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EXTRACTION OF NANOFIBRILLATED CELLULOSE FROM KELEMPAYAN (*NEOLAMARCKIA CADAMBA*) AND ITS USE AS STRENGTH ADDITIVE IN PAPERMAKING

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Nanofibrillated cellulose (NFC) is a cellulose at nanoscale dimension that can offer multitude applications in various industrial sectors such as pulp and paper, composite, cosmetic, pharmaceutical and others. In this study, NFC was prepared from bleached kelempayan (*Neolamarckia cadamba*) pulp. The resulting NFC was added into laboratory handsheets (60 g m^{-2}) at 2, 4, 6, 8 and 10% of addition level. The results showed that incorporation of NFC in papermaking enhanced the strength in tensile, burst and fold with increasing dosage but exhibited reduction in tear and air permeability as more NFC was added.

Keywords: Nanocellulose, kelempayan, papermaking, additive

INTRODUCTION

Cellulose has been used in many traditional and modern applications due to its availability worldwide, estimated in excess of 7.5×10^{10} tonnes (Habibi 2010). This fascinating biopolymer has received enormous attention in the last century due to its increased need for environmental/eco-friendly and sustainable materials in the market. Nanocellulose is known as cellulose with at least one dimension in nanometer and has a combined feature of cellulose and nanomaterial (Klemm et al. 2018). The cellulosic nanomaterials pave the way for many groundbreaking applications in various fields such as biomedical, engineering and environment. In fact, nanocellulose from various resources has been studied for multitude applications in response to the increased demand for such material in biobased economy (Abdul-Khalil et al. 2014).

Cellulose exists as microfibrils in plant cell wall. Depending on the isolation process, the fibrils that contain strong crystalline and amorphous region can be extracted to produce nanofibrillated cellulose (NFC) or nanocrystalline cellulose (NCC) (Salas et al. 2014). The NCC is most commonly obtained by acid hydrolysis from various natural resources using mineral acids such as sulfuric acid, hydrochloric acid or phosphoric acid. The NCC is obtained when acid preferentially hydrolyses less dense amorphous regions, leaving behind the denser crystalline particles. The NFC refers to cellulose fibres that are subjected to fibrillation or delamination of the cell wall via intensive mechanical shearing action to release cellulose fibrils in the form of elementary fibril bundles. The NFC has nanoscale (less than 100 nm) diameter and typical length of several micrometers (Missoum et al. 2013).

The advantages of NFC are as follows: availability as renewable material, high mechanical strength, high surface area, high aspect ratio, good barrier property, biodegradability and biocompatibility (Eichorn et al. 2010, Brodin et al. 2014). The NFC consists of alternating crystalline and amorphous domains which allows flexibility as opposed to NCC that has limited flexibility (Brinchi et al. 2013). High strength and stiffness of NFC as well as its low thermal expansion and optical transparency can be used as reinforcing component in various biopolymer matrices (Zimmermann et al. 2010). Besides that, NFC can be used in many other applications that include coating, food, pharmaceutical and most importantly papermaking industry.

The NFC is typically prepared from pulp suspension via various mechanics such as high-pressure homogeniser, refiners, ball milling, etc. (Klemm 2018). The energy consumption in producing NFC is high and varies from one method/process to another. In order to minimise the high energy consumption some pretreatments are conducted prior to fibrillation such as TEMPO-mediated oxidation, carboxymethylation and enzymatic treatment. Energy consumption has been shown to reduce around 1000 kWh ton^{-1} using these pre-treatments (Siró & Plackett 2010).

Wood is usually used as feedstock for NFC production. Kelempayan, or scientifically known as *Neolamarckia cadamba,* is a fast growing tropical timber species. It is a large, deciduous and fast growing tree species from the Rubiaceae family that can give early economic return within 8–10 years (Lai et al. 2013). Kelempayan trees are able to reach 45 m in height and a diameter of 100–160 cm. This fast growing tree has remarkable economic value for furniture manufacturing, pulp and paper, woody forage and pharmaceutical industry (Lal et al. 2010, Zayed et al 2014). In some countries, it has also been used as landscape tree for urban forest project.

The NFC as a paper additive has been studied by many researchers (Boufi et al. 2016, Kajanto & Kosonen 2012). The NFC may be added directly into the pulp with or without retention aid, or premixed with furnish component such as the filler or long fiber fraction and deposited on the surface of this furnish component by retention aids (Brodin et al. 2014). Although various lignocellulosic materials have been used as additive to improve the strength properties of paper, such as eucalyptus (Gonzalez 2012) and bagasse (Afra et al. 2013), the use of tropical hardwood NFC as an additive in pulp and paper field is limited. Therefore the objective of this study is to prepare nanofibrillated cellulose from kelempayan and use it as a strength additive in papermaking.

MATERIALS AND METHODS

Nine-year-old kelempayan trees were obtained from a thinning exercise in a plantation site in Sentul Forest Reserve, Negeri Sembilan, Malaysia.

