°C; (6) 70°C

Physical Appearance of Cellulose (Steamed, bleached, and alkaline-treated PALF).

Figure 4 shows the physical appearance of cellulose isolated from PALF. The cellulose was obtained after steam, bleached and finally was alkali treated at room temperature for

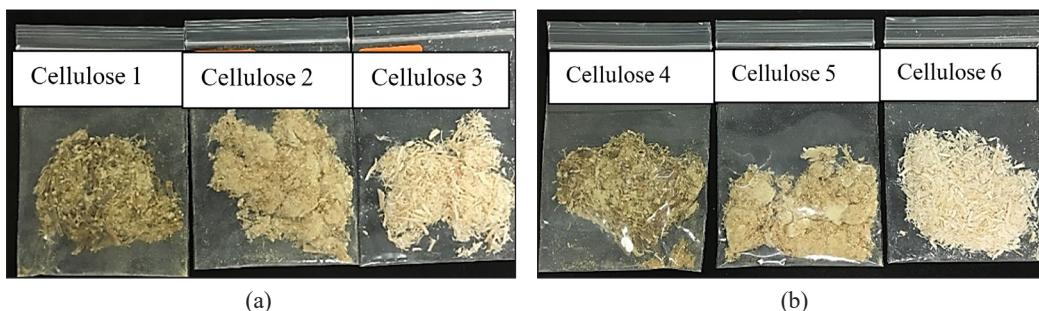


Figure 4. Physical appearance of cellulose after steamed; bleached; and alkaline treated: (a) steam 30 min: bleached (Cellulose 1) room temperature; (Cellulose 2) 50°C; (Cellulose 3) 70°C, then alkaline treated at room temperature, 3 h and (b) steam 60 min: bleached at (Cellulose 4) room temperature; (Cellulose 5) 50°C; (Cellulose 6) 70°C, then alkaline treated at room temperature, 3 h

3h as detailed in Figure 1. The sample portrays the difference in the colour gradient of the cellulose. A lighter colour of cellulose indicates that more components have been removed during the treatment (Kouadri & Satha, 2018; Sharma et al., 2018). The cellulose surface was also smoother and appeared cottony after the treatment, compared to that from the previous treatment. Moreover, the major effect of these alkaline treatment is to de-lignify the biomass, disrupt the connection between cellulose and alter the structure of treated biomass to increase the accessibility of cellulose (Sindhu et al, 2014). Since cellulose is moderately resistant to alkali treatment, it is an effective way to remove lignin and hemicellulose with less degradation of the cellulose component (Mishra et al., 2004).

Thermal Stability of Cellulose. Figure 5 shows the thermal degradation temperature of cellulose, while Figure 6 illustrates the differential thermal gravimetry of cellulose.

All samples had a one-step degradation between 200 and 400°C except for Cellulose 1. Cellulose 1 demonstrated two-step degradation, at temperature of 200 to 300°C and at temperature of 300 to 400°C. The first weight loss transition occurred between 50 and 150°C due to the evaporation of water molecules in the cellulose (Satha et al., 2020). The second transition, which occurred from 180 to 350°C, is mainly contributed to the degradation of the cellulose component, and low molecular weight lignin components (Najeeb et al., 2020; Paluvai et al., 2015). The third transition occurred between 350 to 500°C due to the degradation of lignin, which has a higher molecular weight.

Cellulose 1 may content more percentage of lignin, due to bleaching treatment conducted at room temperature. It seems that room temperature is not an effective condition for bleaching of lignin. That may explain the trend of thermal degradation of Cellulose 1. Cellulose 4, Cellulose 5, and Cellulose 6 experienced more weight loss at temperature

