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The Characterization of Nanocellulose with Various Durations and NaOH Concentration

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Abstract

Nanotechnology has become an important part of industrial construction, especially in concrete structural materials. Additionally, waste is a severe environmental problem when using paper can impact waste caused by waste. The inner nanocellulose is sourced from paper waste and then synthesized using natrium hydroxide (NaOH) and natrium hypochlorite (NaClO). Nanocellulose from waste paper was synthesized with a 5%, 10%, and 15% NaOH solution and a 2% NaClO solution with variations in heating time (2 hours, 4 hours, and 6 hours) at a temperature of 125℃. The physical, chemical, and microstructural properties were analyzed using Scanning Electron Microscope (SEM), X-Ray Diffraction (XRD), Energy Dispersive X-ray (EDX), and Fourier-transform Infrared Spectroscopy (FT-IR). The XRD results show a higher amorphous percentage after chemical synthesis. A 15% concentration heated for 6 hours, resulted in 79.68% amorphous content, and the lowest amorphous percentage in 5% NaOH with a heating duration of 4 hours, resulting in 63.69% amorphous content. Conversely, if added, acid can reduce the amorphous content and increase the crystal structure. The results of the FT-IR analysis show that treatment using NaOH and NaClO can remove lignin and hemicellulose structures in cellulose fibers. The SEM results show that the size of the structure is smoother and cleaner after treatment with NaOH and NaClO. The results of the EDX test show that with the NaOH and NaClO treatment, the chemical content was close to the chemical content of OPC. Thus, nanocellulose can be used as a substitute for some cement materials in concrete mixtures.

Keywords: NaClO, NaOH, Nanocellulose, Nanotechnology, Nanomaterial, Waste paper.

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1. Introduction

Modern society consumes a lot of paper. So much so that it produces large quantities of paper waste that have become a problem [1]. One of the primary sources of waste in urban areas is paper waste caused by the recycling industry, which has not been fully able to handle it, according to Oliva, et al. [2]. The low recycling rate causes an increase in greenhouse gas emissions, so serious processing and maintenance are needed to achieve desired environmental goals [3]. Recycling waste paper is one of the most appropriate ways to protect the environment from the negative impacts of increasing paper production [4]. Additionally, the construction industry is trying to find materials with low carbon content that are environmentally friendly and sustainable [5].

The use of construction materials from waste recycling further increases efficiency, has a tremendous impact on the economy, and has a huge impact on the environment [6, 7]. Using materials from the waste recycling industry can reduce the level of greenhouse gas emissions. Thus, it is highly recommended that the construction industry uses recycled materials. The utilization of waste as an additional material in construction can be applied in various construction works, one of which can be optimized in the concrete production process [8]. The advancement of nanotechnology spans a number of disciplines, including the world of sustainable construction in civil and environmental engineering. Excellent results have been obtained by adopting nanotechnology in sustainable construction. However, there are still many challenges to address. Advances in the application of nanotechnology to sustainable construction, including improvements in the rheology, strength, and durability properties of concrete, which has been shown to depend on the nanoscopic characteristics of the constituents.

Nanotechnology has two systems, the top-down approach and the bottom-up approach. The top-down approach is where a large material is reduced to a nano size by retaining the original element without atomic-level control. While the bottom-up approach, or what is often referred to as molecular, is from atomic materials that are engineered and enlarged to the nanoscale [9]. In the construction industry, there are various nanomaterials with various sizes and shapes such as nanosilica, nanoaluminia, TiO_2 nano, Fe_2O_3 nano, nanoclay, carbon tubes, graphenes, nanocellulose, and many others that are used to improve the mechanical properties of Ordinary Portland Cement (OPC). Nanocellulose can reduce autogenous shrinkage in ultra-high-performance concrete (UHPC). Using a mixture of 0–0.30% of the cement mass and combined with variations of silica fume from 25% reduced to 15%, the results are very significant in reducing autogenous shrinkage in UHPC [10]. On the other hand, using cardboard waste material as a concrete mixture material with low compressive strength is suitable for partition walls in a no-load room [11]. Paper industry waste can be used as a concrete mixture as an additional material and used for sidewalks because it is able to absorb water [12].

This study aims to obtain new materials that use waste paper materials so that they have additional use and value and reduce the level of pollution found in the environment. The use of material derived from organic materials is, additionally, able to reduce pollution caused by the production of mining goods, such as the use of cement materials in the construction industry. In this case, the researcher seeks to make waste materials into construction materials that can contribute fully to structures, such as mortar and concrete materials.

A top-down approach is used in this study because the main material used is large and is reduced over several treatments to obtain the desired nano size while still paying attention to important elements, such as the cellulose content. Several tests were carried out to determine the physical, chemical, and microstructure content. These tests include Scanning Electron Microscope (SEM), X-Ray Diffraction (XRD), Energy Dispersive X-ray (EDX), and Fourier-transform Infrared Spectroscopy (FT-IR) tests.

Waste paper has the potential to be used as a base for nanocellulose because of its abundant availability and high cellulose content (60–70%), with relatively less hemicellulose (10–20%) and lignin (5–10%) without rough treatment [13]. Nanocellulose treatment using NaOH can remove hemicellulose and lignin contained in cellulose fiber [14]. Cellulose comes from plants and are the fibers obtained from plants of various shapes and sizes [15]. In the chemical pulp process, NaOH, oleic acid, calcium, chloride, and sodium silica are dissolved in water heated to a temperature of 90°C, and then 2.5 kg of waste paper is added in dry conditions [16].