Fibre morphology

Wood fibres were prepared by digesting the wood splints through successive addition of acetic acid and sodium chlorite over a water bath. Wood splints were obtained from a disc from the billet at breast height. The softened wood splints were shaken up with glass beads in a bottle. The fibres were placed on glass slides and the length and width and wall thickness are determined by measuring the projected image over a stereomicroscope. The fibres were stained with safranin-o for better clarity. The fibre measurement was carried out for at least 50 times (T 232 cm-06).

Chemical composition

Wood disc was cut from kelempayan tree at breast height. The disc was debarked, cut into matchsticks and ground into fine size by Wiley mill. Wood samples for chemical analysis were ground to pass a 40 mesh sieve and retained on a 60 mesh sieve. The samples were then air dried for at least one day. The chemical analysis were carried out according to the following standard procedures: moisture content (T 264 cm-97), hot water soluble (T 207 cm-99), 1% alkali soluble (T 212 om-02), alcohol toluene soluble (T 204 cm-97), ash content (T 211 om-02), alpha-cellulose content (T 203 cm-99), lignin content (T 222 om-02), pentosan (Savard et al. 1954) and holocellulose content (Wise et al. 1946). The analysis was carried out in duplicates.

Kraft pulping

Kraft pulping was carried out on kelempayan at 16% active alkali and 25% sulfidity. The ratio of wood to liquor ratio was maintained at 1:6. The pulping was conducted using a rotary digester for 3½ hours at 170 °C. The resulting pulp was screened using Somerville fractionator to remove impurities, debris and uncooked fibres. Kappa number was determined for unbleached pulp only.

Bleaching process

The pulp obtained in previous step was bleached via 5-stage-elemental chlorine-free bleaching process i.e DEDED. The D refers to chlorine dioxide and E is extraction by sodium hydroxide. At this stage, the bleached pulp was used as a feedstock for nanocellulose production.

Pre-treatment with TEMPO (2,2,6,6-tetramethylpiperidin-1-oxyl)

Bleached pulp was first pre-treated via TEMPO-oxidation method to facilitate subsequent fibrillation process. To a bleached pulp suspension (0.5 wt%, 1000 ml), 297.1 mg of TEMPO and 3.3 g of NaBr were added. The mixture was well-stirred and 36 ml of NaClO was added. The suspension was maintained at pH 10 by addition of diluted NaOH or HCl for 2 hrs. Ethanol was added to stop the reaction. The resulting pulp was vigorously washed with water to remove excess chemicals.

Preparation of Nanofibrillated cellulose (NFC)

The pre-treated pulp was beaten by Papirindustriens Forskningsinstitutt (PFI) Mill at 4000 revolutions followed by homogenisation at 10,000 rpm for two hours. The weight of the nanofibrillated suspension was calculated as 0.8%.

Atomic force microscopy

The NFC was examined under atomic force microscopy (AFM) to verify its nanoscale dimension. The NFC suspension at 0.01 wt% was dropped on freshly cleaved mica and observed under AFM via tapping mode.

Papermaking and testing

Laboratory handsheets (60 ± 3 g m^{-2}) were prepared according to method by Technical Association of the Pulp and Paper Industry (TAPPI) T205 sp-02 using kelempayan bleached pulp as feedstock. Nanofibrillated cellulose prepared previously was added at dosage of 2, 4, 6, 8 and 10% based on dry weight of paper, functioning as an additive. Nanofibrillated ccellulose was added during stock preparation at varying dosage. The handsheets incorporated with NFC was evaluated for their physical, optical and mechanical properties as shown in Table 1.

Table 1 Methods for paper testing

Type of test	Test method
Physical test	TAPPI T220 sp-01
Specific volume (bulk)	MS ISO 534: 2007
Brightness	MS ISO 2470-1:2010
Opacity	MS ISO 2471:2010
Tensile	MS ISO 1924-2:2010
Tear	MS ISO 1974:1999
Burst	MS ISO 2758:2007
Folding endurance	ISO 5626:1993
Air permeance	ISO 5636-3 1992

Table 2 Fibre morphology of kelempayan

Fibre morphology	Kelempayan
Fibre length, l (mm)	1.3 ± 0.13
Fibre width, d (µm)	38.0 ± 7.35
Fibre lumen, L (µm)	24.0 ± 6.48
Fibre wall thickness, w (µm)	14.0 ± 1.39
Felting power (l/d)	34.2

RESULTS AND DISCUSSION

Morphology of kelempayan

The average fibre length of kelempayan shown in Table 2 was similar to other light hardwood timbers such as medang and keledang (Peh et al. 1986). The fibre length was longer than acacia (0.8 mm) and eucalyptus (1.0 mm), both being used as common feedstock for pulp and paper industry in Asean region. Fibre width and lumen of kelempayan were close to the value reported by Peh et al. (1986) in which the values were 42.1 and 32.3 µm respectively. A closer look of lumen and width can be observed from Figure 1.