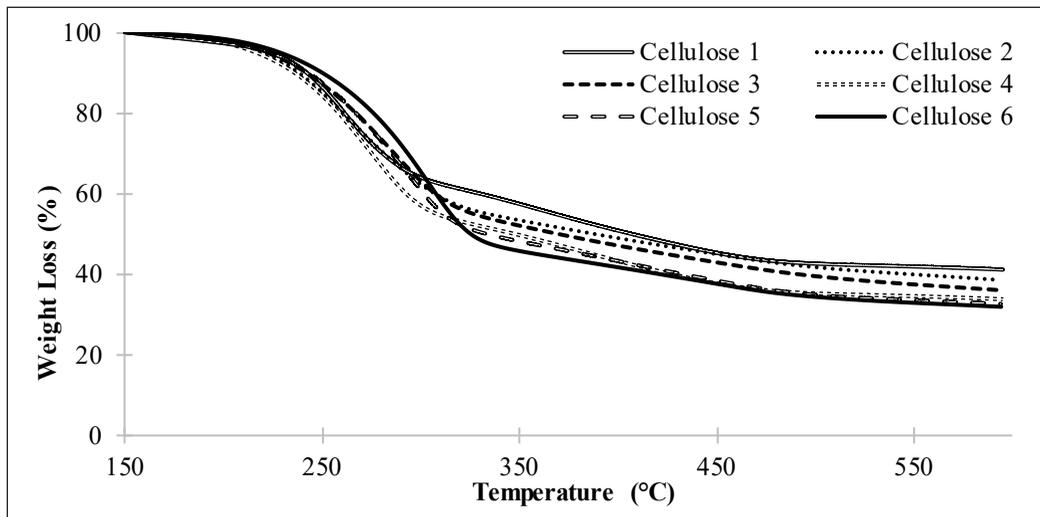


Figure 5. Weight loss of cellulose

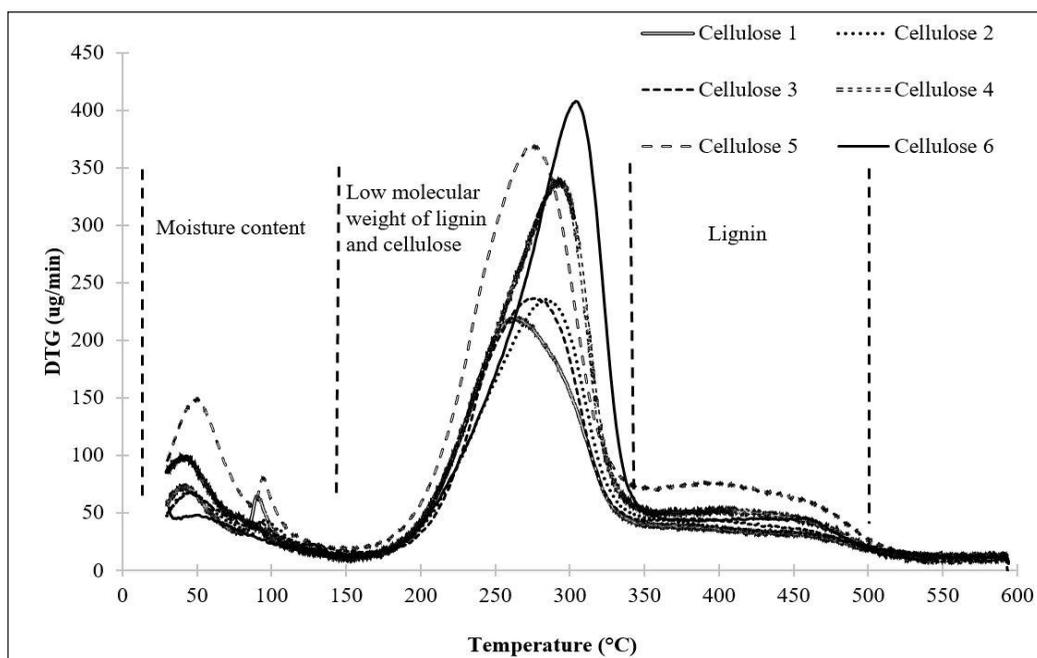


Figure 6. DTG of cellulose

range of 180 to 350°C compared to Cellulose 1, 2, and 3. It could also be observed that the residue at 600°C, is mainly lignin, for Cellulose 4 to 6 was lower compared to Cellulose 1 to 3. Thus, it can be concluded that the percentage of cellulose was higher in samples 4 to 6, and contained less hemicellulose and lignin due to more percentage of these components being removed by the treatment (Lee et al., 2020). But among Cellulose 4 to 6, Cellulose 6 recorded the highest cellulose content. It may be because bleaching at high temperature makes the lignin decomposition by chloride higher (Hefti, 1960). It was also reported by Wan Nadirah et al. (2012) that an increase in the amount of cellulose would increase the percentage of crystallinity of the fibre. Increasing the percentage of crystallinity may also improve its thermal stability (Djafari Petroudy, 2017).