The material was synthesized with 5% NaOH for 2–3 hours at a temperature of 80–100°C using a tool to stir regularly. The cellulose residue obtained was then washed with distilled water and filtered repeatedly to obtain a neutral pH. The next process uses $NaClO_2$, as much as 5%, as a bleach for 2–3 hours at a temperature of 80–100°C while maintaining an acidic pH until the lignin is completely lost. Then re-wash using distilled water until the pH returns to neutral. The cellulose can be dried for 24 hours using an oven [17]. The cellulose slurry was given NaOH (0.5–2.5 M/AGU) then cooked at a temperature of 20–60°C for1–3 hours after which the cellulose was filtered [18].

The material was divided into small sizes and then washed with hot water to clean the dirt from the waste paper, followed by processing a mixture of 1 ml of acetic acid and 1.5 g of sodium chlorite in 130 ml of water heated to 75°C. Synthesis of the nanocellulose using NaOH, using as much as 8% urea/6.5% thiourea, dissolved in water that had previously been cooled to -100°C. Then the bleached slurry was dispersed into the solution and controlled at a temperature of 0–2°C for 30 minutes [19]. From the various explanations above, the synthesis of nanocellulose using Sodium Hydroxide (NaOH) and Sodium Hypochlorite (NaClO), the 5% and 10% NaOH solutions and 15% and 2% NaClO solutions are heated for durations of 2 hours, 4 hours, and 6 hours at a temperature of 125°C. The characterization of nanocellulose includes SEM, XRD, EDX, and FT-IR.

2. Materials and Methods

2.1. Materials

The material used in this research is 70/80gsm HVS paper waste sourced from schools and photocopy shops. The natrium hydroxide (NaOH) used is solid 99%, liquid sodium hypochlorite (NaClO), and distilled water.

2.2. Methods

The paper is cut into small pieces, using up to 20 grams, and cleaned of adhering dirt, clips, and staples so that the chemical content is not affected during synthesis. Then soak for three days at room temperature so that the paper decomposition process results in a paste. Then mill for 30 minutes until it becomes a slurry. After milling, the pulp is synthesized using 5%, 10%, and 15% NaOH and NaClO mixed with 800 ml of distilled water. Then heat for 2 hours, 4 hours, and 6 hours at a temperature of 125°C, stirring constantly. After the synthesis process with NaOH and NaClO is complete, follow with a washing process using distilled water to obtain a neutral pH. The next stage of treatment is the drying process, using an oven with a temperature of 150°C. After the sample is dry, mill to obtain flour-like granules.

2.3. Nanocellulose Characterization

X-Ray Diffraction (XRD) analysis aims to determine the percentage of crystalline and amorphous content in the nanocellulose. XRD was carried out in the physics laboratory of the Faculty of Mathematics and Natural Sciences, Sriwijaya University.

The crystallinity index (CrI) of each sample was calculated using the following equation:

$$Crl = I_{002} - I_{am}/I_{002} \times 100\% \qquad [20]$$

The I_{002} is the crystalline area and I_{am} is the amorphous area multiplied by 100%.

FT-IR was used to determine the functional groups of nanocellulose [21]. FT-IR was carried out on a spectrometer running at a transmission range of 500–4000 cm^{-1} [22]. The FT-IR test was carried out to see the presence of cellulose content. The FTIR spectrum for all test specimens was recorded in several parts of the wave band ranging from 400–4000 cm^{-1} using the ATR method [23]. FT-IR analysis was carried out in the pharmacy laboratory of the Faculty of Mathematics and Natural Sciences, Sriwijaya University.

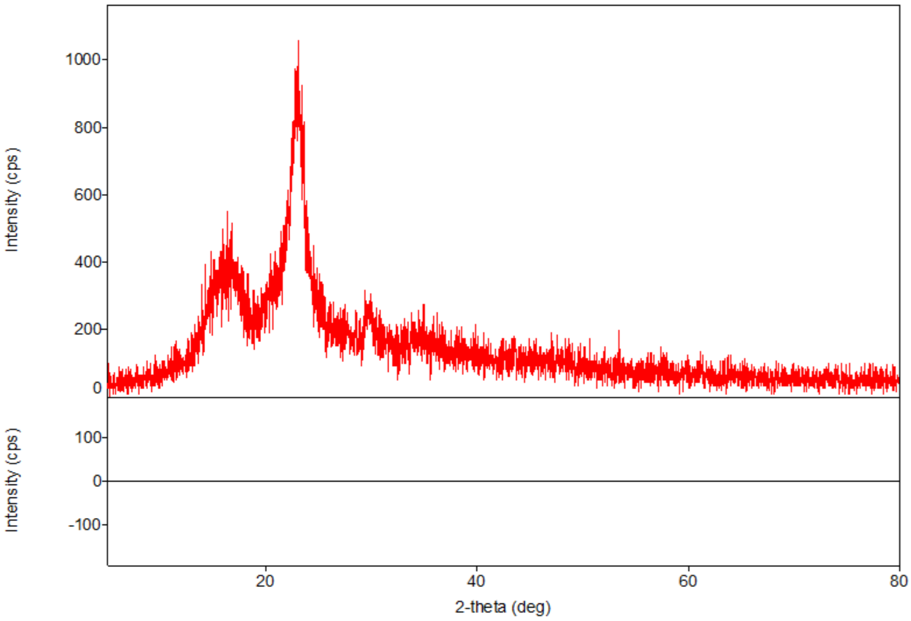
SEM-EDX was used to examine the microscopic structure and surface morphology of cellulose fibers [21]. SEM-EDX was carried out at the Bandung Geological Survey Center.

