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The Effect of Using Cellulose Nanofiber from Kapok (*Ceiba pentandra*, L) as Reinforcement on The Properties of Recycled Papers

Fransiska Angelina Rezekinta [a], Anwar Kasim [b,*], Edi Syafri [c], Firman Ridwan [d], Irawati Chaniago [a], Hideaki Ichiura [e]

[a] Agriculture Faculty, Andalas University, Padang, Indonesia
[b] Agricultural Technology Faculty, Andalas University, Padang, Indonesia
[c] Agricultural Technology Faculty, Politeknik Pertanian Negeri Payakumbuh, Lima Puluh Kota, Indonesia
[d] Mechanical Engineering Faculty, Andalas University, Padang, Indonesia
[e] Agriculture and Marine Science Faculty, Kochi University, Kochi, Japan

Abstract. *Recycling is important for achieving environmentally sustainable products and waste reduction. The trend has led to the addition of reinforcement to recycling process to ensure the effective usage of wasted papers. Therefore, this study was conducted to determine the effect of using cellulose nanofiber (CNF) synthesized from kapok fibers as reinforcement in recycled papers. CNF was applied at different concentrations of 2, 4, 6, 8, and 10% of recycled paper production and the analysis was conducted by testing tensile index, density, and brightness. Moreover, X-ray diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), and UV-Vis analysis were used to determine the characteristics of the paper. The results showed that CNF addition influenced tensile index, brightness, and density of recycled papers. This was observed from an increase in tensile strength by up to 76.32% and density at approximately 0.58 g/cm^3 for 10% CNF addition. Meanwhile, the brightness level was reduced due to the compact nature of paper produced. The trend led to the conclusion that the addition of CNF could impact the characteristics of recycled papers.*
Keywords: *cellulose nanofiber, fiber, kapok, paper, recycling.*

Type of the Paper: Regular Article.

1. Introduction

Wasted papers are a significant component of solid waste produced through residue printed papers, magazines, newspapers, or tissue papers [1]. These papers are normally used as raw materials to produce recycled papers but at low mechanical properties due to reduced cellulose bonding during the pulp-making process [2]. The impurities and contaminants such as inks, adhesives, and other materials from previous usage can also disrupt the bonding process with subsequent effects on the mechanical properties of recycled papers produced [3]. Therefore, reinforcements such as nanocellulose are required to be added in order to enhance the properties of recycled papers.

Nanocellulose can be synthesized from natural fibers and the application of the bio-composites has recently gained a lot of attention due to the advanced characteristics [4-6].

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* First corresponding author
Email: anwar_ks@yahoo.com*

Cellulose nanofiber (CNF) was produced through a series of processes to remove cellulose fibrils from the cell walls. This can be achieved through hydrolysis which is part of the most common methods and focuses on using chemicals such as acid treatment and catalytic oxidation [7]. CNF has been widely used as reinforcement in different fields of science to improve material characteristics in the form of moisture absorption, crystallinity, thermal stability [8], mechanical strength, and high specific surface area [9]. Balea et al. [10] used CNF as reinforcement to increase tensile strength of printed papers. The material has also been used in several studies to reduce the properties of different products. Håkansson [11] observed that poorly dispersed CNF could lead to a non-homogeneous structure, reducing the mechanical properties of the film produced. Moreover, CNF has the ability to cause materials to become non-elastic due to crystal formation in some polymers [12].

CNF can be synthesized from natural sources such as cotton, jute, kapok fiber, and other fibers. This study focuses on kapok fibers obtained from the tropical kapok tree that grows in Indonesia. The significant hollowness, 80 to 90%, increases the suitability of producing low-density polymer composites [13,14] in addition to the significant demand for oil-absorbent agents due to the hydrophobic characteristics [15,16]. Based on some literature, kapok fibers are mostly used because of their hollowness and hydrophobicity. Another important point is that the fibers can be used as a cellulosic material.

Kapok fibers constitute 5.7% of all fiber production in the country [17] and the usage is limited to filler materials despite the potential to be used as cellulosic material, considering the existence of 55.69% cellulose and 16.92% hemicellulose [18]. The trend shows the possible application as raw materials for CNF as analyzed in some studies [19,20]. Therefore, this study aimed to investigate the effect of using CNF produced from kapok fibers as reinforcement on the properties of recycled papers.