Table 1 summarises the degradation of cellulose at 5%, 20%, and 50% weight loss. As mentioned, temperature around 200°C will degrade low molecular weight of lignin, thus Cellulose 1 had the lowest temperature for 5% weight loss. The same trend occurred at 20% of weight loss. However, at the temperature above 350°C, the lignin component with high molecular weight would start to degrade. Therefore, at this range of temperature, it was observed Cellulose 6 had the lowest degradation temperature due to less lignin composition and more cellulose. This results were in agreement with physical appearance of light brown colour of Cellulose 6 compared to Cellulose 1 (Kouadri & Satha, 2018). Cellulose 6 also had the lowest percentage of residue at 600°C, which was 32.02%, showing the low percentage of lignin. (Lee et al., 2020; Rambabu et al., 2016).

Table 1
Summary of thermal degradation of cellulose

Cellulose	Temperature for 5% weight loss (°C)	Temperature for 20% weight loss (°C)	Temperature for 50% weight loss (°C)	Residue at 600°C (%)
Cellulose 1	216.16	261.22	406.59	41.25
Cellulose 2	220.13	258.22	387.60	38.68
Cellulose 3	221.47	260.48	369.30	36.15
Cellulose 4	222.74	266.82	347.30	33.40
Cellulose 5	223.39	266.93	333.15	32.73
Cellulose 6	228.19	275.80	325.16	32.02

Moisture content of the samples were obtained from the TG analysis at temperature of 150°C, as presented in Table 2. The moisture content of Cellulose 1 to 6 was reduced and Cellulose 6 had the lowest moisture content (8.22%). This may be due to closer packing of the cellulose molecules and the increase in PALF crystallinity (Mohamed et al., 2014). Low moisture content may help to delay early damage of composite reinforced with cellulose due to swelling effect. Swelling may cause structural modification of the cellulose fibre leading to reduced mechanical properties of biocomposite (Célino et al., 2014).

Table 2
Moisture content of cellulose at 150°C

Cellulose	Moisture content at 150°C (%) (dry basis)
Cellulose 1	14.15
Cellulose 2	12.72
Cellulose 3	12.52
Cellulose 4	12.07
Cellulose 5	12.03
Cellulose 6	8.22

Cellulose Morphology. Figure 7 shows the SEM image of untreated PALF. Untreated PALF is glued with lignin and hemicellulose which causes all the fibrils associated with bundles. Impurities could be seen deposited on the fibre surface.

Figures 8 depicts the SEM images of the samples. As illustrated in the figure, the loose structure of the fibres is noticeable. Due to the treatments performed to the PALF, the fibril surface is exposed, making hemicellulose and lignin accessible to steam or

chemical treatment, and also surface impurities were removed. The treatments aided in breaking down the lignocellulosic components and solubilised the lignin and hemicellulose

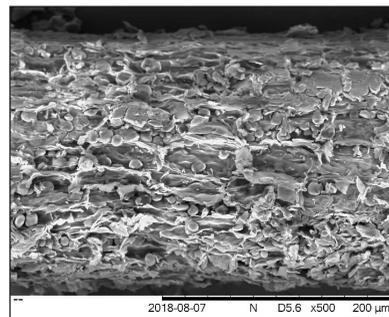


Figure 7. Impurities is deposited on the surface of untreated PALF

to expose the hidden cellulose. In addition, the treatments also assisted in further fibril disintegration.

As shown in Figure 8 (f), the Cellulose 6 is more exposed and has a looser structure, as more protective layers have been removed due to the longer duration of steam treatment and a higher temperature of bleaching (Rambabu et al., 2016). Moreover, due to the alkaline treatment, the fibril surface is smoother than other samples.

