Science World Journal Vol. 15(No 2) 2020
www.scienceworldjournal.org
ISSN 1597-6343
Published by Faculty of Science, Kaduna State University

ISOLATION AND CHARACTERIZATION OF NANOCELLULOSE FROM PINEAPPLE LEAF FIBRES VIA CHEMO-MECHANICAL METHOD

Samuel Wadzani Gadzama [1], O.K. Sunmonu [2], U.S. Isiaku[2], and Abdullahi Danladi[2]

[1]Scientific Equipment Development Institute (SEDI-Enugu), National Agency for Science & Engineering Infrastructure, Nigeria.
[2]Department of Polymer & Textile Engineering, Ahmadu Bello University, Zaria, Nigeria.

*Corresponding Author's Email Address: swadzani@yahoo.com

ABSTRACT
In this study, the extraction, isolation and characterization of nanocellulose from pineapple leaf fibres (PALF) were carried out. The chemical pretreatment used include bleaching, and acid hydrolysis processes aimed at removing lignin, hemicellulose, and extractive substances. This was followed by sonication and milling to produce nanocellulose. Morphological changes to the PALF due to treatment were investigated using scanning electron microscopy (SEM). Transmission electron microscopy (TEM) indicated that the nanofibrils were produced and the micrographs showed PALF nanofibrils in the average range of 25–58 nm in diameters. Fourier transform infrared (FTIR) spectroscopy showed that there was no significant chemical / structural change after the phase changes. Energy Dispersive X-ray (EDX) shows no significant elemental composition difference in the nano celluloses obtained. The results indicated that PALF waste could become a viable source of commercially valuable nanocellulose.

Keywords: nanocellulose, pineapple leaf fibres, characterization, sonicate

1.0. INTRODUCTION

Natural fibres are made of lignin, cellulose, and hemicelluloses. Lignin and hemicelluloses are amorphous in structure while cellulose is semi crystalline and thus, differs in its physical and mechanical properties (Dos Santos *et al*., 2013). Cellulose is used to manufacture textiles and paper. It is a long linear glucose polymer linked by β-1,4 glycosidic bonds (Abraham *et al*., 2011). Cellulose has relatively high strength, high stiffness, low density and good thermal stability (Hossain *et al*., 2014). Therefore, it is a promising candidate for reinforcing biocomposites.

To obtain pure cellulose, the raw material is treated with alkali and bleached (Hossain *et al*., 2014; Moon *et al*., 2011). This chemical treatment breaks intermolecular and intramolecular hydrogen bonding between the hydroxyl groups of cellulose and hemicellulose and can increase the hydrophilicity of fibres (Sari *et al*., 2017; Asrofi *et al*., 2018a)). However, alkali treatment and bleaching do not significantly increase the crystallinity of cellulose fibre. Some studies suggest that subsequent acid hydrolysis can increase crystallinity and reduce the diameter of fibres (Chen *et al*., 2011; Cherian *et al*., 2010).

Several abundantly available renewable natural fibre sources which contain cellulose have been investigated. These include water hyacinth (Asrofi *et al*., 2017; Abral *et al*., 2018b), oil palm empty fruit bunch (Asrofi *et al*., 2018b), banana (pseudo stem), jute (stem) and pineapple leaf fibre (Abral *et al*., 2018), arecanut husk fibre (Candra *et al.* 2016), kenaf bast (Hibiscus cannabinus) (Jonoobi *et al*., 2009), coconut husk (Fahma *et al*., 2011), Helicteres isora plant (Chirayil *et al.,* 2014) and bacterial cellulose (Abral and Mahardika, 2016; Qiu and Natravali, 2017; Abral *et al*., 2018a) .

In 2016, the food and agricultural organization (FAO) in her report on Pineapple cultivation, indicated that Nigeria is the 7th largest world cultivator of pineapple plants, with about 0.9 million tons of fruit harvested annually (FAOSTAT, 2016). Old leaves must also be removed regularly. The weight of these leaves is about 7 % that of the fruit. Hence, in Nigeria, around 63,000 tons of fibre-rich pineapple leaves are removed from the plants each year. One possible way to increase the profitability of pineapple by-products would be to use these discarded leaves commercially to produce nanofibre for biocomposite reinforcement.