Chemical composition of kelempayan

Chemical properties of kelempayan is shown in Table 3. The chemical composition shows no reason to anticipate any difficulty in pulping by kraft process. The holocellulose content (70.9%) shows good indication on the pulp yield due to linear relationship on yield against holocellulose content. The holocellulose content is in the range of Malaysian tropical hardwood i.e between 59.4 to 85.4% (Khoo and

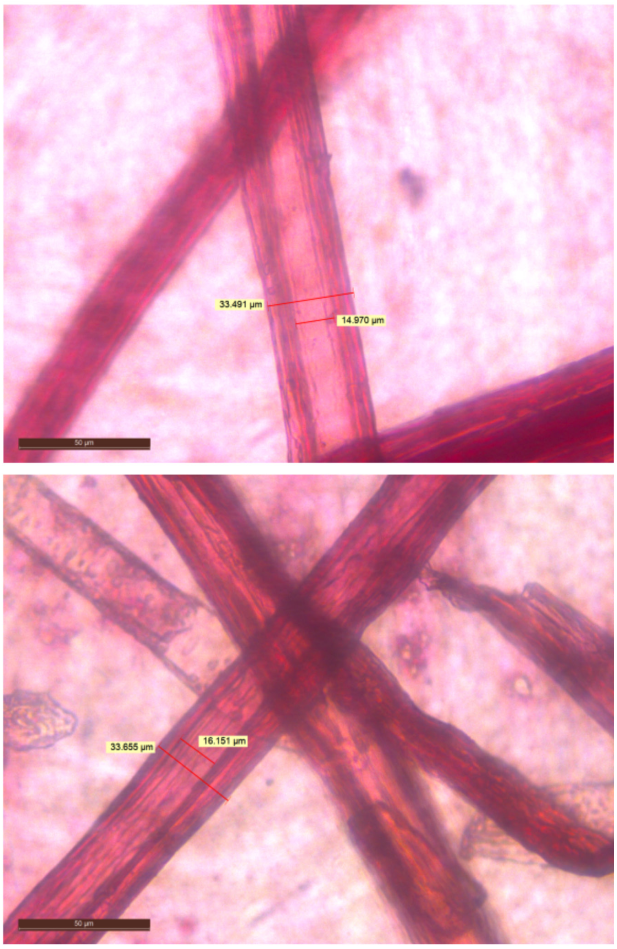

Figure 1 Microscopic image of kelempayan fibre at 4 x magnification

Table 3 Chemical components of kelempayan fibres

Chemical composition	%
Ethanol toluene solubility	3.5
Holocellulose	70.9
Alpha-cellulose	40.1
Acid-insoluble lignin	24.3
Alkali solubility	18.2
Hot water solubility	7.0
Ash	0.5
Silica	0.2
Pentosan	18.2

Peh 1982). High content of cellulose results in high possibility of bleached pulp production, that of which becomes the feedstock for NFC preparation. Lignin content (24.3%) is also in the range of our local hardwood values (12.7 to 34.2%) based on the works by Khoo and Peh (1982).

Morphology of nanofibrillated cellulose (NFC)

After pulping, kelempayan gave 45.6% pulp yield which can be considered as a good yield from pulping point of view. Subsequently, the unbleached pulp with 14.2 Kappa number was subjected to a 5-stage bleaching process and the bleached kelempayan pulp became the feedstock for conversion to NFC. The NFC from kelempayan was prepared through chemical pre-treatment first, i.e (2, 2, 6, 6, -Tetramethylpiperidin-l-yl) oxyl (TEMPO) mediated oxidation followed by fibrillation using PFI mill and homogeniser. The carboxyl groups introduced on the surface of the cellulose during pre-treatment facilitate the subsequent fibrillation process due to charge repulsion between the fibres (Dai et al 2013).

Figure 2 shows the morphology of NFC from kelempayan observed using AFM. It is interesting to note that NFC appeared as normal entangled fibre but with slimmer width. The NFC prepared using the technique in this study had width in the range of 5.14 to 34.74 nm with average width of 18.13 ± 5.3 nm (Figure 2). The NFC length cannot be determined in this study as it is difficult to obtain individual fibre but it is assumed that the length is in several micrometers. Upon successful conversion of kelempayan to nanocellulose, paper handsheets of 60 gsm (g m^{-2}) was produced with the addition of NFC at varying addition levels (2 to 10%). The improvement in respective properties i.e physical, mechanical and optical was compared with controlled handsheets (without NFC addition).

Effect of nanofibrillated cellulose (NFC) on apparent density

As expected, the paper became denser as NFC was gradually added as displayed in Figure 3. Density is affected by the amount of fibre bonding, void space and calendering. The NFC fills up the void in the fibre network during paper formation thus resulting in increased density of the paper. In addition, NFC also contributes to stronger network and bonding capability between fibres (Kajanto & Kosonen 2012).