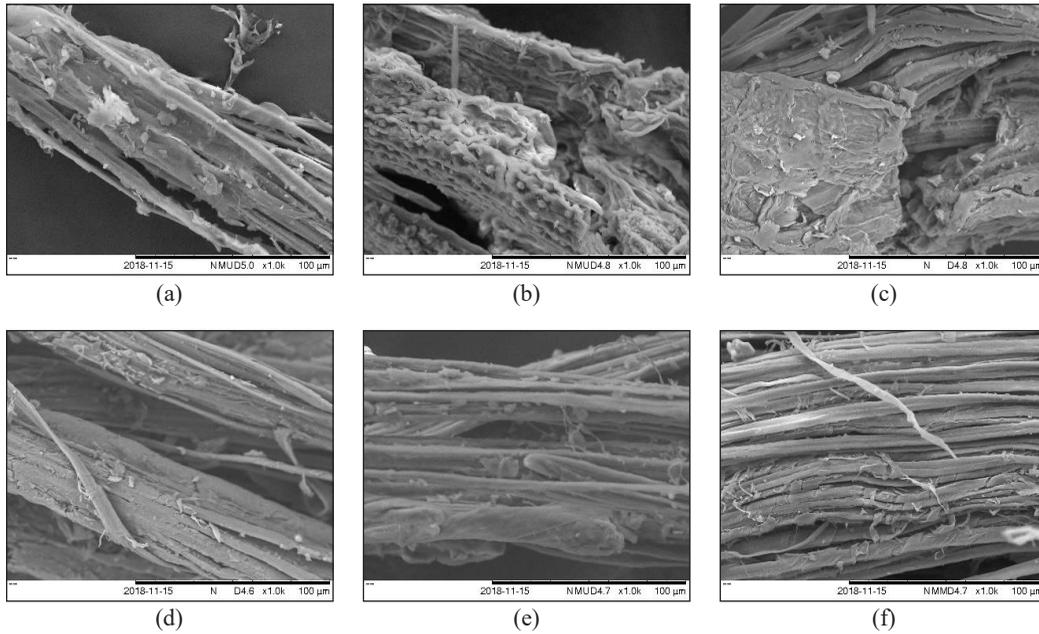


Figure 8. SEM images of (a) Cellulose 1, (b) Cellulose 2, (c) Cellulose 3, (d) Cellulose 4, (e) Cellulose 5 and (f) Cellulose 6

Chemical Composition of Cellulose. The chemical compositions of Cellulose 1–6 are listed in Table 3. Cellulose 6 has the highest percentage of cellulose content and lowest hemicellulose and lignin, which are 85.90%, 3.74%, and 10.36%, respectively, compared to the others sample.

Table 3
Chemical composition of the samples

Cellulose	Chemical composition (wt %)		
	Cellulose	Hemicellulose	Lignin
Cellulose 1	48.91	12.42	38.68
Cellulose 2	54.59	10.17	35.25
Cellulose 3	60.85	7.40	31.76
Cellulose 4	67.99	6.64	25.36
Cellulose 5	74.84	5.64	19.52
Cellulose 6	85.90	3.74	10.36

Steam treatment and alkali treatment did remove some percentage of hemicellulose and lignin as reported by others (Cherian et al., 2010; Rambabu et al., 2016). Longer steam treatment could help in removing hemicellulose content. It can be observed in Cellulose 1 and Cellulose 4, where percentage of hemicellulose was 50% lower in Cellulose 4 when steam treated was 30 min longer. Steam treatment may also contribute in reducing lignin component as Cellulose 4 had 30% lower of lignin composition compared to Cellulose 1.

Bleaching treatment is known to be able to reduce the percentage of lignin (Sena Neto et al., 2013; Garcia et al., 2016; Wu et al., 2019). However, bleaching at room temperature was not efficient to remove lignin content. This result was in agreement with the physical appearance and TG analysis. Cellulose 1 had a darker colour compared to other samples and had higher residue, showing the percentage of lignin was higher.