PALF has a unique combination of properties that make it an ideal source for nanofibre production such as has high strength, stiffness and low density (Nuryati *et al*., 2015). Its cellulose content is higher than that of oil palm empty fruit fibre (Sheltami *et al*., 2012) water hyacinth and mengkuang (screw pine) leaves (Nogi *et al*., 2009). Cherian *et a*l., 2010 reported that PALF has 81.27 % cellulose, 12.31 % hemicellulose, 3.46 % lignin and 10.52 % moisture content. However, these values will depend on the variety and environmental factors where the plant is grown.

Nanofibres of cellulose are more transparent and thermally stable than microfibres (Karimi *et al.,* 2014: Dufresne, 2017) They can be used to manufacture a range of products including flexible electronic film, coating for packaging, receptacles for drug delivery and optical digital storage media (Candra *et al*., 2016; Johar *et al*., 2012). Various nanocellulose extraction methods have been explored; steam explosion along with mild chemical treatment (Sari *et al*., 2017), chemical-ultrasonic process (Fahma *et al*., 2010), steam explosion alone (Cherian *et al*., 2010; Chirayil *et al*., 2014) a chemo-mechanical process using acid hydrolysis and homogenization (Candra *et al*., 2016) wet blending (Abral *et al*., 2018c), sulphuric acid hydrolysis (Dos Santos *et al*., 2013; Fahma *et al*., 2010; Liew *et al*., 2016; Yang and Van de Ven, 2016), a mechanical technique involving refining, cryo-crushing, and high pressure homogenization (Jonoobi *et al*., 2009), high pressure homogenization alone (Sheikhi *et al*., 2016), and high-shear homogenization (Tejado *et al*., 2018).

Liu *et al,* 2011, did a study on banana fibre nanocellulose which gave them nanocellulose with average diameter of < 100 nm, Teixera *et al*., 2010, studied the nanocellulose from sugarcane bagasse and obtained nanocellulose with average diameter of 255 nm, Chen *et al*., 2011, worked on potato peel fibre and isolated nanocellulose with average diameter of 411nm, Yu *et al*, 2012, worked on isolation of nanocellulose from bamboo and got an average diameter of 200 - 500 nm. Santos *et al.,* 2013, isolated nanocellulose from PALF and got average cellulose diameter of 210-240 nm. Zhang *et al*., 2015, worked on softwood and got values average of 100 nm, Nurrudin *et al.,* 2016, worked on kenaf and wheat straw and isolated nanocellulose within the diameter range of 100 nm. Lastly, Phanthang *et al*., 2018 worked on cellulose powder and were able to isolate nanocellulose within the value range of 10-25nm, Mahardika *et al*., 2018, studied the production of nanocellulose from PALF via high shear homogenization and ultrasonication, and were able to obtain nanocellulose with an average diameter of 68 nm and length between 88-1100 nm

It has been shown that high-shear homogenization is a more environmentally friendly method for production of nanocellulose than chemical treatment and results in higher crystallinity, thermal stability, and transparency. Moreover, nanocellulose production via ultrasonication is a very effective way to depolymerize cellulose as reported by earlier researchers. The method used in this study is new and uses chemical treatment, sonication and milling. This has several advantages compared to other existing methods of PALF used in studies by other researchers as it requires only two repeats of the bleaching, acid hydrolysis, sonication and milling. This, in turn, minimized the use of expensive and potentially environmentally damaging chemicals.

The characteristics of PALF before and after treatments were observed by using Fourier Transform Infrared (FTIR), Scanning Electron Microscopy (SEM), and Transmission Electron Microscopy (TEM), and Energy Dispersive X-ray (EDX).