3. Results and Discussion

3.1. X-Ray Diffranction (XRD)

XRD testing of samples with 5%, 10%, and 15% NaOH concentration variants with durations of 2 hours, 4 hours and 6 hours. Figure 1 explained the results of the XRD analysis of nanocellulose which revealed three peaks in the 2θ diffractogram for 5% NaOH with a 2-hour heating duration of about 2θ = 16.34, 23.06, and 29.42. The structural characteristics of cellulose containing approximately 33.10% crystalline and 66.90% amorphous content. The 5% NaOH revealed three peaks in the 2θ diffractogram for NaOH 5% with a 4 hours duration variant of about 2θ = 16.50, 23.00, and 29.84, and structural characteristics of cellulose containing approximately 36.31% crystalline and 63.69% amorphous content. For the duration of 6 hours at a 5% concentration there were three peaks in the 2θ diffractogram 2θ = 16.84, 22.80, and 29.72, and structural characteristics of cellulose containing approximately 35.29% crystalline and 64.71% amorphous content. Figure 2 revealed three peaks in the 2θ diffractogram for 10% NaOH with a 2-hour duration variant of about 2θ = 16.88, 21.00, and 22.06, and structural characteristics of cellulose containing approximately 30.2% crystalline and 69.82% amorphous content. The 10% NaOH reveals three peaks in the 2θ diffractogram for 10% NaOH with a 4-hour duration variant of about 2θ = 12.50, 20.86, and 22.52, and structural characteristics of cellulose containing approximately 25.54% crystalline and 74.46% amorphous content.

Figure 3 with a duration of 6 hours at a 10% concentration revealed three peaks in the 2θ diffractogram 2θ = 13.22, 20.88, and 22.56, and structural characteristics of cellulose containing approximately 25.44% crystalline and 74.56% amorphous content. Figure 3 revealed three peaks in the 2θ diffractogram for 15% NaOH with a 2-hour duration variant of about 2θ = 13.46, 20.46, and 21.82, and the structural characteristics of cellulose containing approximately 24.51% crystalline and 75.49% amorphous content. Figure 3 showed three peaks in the 2θ diffractogram for 15% NaOH with 4-hour duration variants of about 2θ = 13.48, 20.52, and 21.94, and the structural characteristics of cellulose were approximately 22.46% crystalline and 77.54% amorphous content. With a duration of 6 hours at 15% NaOH concentration revealed three peaks in the 2θ diffractogram 2θ = 12.58, 20.78, and 22.08, and structural characteristics of cellulose containing approximately 20.32% crystalline and 79.68% amorphous content. Table 1 explained that the maximum amorphous content was obtained at 15% NaOH concentration with a duration of 6 hours.



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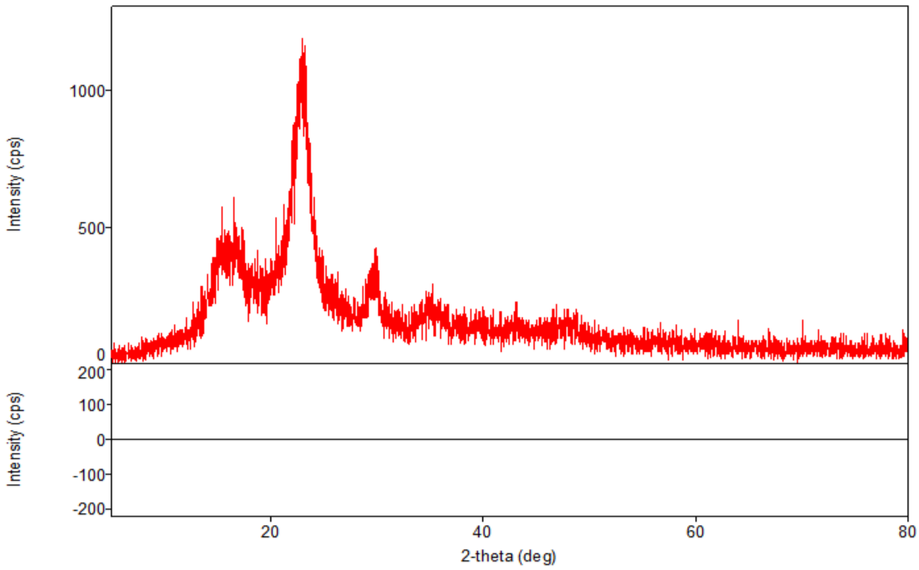

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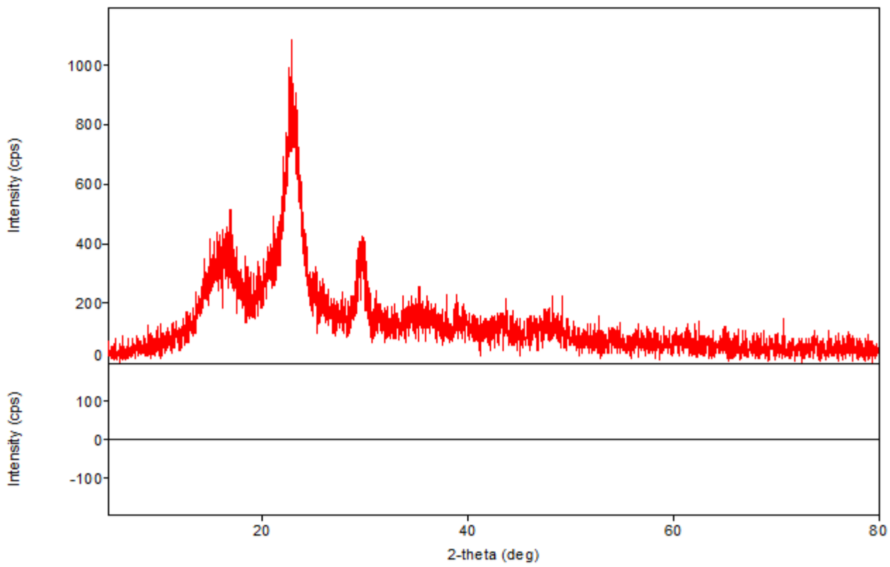


(c)

Figure 1.
XRD diffractogram for 5% NaOH with heating durations of (a) 2 hours, (b) 4 hours, and (c) 6 hours.