It can be concluded that 60 min of steam treatment, bleaching at moderate to high temperature and alkali treatment had better efficiency in modifying fibre composition. The treatments significantly removed more hemicellulose component and solubilised lignin giving more purified cellulose.

CONCLUSIONS

The physical appearance, thermal stability, morphology, and chemical composition of cellulose produced from PALF were studied. For physical appearance, a lighter colour indicates that more components have been removed during the treatment especially lignin. All cellulose samples showed the same trend of weight loss, with a one-step degradation that occurred between 200 and 350°C, except for Cellulose 1. The improved degradation temperature indicates that the Cellulose 6 can withstand moderate to high temperature and contains less moisture compared to the other samples. For the morphological analysis of cellulose, the loose structure of the fibres can be clearly noticed. Due to the treatments performed to the PALF, the fibril surface is exposed as most of the components inside the fibre, such as hemicellulose and lignin, and surface impurities have been removed. Cellulose 6 shows the best result among all samples, where it has the highest cellulose content of 85.90%. It also has the highest degradation temperature of 275.8°C at 20% weight loss and the lowest moisture content of 8.22%. Cellulose 6 exhibits excellent surface morphology, where the fibril is more exposed and has a looser structure. In conclusion, the combination of steam treatment for a longer period and bleaching treatment at a high temperature can remove hemicellulose and lignin significantly and increase the accessibility of cellulose with good thermal stability and morphology.

ACKNOWLEDGEMENT

We would like to thank Universiti Malaysia Pahang for the financial assistance through the research grant RDU1703177.

REFERENCES

- Abd-Halim, N. (2016). Policy intervention for the development of the pineapple industry in Malaysia. *Food and Fertilizer Technology Center for the Asian and Pacific Region*, 65, 79-83.
- Abraham, E., Deepa, B., Pothan, L., Jacob, M., Thomas, S., Cvelbar, U., & Anandjiwala, R. (2011). Extraction of nanocellulose fibrils from lignocellulosic fibres: A novel approach. *Carbohydrate Polymers*, 86(4), 1468-1475. doi:10.1016/j.carbpol.2011.06.034
- Ahmed, O. H., Husni, M. H., Hanafi, M. M., Syed Omar, S. R., & Anuar, A. R. (2000). Macronutrients distribution and cycling of pineapple planted on tropical peat. *Pertanika Journal of Tropical Agricultural Science*, 23(2), 89 - 95.
- Ashik, K. P., & Sharma, R. S. (2015). A Review on mechanical properties of natural fiber reinforced hybrid polymer composites. *Journal of Minerals and Materials Characterization and Engineering*, 3(05), 420–426. doi:10.4236/jmmce.2015.35044
- Asim, M., Abdan, K., Jawaid, M., Nasir, M., Dashtizateh, Z., Ishak, M., & Hoque, M. E. (2015). A review on pineapple leaves fibre and its composites. *International Journal of Polymer Science*, 2015, 1 - 16. doi:10.1155/2015/950567
- Benyahia, A., Merrouche, A., Rahmouni, Z. E. A., Rokbi, M., Serge, W., & Kouadri, Z. (2014). Study of the alkali treatment effect on the mechanical behavior of the composite unsaturated polyester-Alfa fibers. *Mechanics & Industry*, 15(1), 69 – 73. doi:10.1051/meca/2013082
- Céline, A., Fréour, S., Jacquemin, F., & Casari, P. (2014). The hygroscopic behavior of plant fibers: A review. *Frontiers in Chemistry*, 2014(1), 1–12. doi:10.3389/fchem.2013.00043
- Cherian, B. M., Leão, A. L., De Souza, S. F., Thomas, S., Pothan, L. A., & Kottaisamy, M. (2010). Isolation of nanocellulose from pineapple leaf fibres by steam explosion. *Carbohydrate Polymers*, 81(3), 720-725. doi:10.1016/j.carbpol.2010.03.046
- Djafari Petroudy, S. R. (2017). Physical and mechanical properties of natural fibers. In Mizi, F., & Feng, F. (Ed.), *Advanced high strength natural fibre composites in construction* (pp. 59 – 83). Cambridge, England: Woodhead Publishing. doi: 10.1016/B978-0-08-100411-1.00003-0
- García, A., Gandini, A., Labidi, J., Belgacem, N., & Bras, J. (2016). Industrial and crop wastes: A new source for nanocellulose biorefinery. *Industrial Crops and Products*, 93, 26–38. doi:10.1016/j.indcrop.2016.06.004
- Hefti, H. (1960). Sodium chlorite bleaching. *Textile Research Journal*, 30(11), 861–867. doi:10.1177/004051756003001108
- Hubbell, C. A., & Ragauskas, A. J. (2010). Effect of acid-chlorite delignification on cellulose degree of polymerization. *Bioresource Technology*, 101(19), 7410–7415. doi:10.1016/j.biortech.2010.04.029
- Huda, M. S., Drzal, L. T., Mohanty, A. K., & Misra, M. (2008). Effect of chemical modifications of the pineapple leaf fiber surfaces on the interfacial and mechanical properties of laminated biocomposites. *Composite Interfaces*, 15(2–3), 169–191. doi:10.1163/156855408783810920
- Kouadri, I., & Satha, H. (2018). Extraction and characterization of cellulose and cellulose nanofibers from citrullus colocynthis seeds. *Industrial Crops & Products*, 124, 787 – 796. doi:10.1016/j.indcrop.2018.08.051