2.0. MATERIALS AND METHODS

2.1. Materials

The waste pineapples leaves used for this research were harvested from a private pineapple plantation called San Carlos, in Ihe village, Agwu Local Government Area of Enugu State, Nigeria during the harvest period. Awgu is located approximately between latitudes 06 00' and 06 19' North of the Equator and longitudes 07 23' and 07 35' East of the Greenwich Meridian (Nwankwo, 2014)

2.2. The Fibre Extraction

The fibres were extracted mechanically by scrapping the thin outer layers of the spiky semi-dried leaves via the use of sharp broken tile edge.

The fibres extracted were then cleaned, by washing the fibre thoroughly in 2 % detergent solution at 70 ^{0}C and then rinsed with very clean water. Basically, this procedure removed most of the foreign objects and impurities that could contaminate the fibre as a result of the extraction process.

Figure 1. The PALF after extraction

2.3. Pineapple Leaf Fibre Characterization

2.3.1. Chemicals, reagents, and test samples

All chemicals and reagents used in this study were of analytical grade and commercially available. The raw biomass material was the pineapple leaf fibre extracted.

2.3.2. Preparation of Test Samples

The pineapple leaf fibres were chopped to particle sizes of 2 - 6 mm, milled and then stored in clean bags prior to compositional analysis

2.3.3. Compositional Analysis of the Pineapple Leaf Fibre (PALF)

The pineapple leaf fibre was subjected to compositional analysis using the gravimetric method. Chemical composition of PALF was measured as follows: extractives (Tappi Method T204 cm - 97, 1988; Tappi Method T207 cm-88, 1988), acid insoluble lignin (ASTM E1758 - 01, 2015), cellulose, hemicellulose (ASTM 1695-77, 2001) and ashes (UNE 57050:2003, 2003)., based on the procedure reported by Natalie *et al*., 2016.

2.4. Production /Isolation of Nano Particles from the PALF

2.4.1. Procedure

About 100 g of PALF was added into 2 % w/v NaOH solution and digested at 80 ^{0}C for 3 hours using a thermostated hot plate. This removes lignin in the form of soluble complexes. The samples were then washed severally with distilled water and filtered using sieve number 40 mm mesh size. The samples were bleached with 10 % aqueous dilution of sodium hypochlorite for 30 minutes at 100 ^{0}C, washed and then filtered again. The samples were further treated with 17.5 % w/v sodium hydroxide at 80 ^{0}C for 1 hour and the resulting samples were washed thoroughly with water and subjected to bleaching process with 10 % sodium hypochlorite for 15 minutes at 80 ^{0}C. Finally, they were washed with water until neutral. The dried samples obtained were milled until very fine particles were obtained

2.5. Characterization of the Nano Cellulose PALF

The characterization was carried out at the National Center for Nanostructured Materials (NCNSM), Council for Scientific and Industrial Research (CSIR), Pretoria, South Africa

2.5.1. Equipment, Reagents, Chemicals

Emitech K950X Carbon Evaporator (for carbon and gold coater) was used for preparation of the samples while the morphological characterization of the PALF was carried out by using the JEOL JSM 7500F Scanning Electron Microscope (SEM). The characterization of the nano particles of the PALF was carried out by using the JEOL JEM 2100 High Resolution Transmission Electron Microscope (HRTEM).

2.6. Fourier Transform Infrared Spectroscopy of the Pineapple Leaf Fibre (PALF)

Buck Scientific M530 USA FTIR was used for the analysis. This instrument was equipped with a detector of deuterated triglycine sulphate and beam splitter of potassium bromide. The software of the Gram A1 was used to obtain the spectra and to manipulate them. Approximately 1.0 g weight of samples respectively was properly placed on a KBr pellet. During measurement, FTIR spectra was obtained at frequency regions of 4,000 – 600 cm^{-1} and co-added at 32 scans and at 4 cm^{-1} resolution. FTIR spectra were displayed as transmitter values

3.0. RESULTS AND DISCUSSIONS

From literature, the bleaching approach on the process of achieving micro and subsequently nanocellulose reduces the amount of lignin and hemicellulose in natural fibre cellulose. Similar trend was discovered in this research as the same test that was conducted to verify the percentage composition of the presence of main constituents of the PALF namely, Cellulose and lignin and hemicellulose was repeated again after isolating for micro and then nanocellulose. This is so due to the removal of the amorphous content of the PALF which increased the crystalline fraction. Increase in the proportion of crystalline cellulose leads to increases in thermal stability as reported by Mahardika *et al*., 2018.