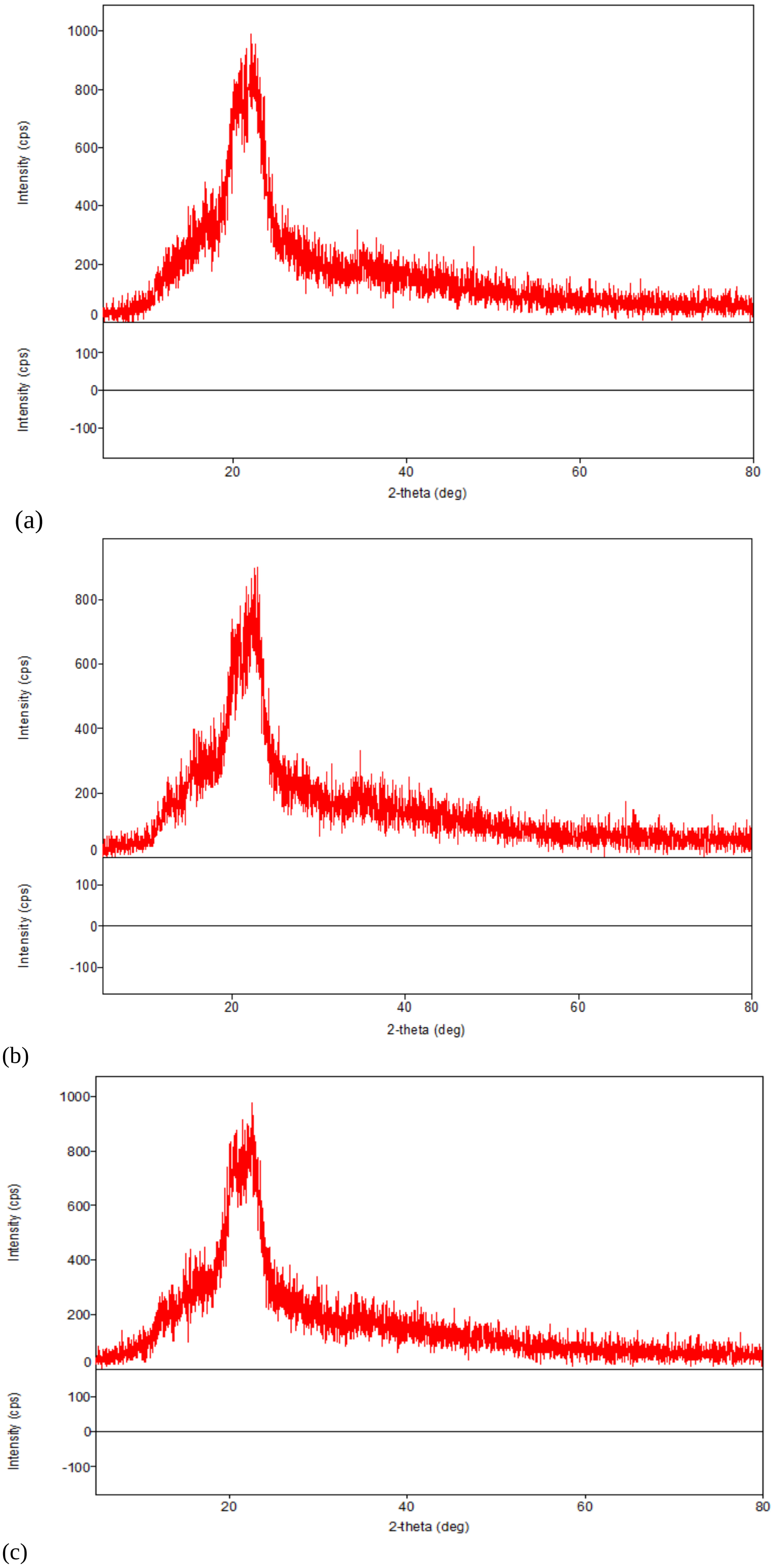
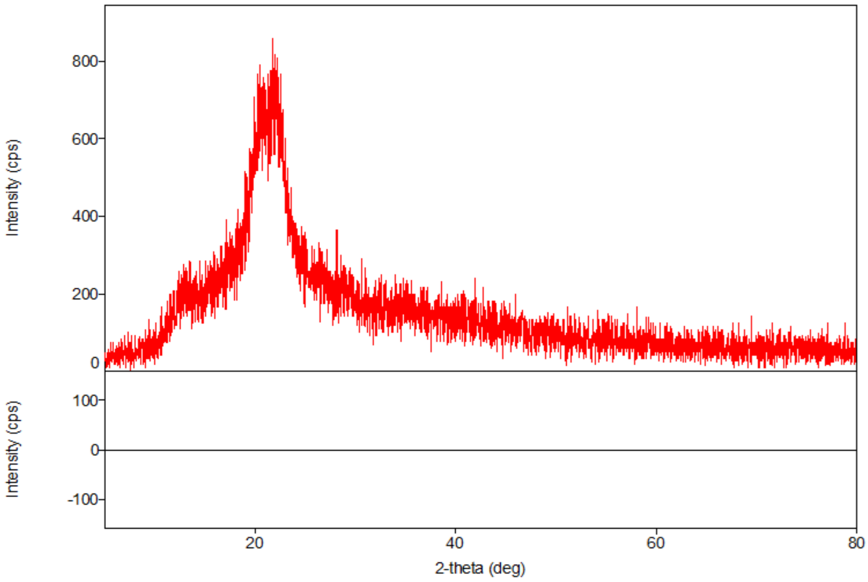


Figure 2.
XRD diffractogram for 10% NaOH with heating durations of (a) 2 hours, (b) 4 hours, and (c) 6 hours.