2. Materials and methods

2.1. Materials

Wasted papers from offices were used as raw materials in this study. The process was initiated by soaking the wasted papers in a 2% w/v sodium hydroxide solution at room temperature overnight. The pulps were later rinsed with distilled water to become neutral and subsequently stored in the refrigerator at 4^{o}C to be used in the paper production process. Moreover, CNF was produced using kapok fibers from Central Java, Indonesia. The analytical grades of Sodium hydroxide (NaOH), Sodium chlorite ($NaClO_2$), and Sulfuric acid (H_2SO_4) used were obtained from Merck, Germany.

2.2. CNF preparation

This study prepared 15 g kapok fibers which were mixed with 3.5% w/v (NaOH) solution and placed at room temperature (25^{o}C) for 60 minutes. The fibers were later treated with 4% (w/v) NaOH solution in the pulping process for 60 minutes at 1 atmosphere pressure and rinsed with water to achieve a neutral pH. The washed fibers were bleached using 2% (w/v) $NaClO_2$ and acetic acid solutions for 32.5 minutes. Moreover, a hydrolysis process was conducted using 7M (v/v) sulfuric acid (H_2SO_4) solution for 75 minutes at 45^{o}C. Centrifugation was later applied to neutralize the suspension produced. The fibers were homogenized using a high-speed blender with a 17,400-rpm speed for 25 minutes followed by the filtration of the suspension with a 500-mesh filter membrane to produce CNF film. Furthermore, the film was dried in a press machine for 5 minutes at 100^{o}C temperature and characterized with Fourier Transform Infrared Spectroscopy (FTIR), X-ray diffraction (XRD), and UV-Vis spectrophotometer analysis.

2.3. Production of papers

The pulps prepared were diluted by adding CNF suspension at 2, 4, 6, 8, and 10% relative to the dry mass. The mixture was poured evenly into a 27 x 28 cm frame immersed in water with a grammage target of 70 g cm^{-2}. After the confirmation, the remaining water was removed followed by the retriever of papers and drying in normal sunlight.

2.4. Tensile strength

Tensile strength was analyzed by using a Universal Testing Machine (Strograph-R1, Shimadzu). This was achieved by cutting papers into 2 cm x 8 cm size for test preparation and subsequent application of 10 k/N force at 5 mm/min crosshead speed.

2.5. Density

The papers produced were cut into 10 cm x 10 cm sample sizes and weighed in analytical measurement (ABJ 220-4NM, Germany). Density was calculated through the division of the paperweight by the volume.

2.6. Brightness

Brightness was analyzed using a ColorFlex EZ Spectrophotometer (HunterLab, Virginia). This was achieved by cutting all samples into 10 cm x10 cm long and subsequently placing them at the center of the Hunter Lab glass to measure brightness. The data recorded were used for the calculation through the following Equation (1).

$$B\ (\%) = \sqrt{(100 - L)^2 a^2 + b^2} \quad (1)$$

Where B was brightness, L was lightness based on achromatic white, grey, and black, a was the chromaticity of the mixed red-green color, and b was the chromaticity of the mixed blue-yellow color.

2.7. Fourier Transform Infrared Spectroscopy (FTIR) analysis

The surface chemistry of CNF film was analyzed by using FTIR (Shimadzu, Japan) through KBr pellet method. The spectrum was recorded at ambient temperature with the wavenumber ranging from 3331 to 410 cm^{-1}.

2.8. X-ray Diffraction (XRD)

The crystallinity of CNF films was measured by using XRD XPERT PRO PANalytical (Malvern, UK). The samples were analyzed at the radiation wavelength of 1.5000 Å and 45 kV–40 mA followed by the scanning from 10° to 100° in the 2θ scan range. The samples, mounted without support using two polyoxy methylene rings, were analyzed in reflection geometry, and crystallinity index (CI) was calculated using the following Equation (2):

$$\mathrm{CI} = \frac{\mathrm{I_c} - \mathrm{I_{am}}}{\mathrm{I_c}} \mathrm{x} 100\% \quad (2)$$

Where, $\mathrm{I_c}$ is the crystalline intensity and $\mathrm{I_{am}}$ is the amorphous intensity of cellulose.

2.9. UV-Vis spectrophotometer analysis

The absorbance of the prepared CNF films was measured using UV-Vis test conducted with UV-Vis Spectrophotometer (UV-1900, Shimadzu, Japan). The sample was cut to a width of 1 cm and a length of 4 cm before placement in the cuvette which was transferred to the spectrophotometer and analyzed at 400 to 700 nm wavenumber.

2.10. Statistical analysis

The data obtained from the experiments were analyzed using a one-way analysis of variance (ANOVA). This was followed by Duncan's multiple range test at a level of $P < 0.05$.