3.1. Characterization of the PALF

Table 1 shows the compositional results of the PALF samples with row 2 showing results obtained when the PALF samples were at the macro cellulose dimension and row 3 showing results obtained when the PALF samples were reduced to nano cellulose dimensions.

Table 1: Compositions of the PALF used in this research work

PALF Samples Dimensions	Cellulose	Lignin	Hemicellulose
Macro Cellulose PALF	70.87	5.34	13.92
Nano Cellulsoe PALF	72.20	3.10	8.83

3.2. Elemental Analysis of the PALF

This aspect of the research was done in two part, as shown in table 2, row 2 shows results obtained from conventional approach. The second part was done using a more accurate method, this was achieved by the use of EDX at the Council for Scientific and Industrial Research (CSIR), Pretoria, South Africa. The results are as shown in row 3 of Table 2.

Table 2: Elemental analysis of the PALF used in this research work

Analytical method	C	O	Ca	Na	Fe	K	Cu	Al	Cl	N
TAPPI method	71.2	26.4	2.2	0.34	0.7	0.94	0.02			
EDX analysis	71.04	24.85	0.10	0.34	-	-	-	0.09	0.43	3.05

3.3. Fourier Transform Infrared Spectroscopy of the Pineapple Leaf Fibre (PALF)

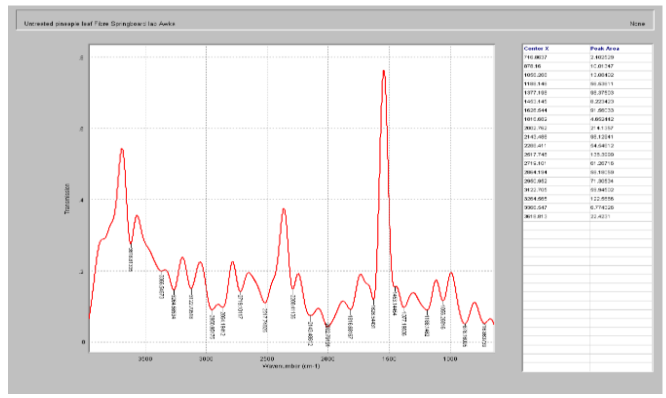

Figure 2: The FTIR spectrum of the PALF

Fourier Transform Spectroscopy (FTIR) is used to detect characteristic chemical functional groups in fibre. Figure 2 which illustrates the FTIR spectrum of untreated PALF sample has a total of 19 well defined peaks as observed corresponding to the stretching vibration mode of intra- and intermolecular hydroxyl (–OH) bond of cellulose which is in agreement with what was obtained by Prasanna *et al.,* 2017 and the peak 2950.85 cm^{-1} corresponding to the asymmetric and the symmetric stretching of methylene ($-CH_2-$) groups in long alkyl chains. These peaks prove the presence of waxes in the PALF as reported by Abidi *et al*., 2018.