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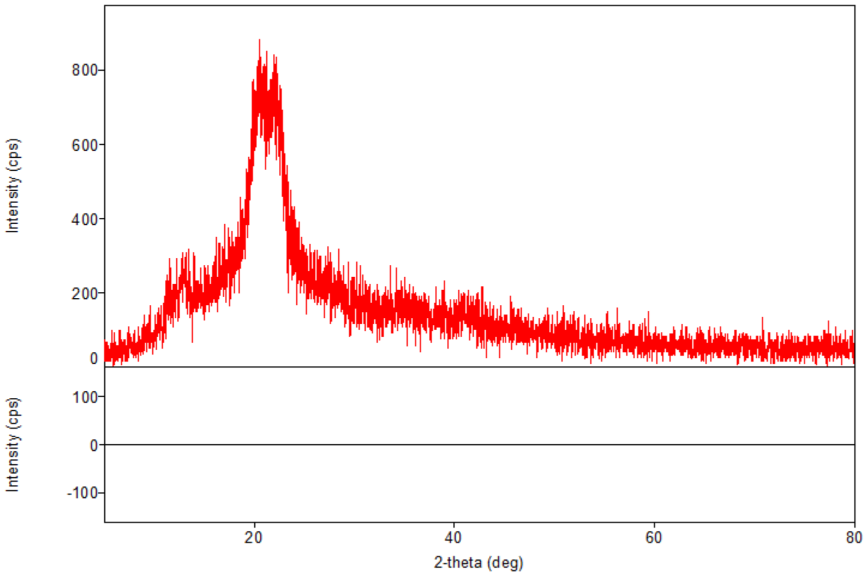

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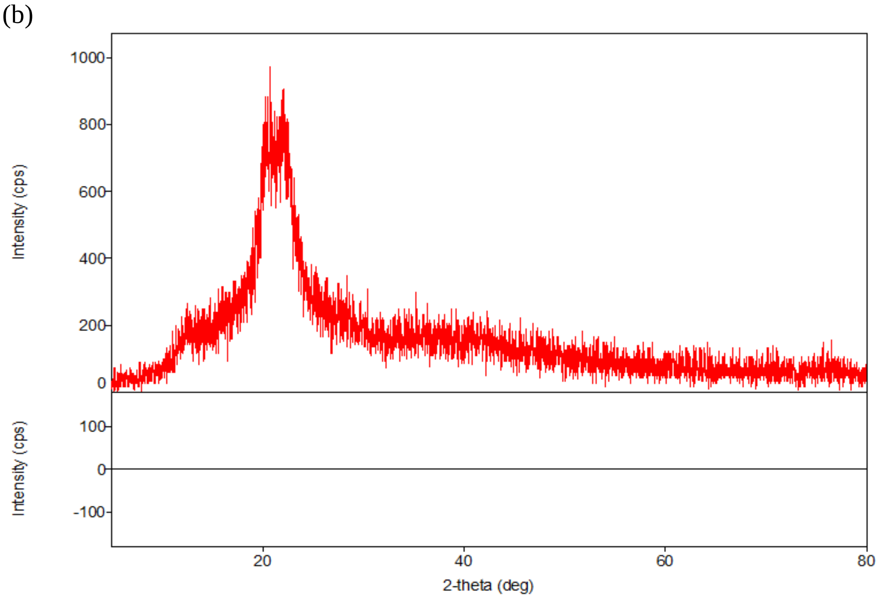


(c)

Figure 3. XRD diffractogram for 15% NaOH with heating durations of (a) 2 hours, (b) 4 hours, and (c) 6 hours.

Table 1.
Percentage of crystalline and amorphous nanocellulose.

Concentration	Percentage (%)	
	Crystalline	Amorphous
5% NaOH 2 Hours	33.10	66.90
5% NaOH 4 Hours	36.31	63.69
5% NaOH 6 Hours	35.29	64.71
10% NaOH 2 Hours	30.18	69.82
10% NaOH 4 Hours	25.54	74.46
10% NaOH 6 Hours	25.44	74.56
15% NaOH 2 Hours	24.51	75.49
15% NaOH 4 Hours	22.46	77.54
15% NaOH 6 Hours	20.32	79.68

3.2. Fourier-Transform Infrared Spectroscopy (FT-IR)

The FT-IR spectra of nanocellulose were synthesized using variants of 5%, 10%, and 15% NaOH concentrations with a durations of 2 hours, 4 hours, and 6 hours. Based on the FTIR spectrum, the samples showed almost identical absorption bands, which means that the samples have the same chemical composition. The reaction with NaOH was needed because the reaction using alkali is able to reduce hydrogen bonds by removing hydroxyl groups [14].

Figure 4 showed that the peak area around the wave number 3,225 cm^{-1} can be associated with O–H, the peak 2,155 to 2,887 cm^{-1} is associated with C–H, while the small area around the wave number 1,565 cm^{-1} can be associated with vibrations of C=C, and the peak at 1,654 cm^{-1} is associated with O–H. The peak at the wave number 1,021 cm^{-1} is associated with C–O. While the peak at 807 cm^{-1} is associated with C–H. In the nanocellulose, this spectrum is associated with the characteristic properties of lignin. While the small shoulder peak is at a wave of 1,565 cm^{-1} can be associated with C=C, which means that the aromatic ring in lignin decreases the effect of NaOH treatment.

Figure 5 showed that the peak area around the wave number 3,216 cm^{-1} can be associated with O–H, the peak at 2,155 to 2,882 cm^{-1} is associated with C–H, while the small area around the wave number 1,553 cm^{-1} can be associated with vibrations of C=C and the peak 1,647 cm^{-1} is associated with O–H [24]. The peak in the wave number 1,019 cm^{-1} is associated with C–O. While the peak of 887 cm^{-1} is associated with C–H. In the nanocellulose spectrum is associated with the characteristic properties of lignin. While the small shoulder peak is at a wave of 1,565 cm^{-1}, which means that by increasing the concentration of NaOH, the peak gets smaller.