3. Results and Discussion

3.1. Effect of CNF on tensile strength

The addition of CNF led to an increase in tensile strength of recycled papers as shown in Fig. 1. The untreated recycled papers had 11.15 MPa which significantly increased at $P < 0.05$ to 13.01 MPa with the addition of 2% CNF, 15.24 MPa at 4%, 16.53 MPa at 6%, 18.48 MPa at 8%, and 19.66 MPa at 10% CNF compared to the other treated papers. This was in line with previous observations that conventional papers produced using cellulose had 40 MPa tensile strength. Zeng et al. [9] also reported that the addition of 5% CNF to recycled papers increased tensile strength by 108.32%.

The increase was attributed to the advancement in the bonding between pulp and CNF [21,22]. Therefore, papers with more CNF concentration had higher tensile strength. Another observation was a high surface area and the existence of several hydroxyl groups in CNF facilitated strong hydrogen bonding with cellulose in the paper matrix, contributing to an improvement in tensile strength [23].

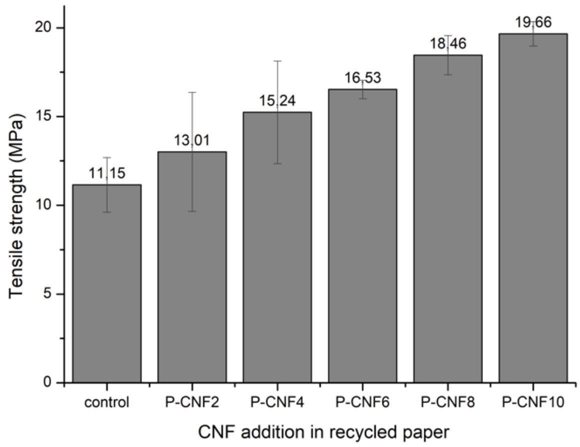

Fig. 1. Tensile strength of recycled papers at different CNF concentrations (control paper, P-CNF2: 2% CNF addition, P-CNF4: 4% CNF addition, P-CNF6: 6% CNF addition, P-CNF8: 8% CNF addition, P-CNF10: 10% CNF addition)

3.2. Effect of CNF on brightness

The addition of CNF decreased brightness of recycled papers. As shown in Table 1, brightness of control paper was 86.23% and decreased to 82.81% with the addition of 10% CNF. The reduction was statistically insignificant (P > 0.05) as presented in Fig. 2 possibly due to the compactness of papers with CNF addition, specifically at 10%. Moreover, the non-uniformly dispersed CNF particles could have caused areas of higher light absorption, leading to the reduction in brightness of papers produced [24].

Table 1. Brightness, thickness, and density of recycled papers at different dosages of CNF

Sample	CNF (%)	Brightness (%)	Thickness (mm)	Density(g/cm^3)
control	0	86.23±0.28	0.17±0.02	0.53±0.10
P-CNF2	2	85.84±0.27	0.17±0.03	0.49±0.07
P-CNF4	4	85.73±0.55	0.18±0.01	0.56±0.11
P-CNF6	6	85.97±0.28	0.18±0.01	0.52±0.04
P-CNF8	8	85.73±0.26	0.18±0.01	0.56±0.04
P-CNF10	10	82.81±4.02	0.18±0.01	0.58±0.02

High density can contribute to the reduction of brightness because CNF with nano-size particles has the ability to fill the pores and spaces in papers, hindering light transmission and reflection. The trend also reduces the scattering of light which is capable of lowering the absorption process [25]. Moreover, the thickness of papers can influence density. This is possible because thicker papers have reduced air spaces between the fibers [26], causing an increase in density.