Effect of nanofibrillated cellulose (NFC) on strength

Properties of paper is usually improved by addition of various additives, depending on the

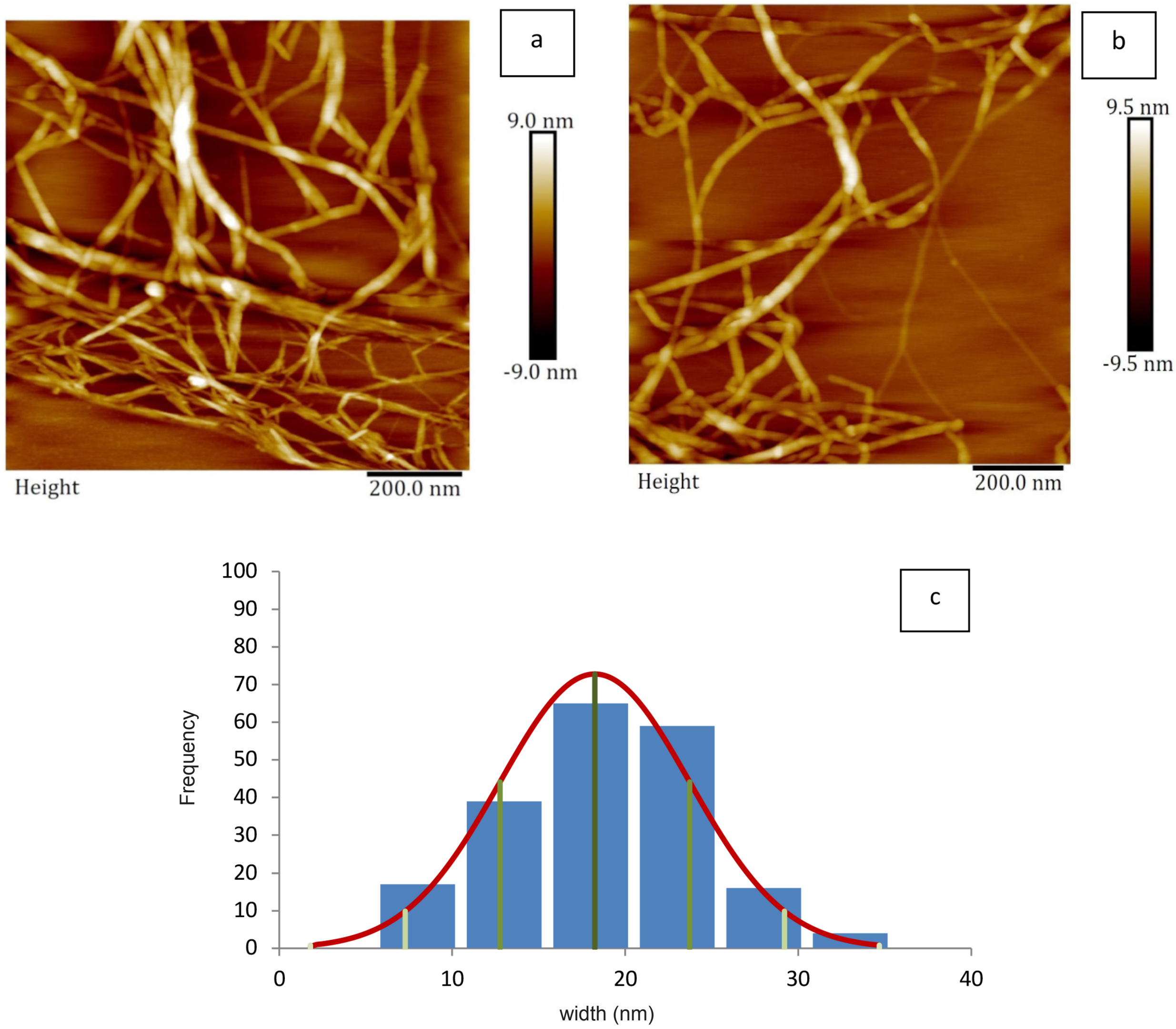

Figure 2 Atomic force microscopy (AFM) images of nanofibrillated cellulose (NFC) from kelempayan pulp (a) and (b), and width distribution of NFC (c)