3.4. Isolation/Production of Nanofibrils PALF

3.4.1. Scanning Electron Microscopy (SEM) of the PALF samples

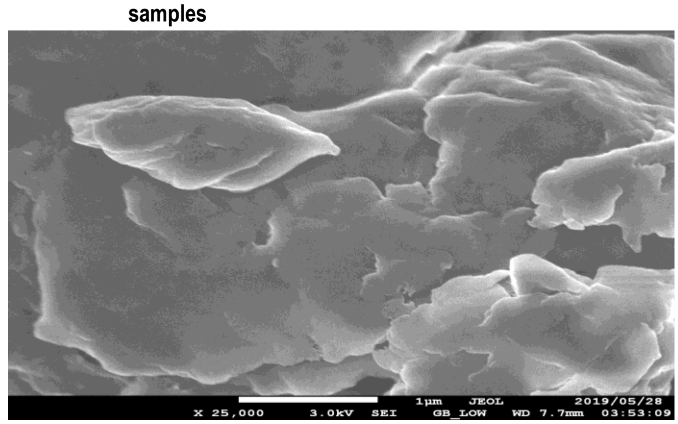

Figure 3: SEM of the PALF

The PALF at the nano cellulose level is considered amorphous in nature. The micrographs of the SEM of the PALF nano cellulose depicting the already established fact that untreated natural fibres are hydrophobic in nature. Figure 3 shows the SEM micrograph of the untreated PALF micro cellulose at 1µm voltage of 3.0 kv and a magnification of x10,000.

3.4.2. High Resolution Transmission Electron Microscopy (HR-TEM) of the PALF Nano Cellulose

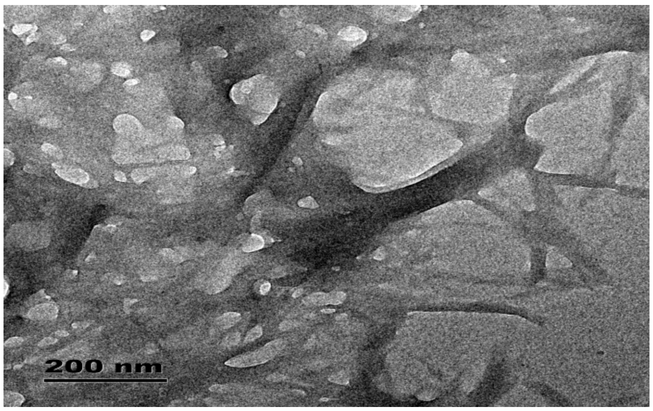

Figure 4. TEM of the untreated PALF nanocellulose agglomerate at 200 nm

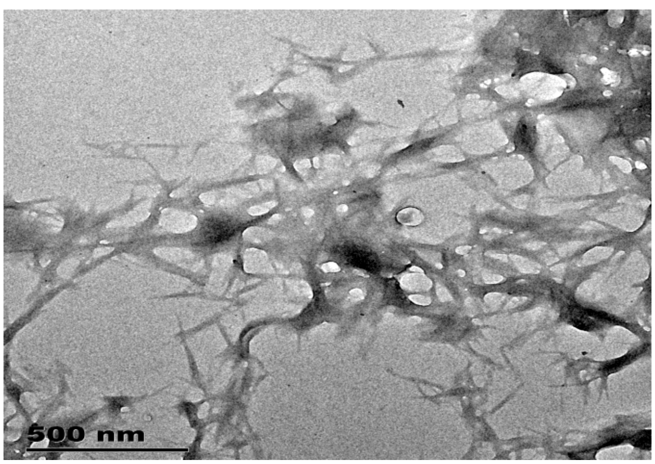

Figure 5. TEM of the PALF nanocellulose agglomerate at higher magnification of 500 nm

The dispersion of some of the nano cellulose samples is a result of ultrasonication which assist in preventing agglomeration. Agglomerations are as a result of the Van der Waals attractive forces between the nano particles, it is imperative to put on record that some of the agglomerates are in bundle forms while some are well dispersed. The above agglomeration of nano particles is reported as a common occurrence when working on natural fibres. (Goh *et al*., 2016; Chen *et al*., 2011)

The results of this research fall with the acceptable limits of nanocellulose particles as reported other researchers.