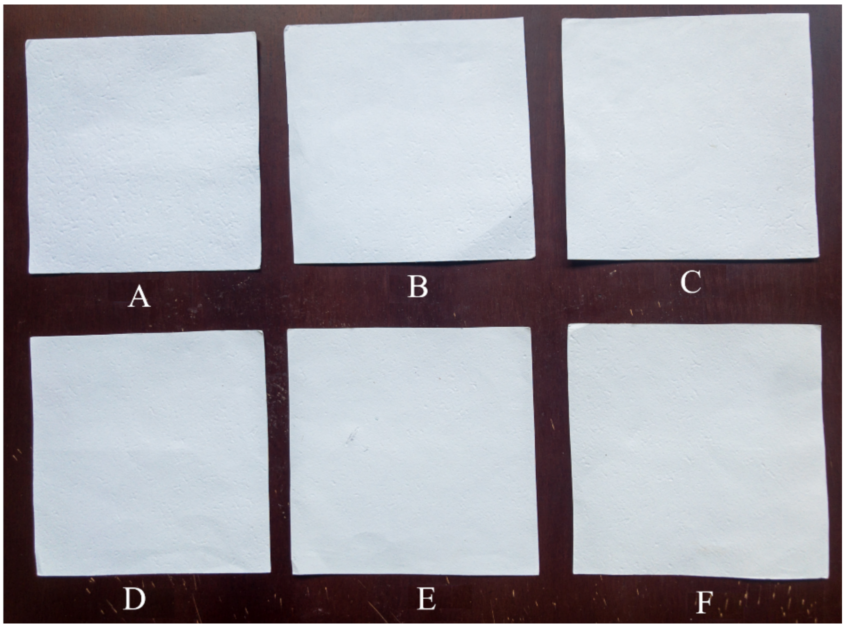

Fig. 2. Recycled papers at different CNF concentrations (A: control paper, B: 2% CNF addition, C: 4% CNF addition, D: 6% CNF addition, E: 8% CNF addition, and F: 10% CNF addition)

3.3. Density of papers

The results presented in Table 1 showed an increase in density of papers due to the addition of CNF. This was confirmed by the fact that control paper had 0.53 g/cm^3 but the value increased after CNF was added because the pores in the fiber networks were filled, reducing the spaces. A similar result was reported in a previous study that strong inter-fiber bonds were formed by CNF due to the nano-sized dimensions, leading to the production of more compact papers [25]. Furthermore, the overall surface area coverage in paper structure was enhanced and this led to the reduction of voids and an increase in density of paper sheets [27].

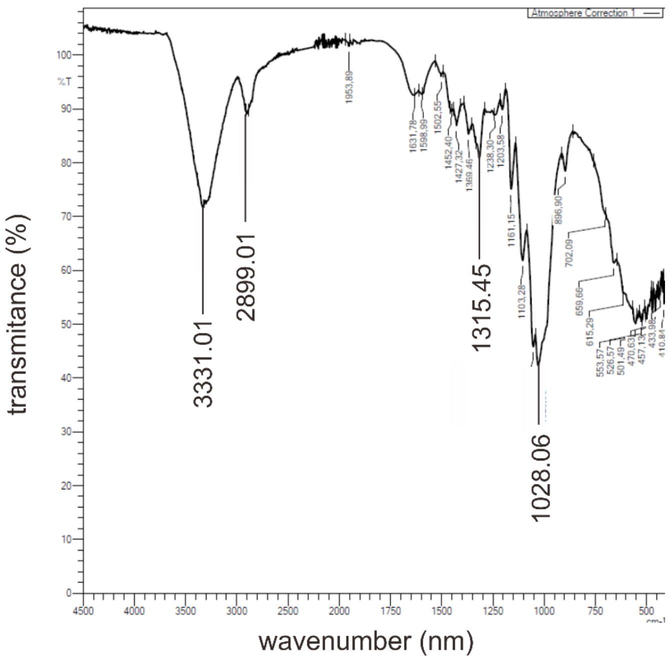

Fig. 3. FTIR spectra of CNF film

3.4. FTIR and XRD analyses of CNF film

FTIR analysis of CNF film used in reinforcement of recycled papers is presented in the graph of Fig. 3 with a focus on the peaks for the cellulose unit. C-H functional group from stretching and deformation of glucose unit was found at a peak of 2899 cm^{-1} while the vibration peak of O-H, corresponding to intra and intermolecular H-bonds [28] was observed at 3331 cm^{-1}. Moreover, the peaks in the 1200-1500 cm^{-1} showed C-O-C vibration in cellulose intra and intermolecular structure. Several fingerprints had a reasonably high intensity in the 1000 cm^{-1} - 400 cm^{-1} peak range, indicating the deformation of the glucose unit (C_1-O-C_4) [29,30].