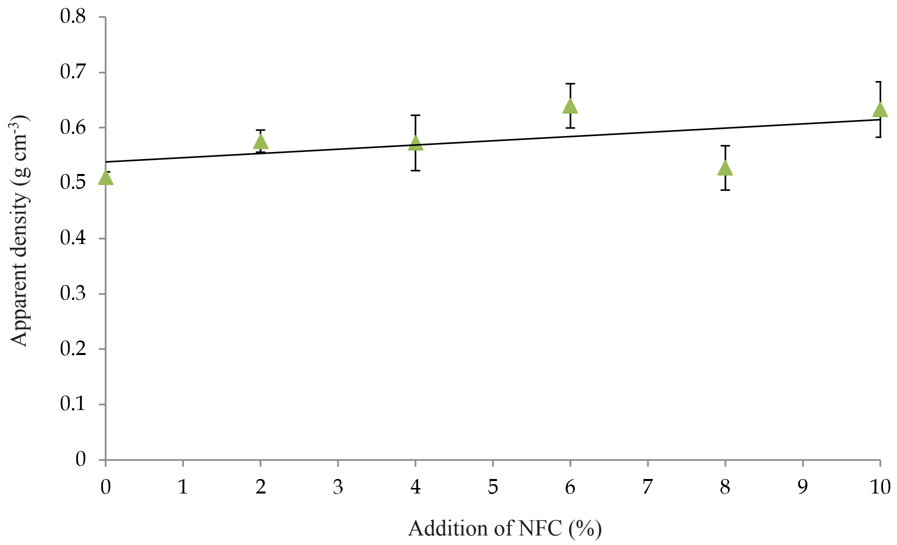

Figure 3 Effect of nanofibrillated cellulose (NFC) on apparent density of paper

end use. In this study, NFC, being a strength additive, was added to promote bonding between fibres, thus resulting in improvement of paper properties. Paper strength is largely influenced by such factors as fibre strength and length, specific bond strength and strength of bonded area, sheet formation and distribution of residual stresses (Boufi et al. 2016, Hubbe 2006, Taipale et al. 2010).

Tensile index

The effect of adding NFC at varying dosage on tensile strength is shown in Figure 4. As expected, incorporation of NFC enhanced the tensile strength with increasing dosage. The fiber network strength is improved by the mechanical entanglements of the fibrils (Hii et al. 2012). Adding 4% NFC increased tensile index to 19%. Paper reinforced with 10% NFC recorded an enhancement of up to 35%. The highest tensile strength was 61.5 Nm g^{-1} at 10% addition level. The positive effect on the strength was attributed to improved bonding contributed by NFC during paper formation by filling up the gap in the fiber network, thus increasing the strength property. In addition, the larger surface area per mass unit contributed by NFC, compared to fibres, increases bonded area and tensile properties of nanofibrils (Eriksen et al 2008).

Burst index

Similarly, burst index generally increased with increased addition of NFC. Burst indicates how much pressure that paper can tolerate before it ruptures. Factors affecting burst strength includes interfibre bonding, fibre length and sheet stretch. The paper sheets experienced an increase in burst strength between 13 to 49% upon addition of NFC from 2 to 10% with values between 3.42 to 4.52 kPam^2 g^{-1}. The maximum increase in burst strength at 4.52 kPam^2 g^{-1} was achieved at addition level 8%. The positive effect of adding NFC on burst strength could be attributed to its high surface area, as a result of its nanoscale dimension. In addition, it also improved bonding network through the establishment of strong hydrogen bond between NFC and fibre during drying.

Tear index

On the other hand, in general, paper containing NFC displayed an opposite trend (versus tensile and burst strengths) in which tear strength decreased with increased NFC (Figure 3). However the reduction is quite minimal with only up to 6%, at most at 8% addition level. This is in agreement with other similar research (Erikson et al. 2008, Petroudy et al. 2014). Tear strength

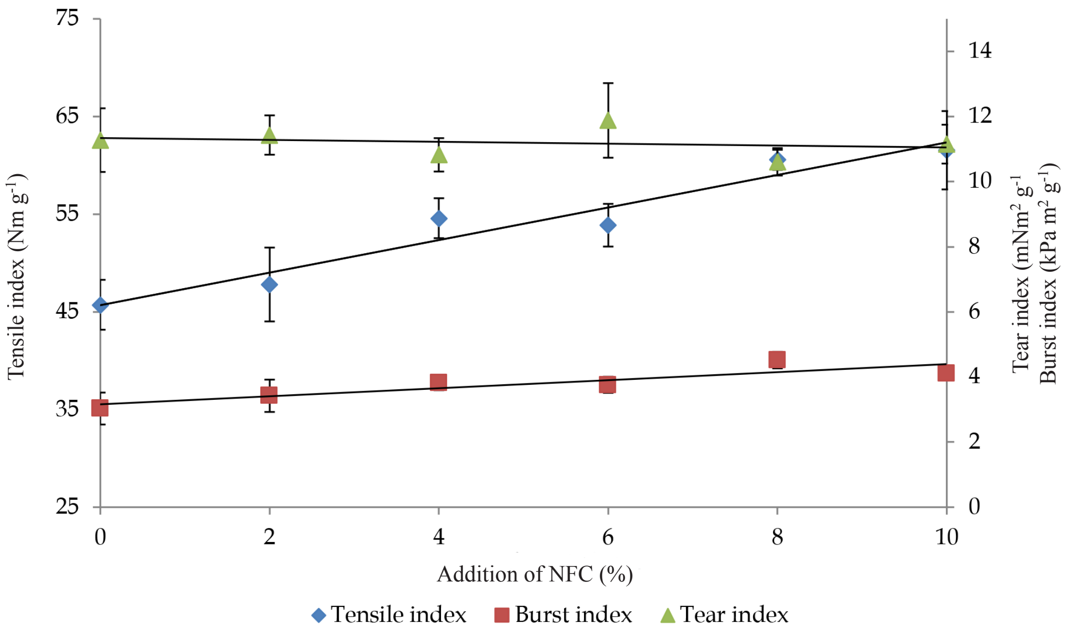

Figure 4 Strength properties of paper added with nanofibrillated cellulose (NFC) at varying dosage