Figure 6 showed that the peak area around the wave number 3,295 cm^{-1} can be associated with O–-H, the peak at 2,159 to 2,974 cm^{-1} is associated with C–H, while the small area around the wave number 1,513 cm^{-1} can be associated with vibrations of C=C and the peak 1,643 cm^{-1} is associated with O–H. The peak in the wave number 1,034 cm^{-1} is associated with C–O. While the peak of 893 cm^{-1} is associated with C–H. These results are in accordance with the analysis guidelines that have been carried out by previous researchers where the effect of NaOH and NaClO can remove lignin.

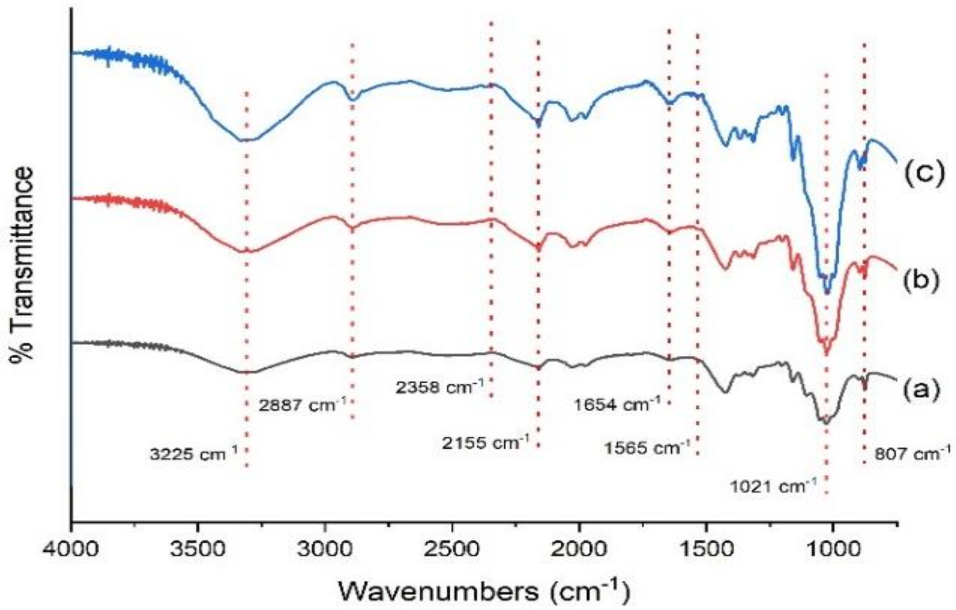


Figure 4.
FT-IR spectrum of nanocellulose waste paper for (a) 5% NaOH, (b) 10% NaOH, and (c) 15% NaOH for a 6-hour duration.

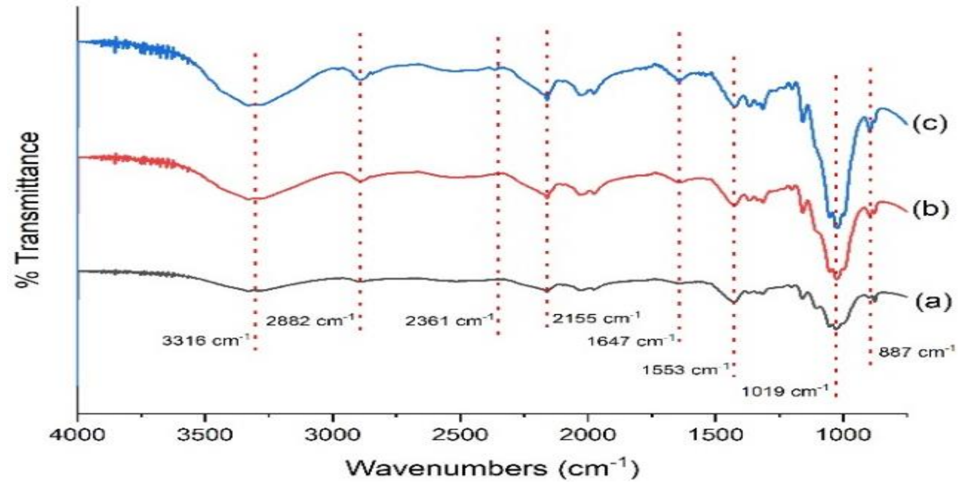


Figure 5.
FT-IR spectrum of nanocellulose waste paper for (a) 5% NaOH, (b) 10% NaOH, and (c) 15% NaOH for a 4-hour duration.

