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Effect of Chemical Treatment on Characterization of Cellulose Nanocrystal Extracted from Banana Fiber: An Experimental Investigation

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ABSTRACT

Cellulose nanocrystal, known as CNC, is a form of material than can be produced by synthesizing carbon from naturally occurring substances, such as plants. In this study, CNCs were synthesized from banana pseudostem, using acid hydrolysis process. Cellulose was hydrolyzed by 65% H_2SO_4 at 50°C for 60 minutes following the raw material pretreatment, alkaline treatment, and bleaching processes. Fiber pre-treatment plays a significant role for its further application. The effects of cellulose pre-treatment on characterization of prepared CNCs were investigated. Alkaline treatment was conducted by extraction method using NaOH at different concentration such as 5% (CNC-1), 10% (CNC-2) and 15% (CNC-3). Isolated CNCs were characterized by X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FT-IR), Energy Dispersive X-Ray (EDX), and Scanning Electron Microscopy (SEM). Average particle size (in length) of 651 nm in CNC-1, 461 nm in CNC-2, and 48 nm in CNC-3 were found. The average diameter of the CNC was examined as 138 nm. Maximum crystallinity index of 62.27% was found for CNC-3. CNC-1 exhibited maximum water absorption of 51.68.

Keywords: Banana fiber, Cellulose Nanocrystals (CNCs), Chemical treatment.

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1. Introduction

In the last couple of decades, cellulose is gaining significant attention as biodegradability, environment friendly and low-cost additives of composite materials and shows promise in replacing petrochemical-based filler [1]. Among biomass, cellulose is the most abundant material globally. Plant cellulose serves as an effective natural reinforcement within appropriate polymer due to its significant mechanical, physical and thermal properties [2]. Usually CNCs are needle like particles with an average length of 100 nm [3]. CNC demonstrates several advantages compared to micro- and macro-cellulose composites, including cost-effectiveness, biodegradability, renewability, high surface reactivity, low density, and reduced energy consumption. Owing to its attractive properties, CNC has enabled in various applications including biomedical, optical, transparent membranes, and as nanoparticle reinforcement [4, 5]. Various literatures are available described the isolation of CNCs from various celluloses [6]. Acid hydrolysis is one of the most common techniques for isolation of CNC. Guo et. al. utilized H_2SO_4 for acid hydrolysis in preparing CNC from tea stalk [7]. Proper utilization of tea stalk has been reported along with provided theoretical guidelines for using tea stalk for preparing CNC. Wulandari et. al. isolated sugarcane bagasse CNC using 50% H_2SO_4 at 40°C [8]. Burhani et. al. isolated CNC from oil palm empty fruit bunches using strong acid (97% H_2SO_4) [9]. Minimum dimension of their prepared rod-loke CNCs were 43 nm in diameter and 147 nm in length. The XRD investigation revealed that crystallinity index of CNC was improved by 76.01%, compared with the cellulose. Cherian et. al. employed steam explosion process for the extraction of CNC from pineapple leaf fiber and resulted the isolation having a width in the range of 5-60 nm [10].

It is reported that, Banana farms generate around 220 tonnes of biomass waste per hectare. Banana Pseudostem is one of the vital parts of such wastes from which cellulose can be extracted. Significant researches have been conducted regarding the isolation of CNC from banana cellulose. Kumar et. al. extracted CNC from banana cellulose by oxidizing it with HNO_3-$NaNO_2$ mixture [11]. It is reported that the diameter of fiber reduced from microscale to nanoscale. Basumatary et. al. synthesized the CNC from banana rachis and found the particle size ~122 nm [12]. Zaini et. al. conducted the experiment to synthesize CNC where banana peel was the source of cellulose through 40% H_2SO_4 hydrolysis for 106.316 min and at a temperature of 77.02°C [13]. Crystallinity index was found 21.46% and a particle size was measured 152.6 nm. Sijabat et. al. obtained Bacterial Nanocellulose (BNC) from Nangka banana peel with a diameter of 30-50 nm and crystallinity index of 86.94% [14]. Pre-treatment of fiber played significant role in characterization of cellulose and synthesized further product by remove the hemicellulose and lignin from the fiber [15]. Characteristics of CNC depend on the raw materials used for their synthesis and reaction parameters during the acid hydrolysis. Reaction time, concentration and the temperature are the mostly investigated factors while studied of CNC. From various studies, it is found that the pre-treatment of fiber can be considered as the first step of CNC isolation. Alkalization is the most common fiber pre-treatment technique for further processing. Though different fiber pre-treatment methods other than alkalization were employed but the effects of various concentrations of NaOH on the CNC characterization are yet to be investigated. The objective of this study is to isolate CNC from banana pseudostem using acid hydrolysis technique for different concentrations of NaOH as the fiber pre-treatment chemical and the effects

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were investigated on particle size distribution, crystallinity index, surface morphology and water absorption characteristics of isolated CNCs. The article is organized as follows: Section 2 consisted with the experimental details. Section 3 composed with data reductions. Experimental results are analyzed in Section 4. Finally, outcomes of this study are concluded in Section 5.