- Lavanya, D., Kulkarni, P., Dixit, M., Raavi, P. K., Krishna, L., & Vamsi, N. (2011). Sources of cellulose and their applications - A review. *International Journal of Drug Formation and Research*, 2(6), 19-38.
- Lee, C. H., Khalina, A., Lee, S. H., Padzil, F. N. M., & Ainun, Z. M. A. (2020). Physical, morphological, structural, thermal and mechanical properties of pineapple leaf fibers. In Jawaid, M., Asim, M., Md. Tahir, P., & Nasir, M. (Ed.), *Green energy and technology* (pp. 91 – 121). Singapore: Springer. doi:10.1007/978-981-15-1416-6_6
- Lee, H. V., Hamid, S. B. A., & Zain, S. K. (2014). Conversion of lignocellulosic biomass to nanocellulose: Structure and chemical process. *Scientific World Journal*, 2014, 1 – 20. doi:10.1155/2014/631013
- Listiyani, M. A. D., Campbell, R. E., Miracle, R. E., Barbano, D. M., Gerard, P. D., & Drake, M. A. (2012). Effect of temperature and bleaching agent on bleaching of liquid Cheddar whey. *Journal of Dairy Science*, 95(1), 36–49. doi:10.3168/jds.2011-4557
- Mahardika, M., Abral, H., Kasim, A., Arief, S., & Asrofi, M. (2018). Production of nanocellulose from pineapple leaf fibers via high-shear homogenization and ultrasonication. *Fibers*, 6(2), 1–12. doi:10.3390/fib6020028
- Mishra, S., Mohanty, A. K., Drzal, L. T., Misra, M., & Hinrichsen, G. (2004). A review on pineapple leaf fibers, sisal fibers and their biocomposites. *Macromolecular Materials and Engineering*, 289(11), 955–974. doi:10.1002/mame.200400132
- MPIB. (2010). *Peneraju industri nanas negara*. Johor Bahru, Malaysia: Malaysian Pineapple Industry Board.
- Mohamed, A. R., Sapuan, S. M., & Khalina, A. (2014). Mechanical and thermal properties of josapine pineapple leaf fiber (PALF) and PALF-reinforced vinyl ester composites. *Fibers and Polymers*, 15(5), 1035–1041. doi:10.1007/s12221-014-1035-9
- Najeeb, M. I., Sultan, M. T. H., Yoshito Andou, Shah, A. U. M., Kubra Eksiler, Jawaid, M., & Ariffin, A. H. (2020). Characterization of silane treated Malaysian yankee pineapple AC6 leaf fiber (PALF) towards industrial applications. *Journal of Materials Research and Technology*, Article in press, 1-12. doi:10.1016/j.jmrt.2020.01.058
- Nordin, N. I. A. A., Ariffin, H., Hassan, M. A., Shirai, Y., Ando, Y., Ibrahim, N. A., & Yunus, W. M. Z. W. (2017). Superheated steam treatment of oil palm mesocarp fiber improved the properties of fiber-polypropylene biocomposite. *BioResources*, 12(1), 68-81. doi:10.15376/biores.12.1.68-81
- Paluvai, N. R., Mohanty, S., & Nayak, S. K. (2015). Studies on thermal degradation and flame retardant behavior of the sisal fiber reinforced unsaturated polyester toughened epoxy nanocomposites. *Journal of Applied Polymer Science*, 132(24), 15–17. doi:10.1002/app.42068
- Prado, K. S., & Spinacé, M. A. S. (2019). Isolation and characterization of cellulose nanocrystals from pineapple crown waste and their potential uses. *International Journal of Biological Macromolecules*, 122, 410–416. doi:10.1016/j.ijbiomac.2018.10.187
- Rambabu, N., Panthapulakkal, S., Sain, M., & Dalai, A. K. (2016). Production of nanocellulose fibers from pinecone biomass: Evaluation and optimization of chemical and mechanical treatment conditions on mechanical properties of nanocellulose films. *Industrial Crops and Products*, 83, 746–754. doi:10.1016/j.indcrop.2015.11.083