is influenced by several factors such as total number of participating fibres in paper rupture, fibre length and number and fibre-to-fibre bond strengh. In theory, there are two contributions involved in tearing action, i.e. pulling fibres out of the paper sheet and rupturing the fibre. The fibres taking part in rupture usually depends on the grammage (gram per meter square of a paper). In flexible sheets, the force is distributed through a larger area and involves more number of fibres, whereas in rigid paper, the force is focused on a small area, thus only small number of fibres are involved. Due to incorporation of nanoscale fibres, the energy to pull out is lower than the energy to severe individual fibres, causing a paper rupture. This behavior is similar to paper made from highly beaten fibre due to the high amount of fine fibres.

According to Boufi et al. (2016), the strengthening effect of NFC on paper could be further explained by two mechanisms. First, NFC acts as an adhesive agent that bridges the neighbouring fibres and improves the fibre-fibre bonding, and thus increase the bonded area. Secondly, NFC generates a new different fibre network surrounded by larger fibres that results in the increase of paper strength. In addition, strong interaction between fibres and NFC is expected due to their similar structure. Incorporation of NFC onto paper might similarly be viewed to the behavior of beaten fibres in papermaking.

Fold

Folding endurance test is determined to measure the paper's ability to maintain its strength after repeated folding under a predetermined load (usually 800g or 1 kg). Folding test is meaningful for paper that are exposed to bending, folding and creasing. Folding endurance is affected by fibre length and fibre bonding. Adding 8% NFC to paper sheet improved folding endurance up to 223% from controlled sample (Figure 5). The increase in fold was between 39 to 226% with values between 64 and 150. However, folding decreased as more NFC was added. This maybe attributed to the increased brittleness of paper as a result of stronger interfibre bonding.

Effect of nanofibrillated cellulose (NFC) on optical properties

Addition of 2 to 10% NFC did not have much effect on brightness as the change is too small to observe (Figure 6). Likewise, adding NFC on paper did not change the opacity value much, as shown in Figure 5. Opacity is an essential property in printing paper to ensure that printed object cannot be seen from the back of paper. Opacity is affected by thickness, filler added, extent of beating and bleaching. The higher the opacity, the less objects can be seen through the paper.

Effect of nanofibrillated cellulose (NFC) on air permeability

Air permeability decreased as more NFC was gradually added (Figure 7). Reduction in 60% air permeability was observed in controlled sample when 8% NFC was added into paper, with value of 2.49 μm $(Pa.s)^{-1}$. For paper packaging or greaseproof paper, low air permeability is desired;

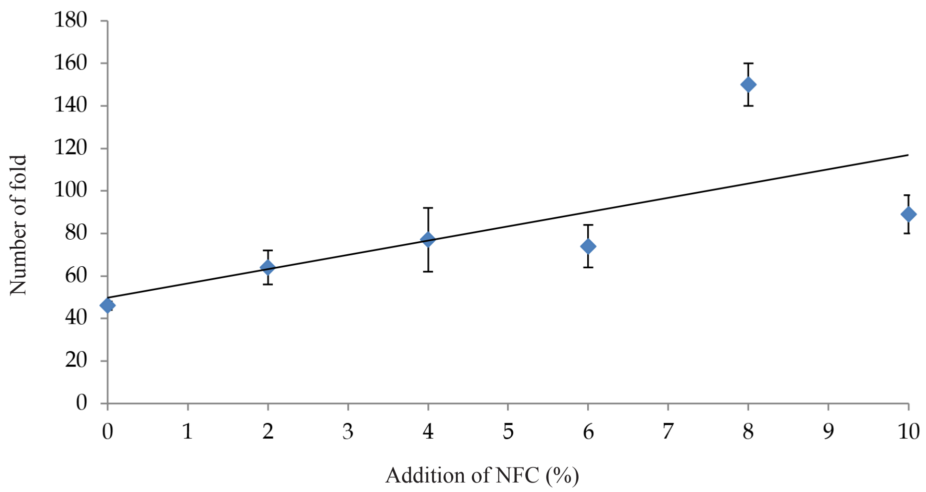

Figure 5 Folding endurance of paper with increasing addition of nanofibrillated cellulose (NFC)