4.0. Conclusions

PALF nanocellulose has been successfully fabricated using a new method. A combination of chemo-mechanical method highly reduced the need for extensive chemical pretreatment. Single stage pre-bleaching and acid hydrolysis were sufficient to produce nano-cellulose with a high degree of purity. This treatment is more environmentally friendly than previous methods using repeated bleaching. Milling of the PALF cellulose was sufficient to produce high purity nanocellulose fibres with an average of 25-68 nm diameters and length less than 120–1700 nm. EDX analysis and FTIR testing of PALF indicates that most lignin, hemicelluloses, and extractive substances have been eliminated and the functional groups in the PALF cellulose were not significantly altered.

REFERENCES

Abraham, E, Deepa, B, Pothan, L, A.,Jacob, M, Thomas, S, Cvelbar, U and Anandjiwala, R. (2011). Extraction of nanocellulose fibrils from lignocellulosic fibres: A novel approach. *Journal of Carbohydrate Polymer* 86, 1468–1475.

Abral, H, Putra, G.J, Asrofi, M, Park, J.W and Kim, H.J. (2018). Effect of vibration duration of high ultrasound applied to bio-composite while gelatinized on its properties. *Ultrasonic Sonochemical* 40, 697–702.

Abral, H, Dalimunthe, M.H, Hartono, J. Efendi, R.P. Asrofi, M, Sugiarti, E. Sapuan, S.M, Park, J.W and Kim, H.J. (2018). Characterization of tapioca starch biopolymer composites reinforced with micro scale water hyacinth fibres. *Tapioca Starch Biopolymers. 2018, 70, 1700287. DOI: 10.1002/star.201700287*

Abral, H. and Mahardika, M. (2016). Tensile properties of bacterial cellulose nanofibres-polyester composites. IOP *Conf.erence Ser.ies for Material Science and Eng*ineering. Vol., 137, 12019

Abidi N, Hequet E, Cabrales L, Gannaway J, Wilkins T, Wells L.W. (2008). Evaluating cell wall structure and composition of developing cotton fibers using Fourier transform infrared spectroscopy and thermogravimetric analysis. *Journal of Applied Polymer Science*. 107:476- 486.

Asrofi, M, Abral, H, Kasim, and Pratoto A. (2017). A. XRD and FTIR studies of nanocrystalline cellulose from water hyacinth (Eichorniacrassipes) fibre. *Journal of Metastabilized Nanocrystalline Materilals.* Vol. 29, 9–16.

Abral, H, Lawrensius, V, Handayani, D and Sugiarti, E. (2018).Preparation of nano-sized particles from bacterial cellulose using ultrasonication and their characterization. *Carbohydrate Polymers.* 191, 161–167.

Asrofi, M, Abral, H, Kasim, and Pratoto A. (2018). A. Characterization of the microfibrillated cellulose from water hyacinth pulp after alkali treatment and wet blending. *IOP Conference Series of Materila Science and Engineering.* 204, 12018.

Asrofi, M, Abral, H, Putra, Y.K, Sapuan, S.M and Kim, H.J. (2018). Effect of duration of sonication during gelatinization on properties of tapioca starch water hyacinth fibre biocomposite. *International Journal of Biological Macromolecules* Vol. 108, 167–176.

Candra, C.S.J, George, N and Narayanankutty, S.K. (2016). Isolation and characterization of cellulose nanofibrils from arecanut husk fibre. *Carbohydr.ate Polym*ers. 142, 158–166.

Chen, W., Yu, H., Liu, Y.; Hai, Y., Zhang, M and Chen, P. (2011). Isolation and characterization of cellulose nanofibres from four plant cellulose fibres using a chemical-ultrasonic process. *Cellulose*, 18, 433–442.

Cherian, B.M., Leão, A.L, de Souza, S.F, Thomas, S and Pothan, L.A. (2010). Kottaisamy, M. Isolation of nanocellulose from pineapple leaf fibres by steam explosion. *Carbohydrate Polym*ers. 81, 720–725.

Chirayil, C.J., Joy, J., Mathew, L., Mozetic, M., Koetz, J and Thomas, S.(2014). Isolation and characterization of cellulose nanofibrils from Helicteres isora plant. *Journal of Industrial Crops and Products.* 59, 27–34.