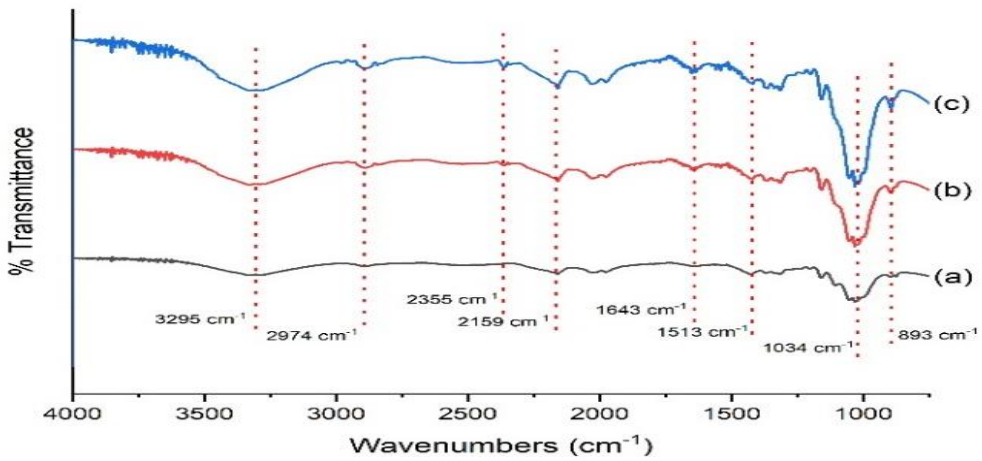


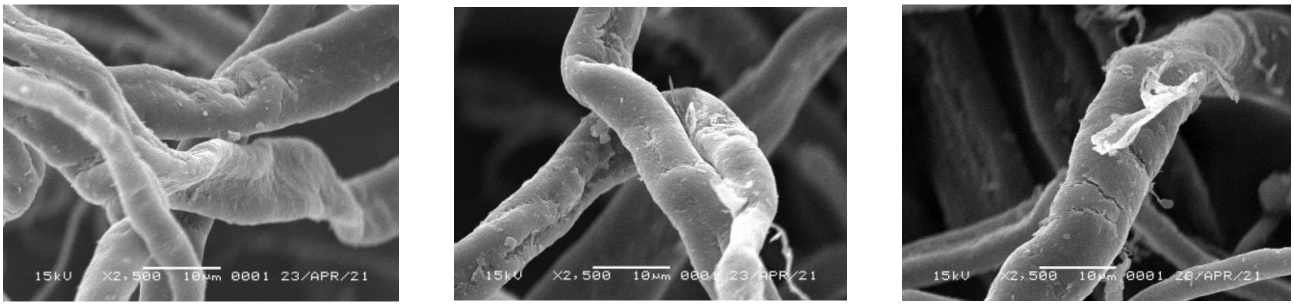
Figure 6.
FT-IR spectrum of nanocellulose waste paper for (a) 5% NaOH, (b) 10% NaOH, and (c) 15% NaOH for a 2-hour duration.

3.3. Scanning Electron Microscop (SEM)

SEM sample testing with NaOH concentration variants of 5%, 10% and 15% with a duration of 2 hours, 4 hours, and 6 hours. After synthesizing nanocellulose using a mixture of NaOH and NaClO the material looks cleaner, purer and more biodegradable. This is because the NaOH content is able to decompose organic materials quickly, while the NaClO content is able to thoroughly clean cellulose until it is whiter in color and all traces of dirt that are left behind. Impurities present in the sample disappear. Figure 7 showed that with a low NaOH concentration, sticking granules were still visible. Figure 8 showed by increasing the percentage of NaOH can reduce the grains that stick so that the results look clean. Figure 9 showed that using a high percentage of NaOH was able to remove granules attached to cellulose fibers until the results look clean, thus the effect of adding NaOH and NaClO can clean cellulose fibers.

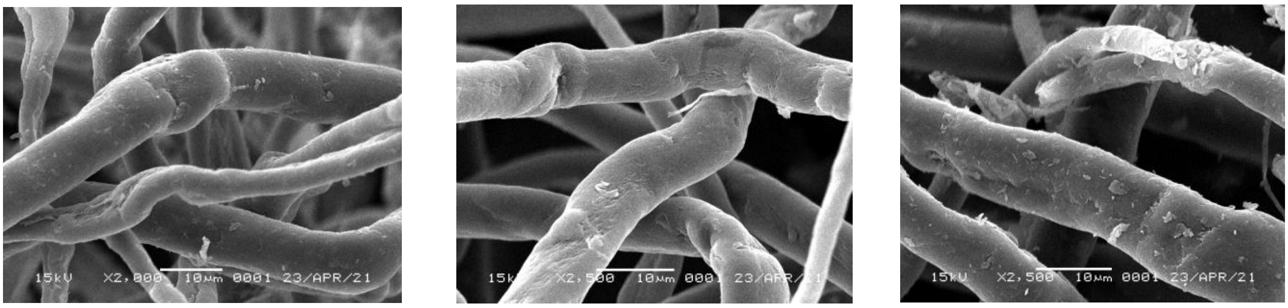
(a) (b) (c)

Figure 7.
SEM results of 2500X 5% NaOH with heating durations of (a) 2 hours, (b) 4 hours, and (c) 6 hours.



(a) (b) (c)

Figure 8.
SEM results of 2500X 10% NaOH with heating durations of (a) 2 hours, (b) 4 hours, and (c) 6 hours.

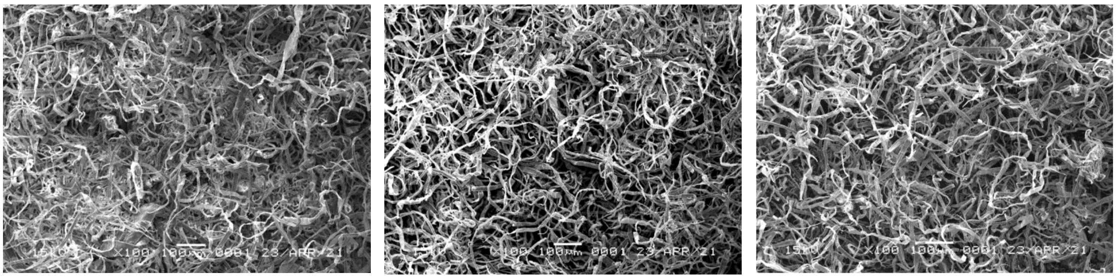


(a) (b) (c)

Figure 9.
SEM results of 2500X 15% NaOH with heating durations of (a) 2 hours, (b) 4 hours, and (c) 2 hours.