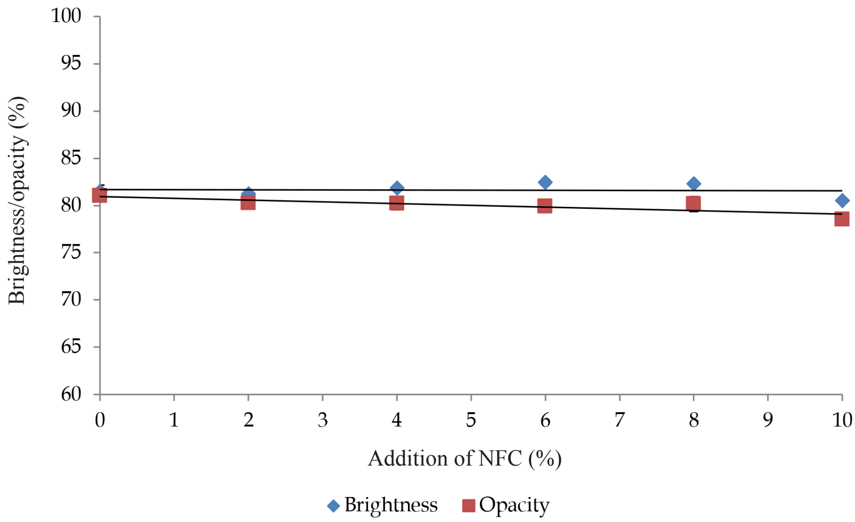

Figure 6 Effect of nanofibrillated cellulose (NFC) on optical properties of paper

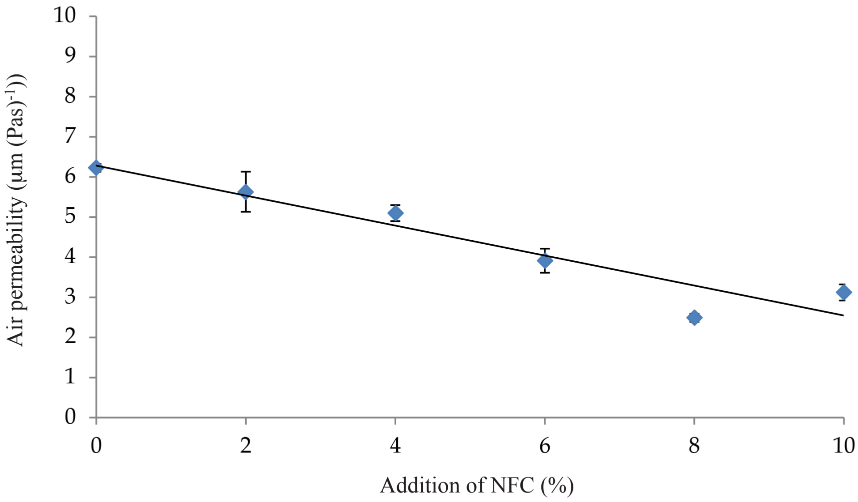

Figure 7 Effect of nanofibrillated cellulose (NFC) at increasing addition level on air permeability of paper

slower air passage is allowed due to compact structure of paper. This is extremely useful for food grade packaging as this will reduce microorganism contact with food materials.

CONCLUSIONS

In conclusion, NFC was successfully produced from kelempayan bleached pulp. Paper reinforced with NFC showed enhancement in mechanical strength such as tensile, burst and fold and also increased density. At the same time, the presence of NFC decreased air permeability and tear. The NFC facilitated the formation of fibre to fibre bonding that contributed to increased strength.

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REFERENCES

Abdul-Khalil HPS, Davoudpour Y, Islam MN et al. 2014. Production and modification of nanofibrillated cellulose using various mechanical processes: a review. *Carbohydrate Polymers* 99: 649–665. http://doi.org/10.1016/j.carbpol.2013.08.069.

Afra E, Yousefi H, Hadilam MM & Nishino T. 2013. Comparative effect of mechanical beating and nanofibrillation of cellulose on paper properties made from bagasse and softwood pulp. *Carbohydrate Polymers* 97: 725–730. https://doi.org/10.1016/j.carbpol.2013.05.032.

Boufi S, González I, Delgado-Aguilar M, Tarrès Q, Pèlach, MA & Mutjé P. 2016. Nanofibrillated cellulose as an additive in papermaking process: a review. *Carbohydrate Polymers* 154: 151–166. https://doi.org/10.1016/j.carbpol.2016.07.117.

Brinchi L, Cotana F, Fortunati E & Kenny JM. 2013 Production of nanocrystalline cellulose from lignocellulosic biomass: technology and applications. *Carbohydrate Polymers* 94: 154–69. https://doi.org/10.1016/j.carbpol.2013.01.033.

Brodin FW, Gregersen ØW & Syverud K. 2014. Cellulose nanofibrils: challenges and possibilities as a paper additive or coating material — a review. *Nordic Pulp and Paper Research Journal* 29: 156–166. http://doi.org/10.3183/NPPRJ-2014-29-01-p156-166.

Dai L, Losng Z, Lv Y & Feng Q. 2014. The role of formic acid pretreatment in improving the carboxyl content of tempo-oxidized cellulose. *Cellulose Chemistry and Technology* 48: 469–475.

Eichhorn SJ, Dufresne A, Aranguren M et al. 2010. Review: current international research into cellulose nanofibres and nanocomposites. *Journal of Materials Science* 45: 1–33.