- Santos, R. M. dos, Flauzino Neto, W. P., Silvério, H. A., Martins, D. F., Dantas, N. O., & Pasquini, D. (2013). Cellulose nanocrystals from pineapple leaf, a new approach for the reuse of this agro-waste. *Industrial Crops and Products*, 50, 707–714. doi:10.1016/j.indcrop.2013.08.049
- Satha, H., Kouadri, I., & Benachour, D. (2020). Thermal, structural and morphological studies of cellulose and cellulose nanofibers extracted from bitter watermelon of the Cucurbitaceae family. *Journal of Polymers and the Environment*, 28(7), 1914-1920. doi:10.1007/s10924-020-01735-6
- Sena Neto, A. R., Araujo, M. A., Souza, F. V., Mattoso, L. H., & Marconcini, J. M. (2013). Characterization and comparative evaluation of thermal, structural, chemical, mechanical and morphological properties of six pineapple leaf fibre varieties for use in composites. *Industrial Crops and Products*, 43, 529 - 537. doi:10.1016/j.indcrop.2012.08.001
- Sindhu, R., Pandey, A., & Binod, P. (2014). Alkaline treatment. In Pandey, A., Negi, S., Binod, P. & Larroche, C. (Ed.), *Pretreatment of biomass: Processes and technologies* (pp. 51–60). Amsterdam, Netherlands: Elsevier. doi:10.1016/B978-0-12-800080-9.00004-9
- Shafie, S. M., Mahlia, T. M. I., Masjuki, H. H., & Ahmad-Yazid, A. (2012). A review on electricity generation based on biomass residue in Malaysia. *Renewable and Sustainable Energy Reviews*, 16(8), 5879-5889. doi:10.1016/j.rser.2012.06.031
- Sharma, A., Thakur, M., Bhattacharya, M., Mandal, T., & Goswami, S. (2018). Commercial application of cellulose nano-composites – A review. *Biotechnology Reports*, 21, 1-15. doi:10.1016/j.btre.2019.e00316
- Stelte, W. (2013). *Steam explosion for biomass pre-treatment*. Gregersensevej, Denmark: Danish Technological Institute.
- Wan Nadirah, W. O., Jawaid, M., Al Masri, A. A., Abdul Khalil, H. P. S., Suhaily, S. S., & Mohamed, A. R. (2012). Cell wall morphology, chemical and thermal analysis of cultivated pineapple leaf fibres for industrial applications. *Journal of Polymers and the Environment*, 20(2), 404–411. doi:10.1007/s10924-011-0380-7
- Wu, Y., Wu, J., Yang, F., Tang, C., & Huang, Q. (2019). Effect of H₂O₂ bleaching treatment on the properties of finished transparent wood. *Polymers*, 11(5), 1–13. doi:10.3390/polym11050776