Dos Santos, R.M.,Neto, W.P.F., Silvério, H.A., Martins, D.F., Dantas, N.O and Pasquini, D. (2013). Cellulose nanocrystals from pineapple leaf, a new approach for the reuse of this agro-waste. *Journal of Industrial Crop s and Products.* 50, 707–714.

Dufresne, A. (2017). Cellulose nanomaterial reinforced polymer nanocomposites. Current Opinion. *Colloid Interface Sciences.* 29, 1–8.

Fahma, F, Iwamoto, S, Hori, N, Iwata, T and Takemura, A. (2010). Isolation, preparation, and characterization of nanofibres from oil palm empty-fruit-bunch (OPEFB). *Cellulose*, 17, 977–985.

Fahma, F., Iwamoto, S., Hori, N., Iwata, T. and Takemura, A. (2011). Effect of pre-acid-hydrolysis treatment on morphology and properties of cellulose nanowhiskers from coconut husk. *Cellulose*, 18, 443–450.

FAOSTAT (2016). Available:http;/ www. fao. Org/. 6/12/2016. retrived on 6th July 2019.

Goh, K.Y., Ching, Y.C, Chuah, C.H., Abdullah, C.L. and Liou, N.-S.(2016). Individualization of microfibrillated celluloses from oil palm empty fruit bunch: Comparative studies between acid hydrolysis and ammonium persulfate oxidation. *Cellulose* 23, 379–390.

Hossain, M.K, Karim, M.R, Chowdhury, M.R, Imam, M.A, Hosur, M, JeelAppani, S and Farag, R. (2014). Comparative mechanical and thermal study of chemically treated and untreated single sugarcane fibre bundle. *Journal of Industrial Crops and Products.* 58, 78–90.

Johar, N, Ahmad, I and Dufresne, A. (2012). Extraction, preparation and characterization of cellulose fibres and nanocrystals from rice husk. *Journal of Industrial Crops and Products.* , 37, 93–99.

Jonoobi, M, Harun, J, Mishra, M and Oksman, K. (2009). Chemical composition, crystallinity and thermal degradation of bleached and unbleached kenaf bast (Hibiscus cannabinus) pulp and nanofibre. *BioResources* , 4, 626–639.

Karimi, S., Tahir, P.M.,Karimi, A., Dufresne, A and Abdulkhani, A. (2014). Kenaf bast cellulosic fibres hierarchy: A comprehensive approach from micro to nano. *Carbohydr.ate Polymers.* 101, 878–885.

Liew, S.Y., Thielemans, W., Hewakandamby, B. (2016). Separation of sulphuric acid from an acid suspension of cellulose nanocrystals by manual shaking. *J. Nano Res.* 38, 58–72.

Liu K, Tian Y and Liang L (2013). Bio-inspired superoleophobic and smart materials: Design, fabrication, and application. *Progress in Materials Science*. Volume 58, Issue 4, May 2013, Pages 503-564. https://doi.org/10.1016/j.pmatsci.2012.11.001

Liu, Y.; Hai, Y., Chen, W., Yu, H., Zhang, M. and Chen, P. (2011). Isolation and characterization of cellulose nanofibres from four plant cellulose fibres using a chemical-ultrasonic process. *Cellulose*, 18, 433–442.

Mahardika Melbi , Hairul Abral, Anwar Kasim, Syukri Arief and Mochamad Asrofi. (2018). Production of Nanocellulose from Pineapple Leaf Fibers via High-Shear Homogenization and Ultrasonicatio. retrived from www.mdpi.com/journal/fibers on 6th July, 2019. *Fibers* 6, 28; doi:10.3390/fib6020028

Moon, R.J., Martini, A., Nairn, J. Simonsen, J. and Youngblood, J. (2011). Cellulose nanomaterials review: Structure, properties and nanocomposites. *Chemical Society Rev*iews. 40, 3941–3994.