Eriksen Ø, Syverud K. & Gregersen Ø. 2008. The use of microfibrillated cellulose produced from kraft pulp as strength enhancer in TMP paper. *Nordic Pulp and Paper Research Journal* 23: 299–304.

González I, Boufi S, Pèlach MA, Alcalà M, Vilaseca F & Mutjé P. 2012. Nanofibrillated cellulose as paper additive in eucalyptus pulps. *BioResources* 7: 5167–5180. http://doi.org/DOI:10.15376/biores.7.4.5167-5180.

Habibi Y, Lucia LA & Rojas OJ. 2010. Cellulose nanocrystals: chemistry, self-assembly, and applications. *Chemical Reviews* 110: 3479–3500. https://doi.org/10.1021/cr900339w.

Hii C, Gregersen ØW, Chinga-Carrasco G & Eriksen Ø. 2012. The effect of MFC on the pressability and paper properties of TMP and GCC based sheets. *Nordic Pulp and Paper Research Journal* 27: 388–96.

Hubbe MA. 2006. Bonding between cellulosic fibres in the absence and presence of dry-strength agents: A review. *BioResources* 1: 281–318.

Kajanto I & Kosonen M. 2012. The potential use of micro- and nanofibrillated cellulose as a reinforcing element in paper. *Journal of Science and Technology for Forest Products and Processes* 2: 42–48.

Khoo KC & Peh TB. 1982. Proximate chemical composition of some Malaysian hardwoods. *Malaysian Forest* 45: 244–262.

Klemm D, Cranston ED, Fischer D et al. 2018. Nanocellulose as a natural source for groundbreaking applications in materials science: Today's state. *Materials Today* 21: 720–48. https://doi.org/10.1016/j.mattod.2018.02.001.

Lai PS, Ho WS & Pang SL. 2013. Development, characterization and cross-species transferability of expressed sequence tag-simple sequence repeat (EST-SSR) markers derived from kelampayan tree transcriptome. *Journal of Chemical Information and Modeling* 12: 225–235. http://doi.org/10.1017/CBO9781107415324.004.

Lal M, Dutt D, Tyagi CH, Upadhyay JS & Upadhyay S. 2010. Characterization of *Anthochephalus cadamba* and its delignification by kraft pulping. *Tappi Journal* 9: 30–37.

Missoum K, Belgacem MN & Bras J. 2013. Nanofibrillated cellulose surface modification: a review. *Materials* 6: 1745–1766. http://doi.org/10.3390/ma6051745.

Peh TB, Khoo KC, Lee TW & Md Yusoff MN. 1986. *Pulp and Paper Industry and Research in Peninsular Malaysia.* Malayan Forest Record No. 31. FRIM, Kepong.

Petroudy SR, Syverud K, Chinga-Carrasco G, Ghasemain A & Resalati H. 2014. Effects of bagasse microfibrillated cellulose and cationic polyacrylamide on key properties of bagasse paper. *Carbohydrate Polymers* 99: 311–318. https://doi.org/10.1016/j.carbpol.2013.07.073.

Salas C, Nypelö T, Rodriguez-Abreu C, Carrillo C & Rojas OJ. 2014. Nanocellulose properties and applications in colloids and interfaces. *Current Opinion in Colloid and Interface Science* 19: 383–396. http://doi.org/10.1016/j.cocis.2014.10.003.

Savard J, Besson A & Morize S. 1954. *Analyse Chimique des Bois Tropicaux.* Centre Technique Forestier Tropical No. 5. Nogent-sur-Marne, France.

Sim K, Ryu J & Youn HJ. 2015. Structural characteristics of nanofibrillated cellulose mats: effect of preparation conditions. *Fibers and Polymers* 16: 294–301. http://doi.org/10.1007/s12221-015-0294-4.

Siró I & Plackett D. 2010. Microfibrillated cellulose and new nanocomposite materials: a review. *Cellulose* 17: 459–494. https://doi.org/10.1007/s10570-010-9405-y.

Taipale T, Österberg M, Nykänen A, Ruokolainen J & Laine J. 2010. Effect of microfibrillated cellulose and fines on the drainage of kraft pulp suspension and paper strength. *Cellulose* 17: 1005–1020. https://doi.org/10.1007/s10570-010-9431-9.

Wise LE, Murphy M & D'addieco AA. 1946. Chlorite holocellulose: its fractionation and bearing on summative wood analysis and on studies on the hemicelluloses. *Paper Trade Journal* 122: 35–43.

Zayed MZ, Zaki MA, Ahmad FB, Ho WS & Pang SL. 2014. Comparison of mimosine content and nutritive values of *Neolamarckia cadamba* and *Leucaena leucocephala* with *medicago sativa* as forage quality index. *International Journal of Scientific and Technology Research* 3: 146–150.

Zimmermann T, Bordeanu N & Strub E. 2010. Properties of nanofibrillated cellulose from different raw materials and its reinforcement potential. *Carbohydrate Polymers* 79: 1086–1093. http://doi.org/10.1016/j.carbpol.2009.10.045