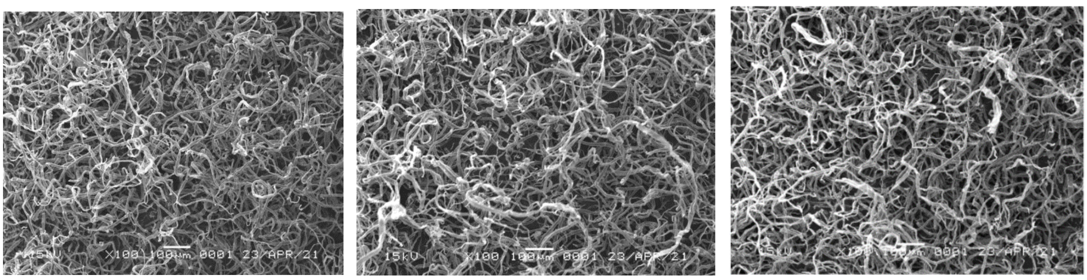
3.4. Energy Dispersive X-ray (EDX)

EDX testing was used for the characterization of nanoparticles [25]. EDX was used for qualitative mapping of chemical content and quantitative point analysis [26]. Figure 10 shows the coarser cellulose particles. In Figure 11 the cellulose particles appear smoother and Figure 12 shows the cellulose fibers getting finer, which means that the increase in the percentage of NaOH it is able to break down cellulose fibers [25]. The EDX test resulted in differences in the chemical content of the 5%, 10%, and 15% NaOH concentration variants with a duration of 2 hours, 4 hours, and 6 hours. The results of the EDX analysis showed that there were elements of Na_2O, MgO, Al_2O_3, SiO_2, K_2O, CaO, TiO_2, MnO, Fe_2O_3, and SO_3 which were produced in each sample variant. The synthesis process can be seen in Table 2-4. It showed the various percentages of chemical constituents present in nanocellulose. For example, the percentage of NaOH 15% with a duration of 2 hours of heating produces 30.36% CaO of which in OPC there is approximately 60% CaO.



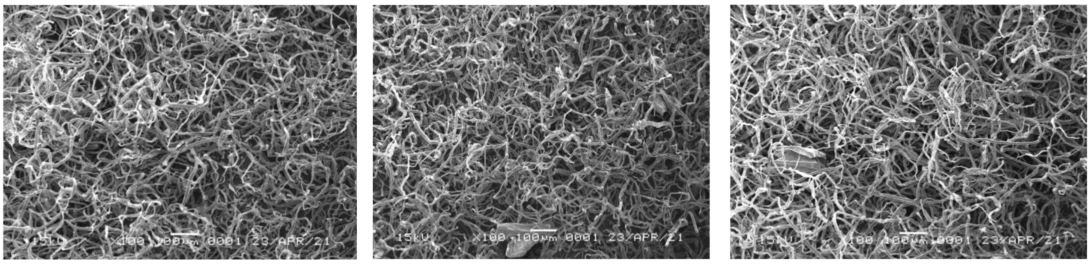
(a) (b) (c)

Figure 10.
SEM-EDX for 5% NaOH at (a) 2 hours (b) 4 hours, and (c) 6 hours.



(a) (b) (c)

Figure 11.
SEM-EDX for 10% NaOH at (a) 2 hours (b) 4 hours, and (c) 6 hours.



(a) (b) (c)

Figure 12.
SEM-EDX for 15% NaOH at (a) 2 hours (b) 4 hours, and (c) 6 hours.

Table 2.
Energy dispersive X-ray (EDX) Test Results for 5% NaOH.

Chemicals Content	5% NaOH		
	2 Hours	4 Hours	6 Hours
Na_2O	1.75	-	1.24
MgO	-	0.82	0.22
Al_2O_3	2.91	4.86	2.10
SiO_2	-	-	0.05
K_2O	0.91	0.97	-
CaO	87.28	89.10	96.38
TiO_2	-	-	-
MnO	-	4.25	-
FeO	7.15	-	-
SO_3	-	-	-

Table-3.
Energy Dispersive X-ray (EDX) Test Results for 10% NaOH.

Chemicals Content	10% NaOH		
	2 Hours	4 Hours	6 Hours
Na_2O	1.60	-	18.40
MgO	-	-	-
Al_2O_3	17.99	48.95	0.52
SiO_2	-	-	-
K_2O	-	-	-
CaO	23.92	21.33	24.92
TiO_2	-	6.50	2.23
MnO	38.93		35.21
FeO	17.49	23.22	18.73
SO_3	-	-	-

Table 4.
Energy dispersive X-ray (EDX) Test Results for 15% NaOH.

Chemicals Content	15% NaOH		
	2 Hours	4 Hours	6 Hours
Na_2O	-	-	31.99
MgO	0.67	8.83	-
Al_2O_3	6.64	35.35	1.85
SiO_2	-	2.10	-
K_2O	16.65	3.77	-
CaO	30.36	3.13	20.83
TiO_2	41.08	13.03	9.88
MnO	-	33.59	-
FeO	4.53	0.19	35.46
SO_3	-	-	-

4. Conclusion

In this paper, nanocellulose was produced from paper waste obtained from various places such as offices, schools, and photocopy shops, resulting in several conclusions, including:

- The XRD results showed that the amorphous content was around 79.68% at a 15% NaOH concentration with a heating duration of 6 hours and the lowest amorphous content was found in 5% NaOH with a duration of 4 hours, at 63.69%.
- In the FT-IR test, it can be concluded that chemical synthesis using NaOH and NaClO can remove the lignin and hemicellulose structures found in cellulose fibers.
- In the SEM test, it was seen that using a high percentage of NaOH, the cellulose fibers looked more biodegradable and clean.
- The EDX test described the characterization of nanocellulose using NaOH and NaClO containing chemical content close to that of OPC.

The analysis of the characteristics of nanocellulose resulted in the synthesis of nanocellulose using waste paper that was processed using NaOH and NaClO which was able to produce new environmentally friendly materials. For future research, adding more test methods that increasingly complement various variants of chemical concentrations should be explored. Additional research products can be tested with a mixture of concrete or mortar. Testing on concrete or mortar such as compressive strength, tensile, specific gravity, durability to chemical exposure, and permeability to water absorption should also be explored.

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