Nathalie B, Bopha Y and Lefebvre R (2016) From organic pollutants to bioplastics: insights into the bioremediation of aromatic compounds by Cupriavidus necator. *New Biotechnol* 32:1871–6784. doi:10.1016/j.nbt.2014.09.003

Nogi, M., Iwamoto, S., Nakagaito, A.N. and Yano, H. (2009). Optically transparent nanofibre paper. *Advanced Mater.ials.* 21, 1595–1598.

Nuruddin, M.D. Mahesh Hosur, M.D, Jamal Uddin, David Baah and Shaik Jeelan. (2016). A novel approach for extracting cellulose nanofibers from lignocellulosic biomass by ball milling combined with chemical treatment. *Journal of Applied Polymer Sciences,* DOI: 10.1002/APP.42990

Nuryati, L., Noviati, Astrid Susanti, A. (2015). *Outlook Komoditas Pertanian Subsektor Hortkultura Nenas*; Pusat Data dan Sistem Informasi Pertanian; Portal Epublikasi Pertanian: Jakarta, Indonesia, pp. 1507–1907.

Nwankwo CF (2014). Residential Water Demand and Supply in Awgu Local Government Area, Enugu State, Nigeria. Department of Geography, University of Nigeria: Unpublished B Sc Project.

Phanthong Patchiy A, Prasert Reubroycharoen C, Xiaogang Hao dd Guangwen Xue, EAbuliti Abudula A and Guoqing Guan. (2018). Nanocellulose: Extraction and application. *Carbon Resources Conversion* Vol. 1, 32–43.

Prasanna SC, Ramesh C, Manivel R and Karthe M (2017)..An Alternative Method . for Synthetic Fibers and categorization of Natural Fiber Extracted from Sesbania rostrata, *Journal of Chemical and Pharmaceutical Sciences.* ISSN: 0974-2115. 2:203-207.

Qiu, K and Netravali, A. (2017). In Situ Produced Bacterial Cellulose Nanofibre-Based *Hybrids for Nanocomposites Fibres*, 5, 31.

Sari, N.H., Wardana, I.N.G., Irawan, Y.S. and Siswanto, E. (2017). The Effect of Sodium Hydroxide on Chemical and Mechanical Properties of Corn Husk Fibre. *Oriental Journal of Chemistry* 33, 3037–3042.

Sheikhi A, H. Yang, Alam M and Van de Ven T. (2016). Highly stable, functional hairy nanoparticles and biopolymers from wood fibers: towards sustainable nanotechnology, *Journal of Visualized Exp*eriments. 113 e54133.

Sheltami, R.M., Abdullah, I., Ahmad, I., Dufresne, A. and Kargarzadeh, H.(2012). Extraction of cellulose nanocrystals from mengkuang leaves (Pandanus tectorius). *Carbohydrate Polym*ers. 88, 772–779.

Siqueira G, Bras J , A. Dufresne (2010). Cellulosic bionanocomposites: a review of preparation, properties and applications, *Polymers.* 2 , 728–765.

Tejado A, Alam M, Antal M, Yang H and Van de Ven T. (2012). Energy requirements for the disintegration of cellulose fibers into cellulose nanofibers, *Cellulose* 19, 831– 842.

Teixeira, E. M., Corrêa, A. C., Manzoli, A., Leite, F. L., Oliveira, C. R., & Mattoso, L. H. C. (2010). Cellulose nanofibers from white and naturally colored cotton fibers. *Cellulose,* 17, 595–606.

Yang H, Van de Ven, T. (2016). Preparation of hairy cationic nanocrystalline cellulose, *Cellulose.* 23 , 791–1801.

Yu, Mengjiao, Yang, Rendang, Huang, Lianghui, Cao, Xiaodong, Liu, Detao. (2012). Preparation and characterization of bamboo nanocrystalline cellulose. *BioResources.* Vol., 7.2.1802-1812. doi:10.15376/biores.7.2.1802-1812

Zhang, L., Tsuzuki, T., and Wang, X., (2015). Preparation of cellulose nanofiber from softwood pulp by ball milling,. *Cellulose* 22(3):1729-1741.