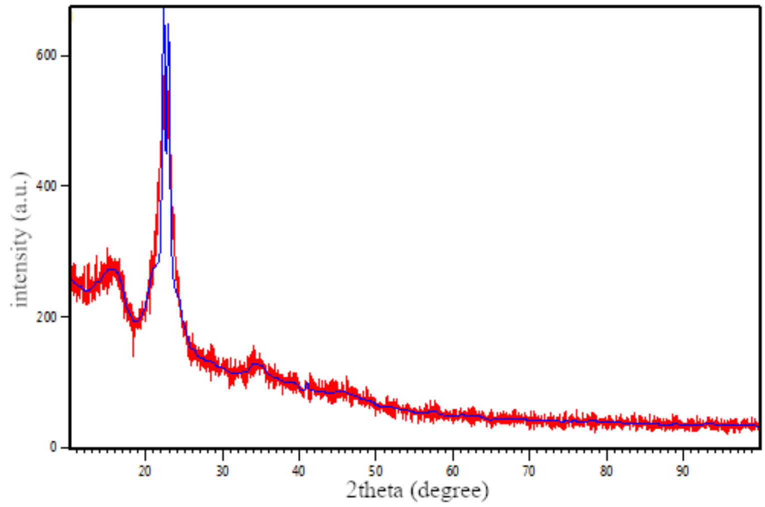

Fig. 4. XRD pattern of CNF film

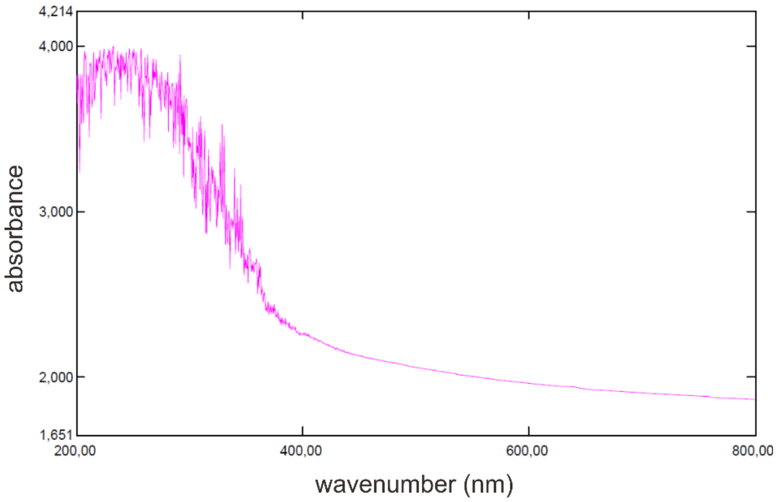

Fig. 5. UV-Vis of CNF film

XRD pattern showed that crystallinity of CNF film was 48.99%. The index was observed to be lower than the 68% reported in previous literature [31] possibly due to the application of different synthesis methods. CNF was produced in this study using a high-speed blender which could have led to a higher presence of amorphous components not fully reacting during the hydrolysis process [32]. Fig. 4 shows the peaks of diffraction intensity of CNF papers at 17.3°, 22.3°, and 34.9° which were an indication of the characteristic crystalline structure of cellulose I in the film [33]. Crystallinity index was reported to be capable of increasing tensile strength of papers due to the existence of stronger hydrogen bonding, leading to improved inter-fiber adhesion [34,35].

3.5. Uv-Vis measurement of CNF film

UV-Vis measurement was conducted to determine the transparency of CNF film. The results presented in Fig. 5 showed that CNF film absorbance was between 200 and 400 nm as observed from the cellulose absorbance peaks. The highest transmittance was found to be only 1% and this showed that CNF film obtained was not transparent. Similar results were reported in a previous study [36] that produced an opaque film with the lowest transmittance at less than 3%. The transmittance measurement can also be related to the size or diameter of CNF with low values indicating a relatively large thickness and size of CNF [9].

4. Conclusions

In conclusion, the addition of CNF improved the physical properties of recycled papers produced. This was observed in the increase in tensile strength compared to control paper and the highest was found with 10% CNF addition. The trend was associated with the ability of CNF to fill the pores in papers to ensure rigidity and improved physical characteristics. The addition of more concentration of CNF was observed to have led to higher density of recycled papers produced with the highest found to be 0.58 g/cm^3. However, brightness reduced as the concentration increased due to the compact structure caused by the addition of CNF. The analysis of CNF film for paper reinforcement showed 48.9% crystallinity and the absorbance was estimated at 200-400 nm.

Data availability statement

Data will be shared upon request by the readers.

Author’s contributions

Fransiska Angelina Rezekinta: Writing – original draft, Conceptualization, Methodology, Resources, Formal analysis, Investigation, Data curation. Anwar Kasim: Writing – review & editing, Validation, Data curation, Formal analysis, Conceptualization, Supervision, Funding acquisition. Edi Syafri: Conceptualization, Supervision, Data curation, Writing – review & editing. Irawati Chaniago: Formal analysis, Investigation, Writing – review & editing. Firman Ridwan: Validation, Data curation, Writing – review & editing. Hideaki Ichiura: Writing – review & editing, Data curation, Validation.

Declaration of Competing Interest

The authors of this manuscript declare no conflict of interest or competing interest.

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