2. Experimental

2.1 Materials

Raw materials for the cellulose preparation were collected from local area, in Chattogram, Bangladesh. Chemicals have been used in the study such as, Sulfuric acid (96.1% concentration), hydrogen peroxide, sodium hypochlorite (NaClO), acetic acid (100%), and sodium hydroxide (99%) were collected from Raj Scientific store, Chittagong, Bangladesh.

2.2 Preparation of cellulose

Banana stem is immersed in water for few hours in order to facilitate the removal of fibers. The split fibers are manually combed using a metal comb to remove any lingering traces of the pseudostem. The fibers are subjected to a process of rinsing with distilled water. Next, the collected fibers were dried using sunlight, depicted in Fig. 1.

2.3 Purification of cellulose

The fibers are cut to length of about 1 cm. Specific quantity of was submerged in hot distilled water (dH_2O). Temperature of the water bath was ranging from 70 °C to 80 °C in a water bath (Fig. 2). The cycle was finished within duration of 12 hours. Subsequently, the fibers were cleansed using water and separated using filtration. The specimen was subsequently dehydrated by placing it in an oven with the temperature of 60 °C for duration of 24 hours. This process was performed in order to remove contaminants and sizable particles, including foreign substances.

2.3 Pre-treatment of cellulose

Among various available cellulose pre-treatments, alkalization is used in this study as this process can be considered as the most common one. Banana cellulose was treated with three different concentrations of NaOH (5%, 10%, and 15%). The solid to liquid ratio was maintained as 1:50. A beaker was filled with a solution of NaOH, and then banana cellulose was introduced. The solution was incubated at 40 °C and stirred for 4 hours at 500 rpm. The solution underwent filtration, and the solid residue on the filter was rinsed several times with deionized water to attain a neutral pH. Resulting materials dried in the oven at 60 °C overnight. Entire process is illustrated in Fig. 3.

2.4 Bleaching process

The bleaching procedure was carried out by using NaClO (2%) at a temperature of 70°C for 2 hours with continuous stirring, all while maintaining acidic conditions with a pH range of 4 to 5. Concentrated acetic acid (2 mL) was introduced to modify the the acidity level. The ratio of solid to liquid was 1:10. The fiber's colour becomes lighter as a result of removing lignin and small amounts of hemicellulose. In contrast, the residue had a yellowish hue. The residual bleaching agent was separated from the fiber using centrifugation, and the fiber was thoroughly rinsed with distilled water. This procedure was repeated several times (5-7 times) until the pH level of the treated fiber reached 7. Prior to commencing the acid hydrolysis method, the treated fiber was subjected to drying at a temperature of 40 ˚C and thereafter stored. This storage allowed for the attainment of chemically purified cellulose (CPC). Fig. 4 depicted the bleaching procedure.

Fig.1 Banana Pseudostem (left) and extracted cellulose (right).

Fig.2 Hot water bath of banana cellulose.

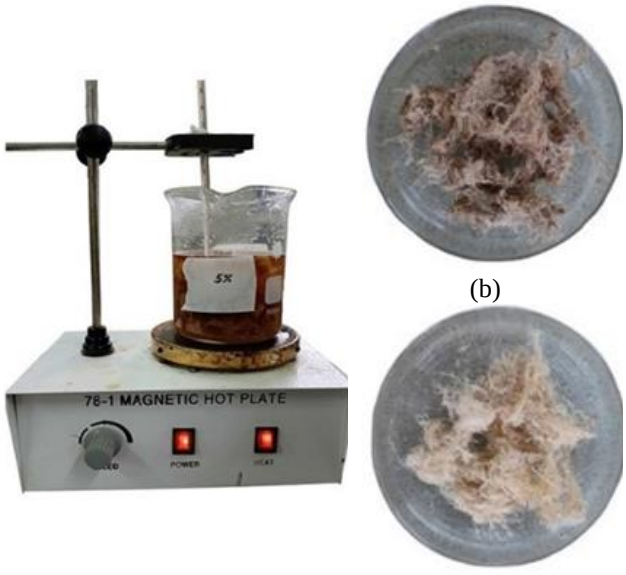


Fig.3 (a) Alkali Treatment at 40°C, Cellulose condition after treatment (b) 1st stage, (c) 2nd stage.

2.5 Isolation of Cellulose Nanocrystals (CNCs)

The dehydrated and whitened banana fiber produced from the aforementioned procedure was treated with acid hydrolysis. Sulfuric acid of 200 mL (65% v/v) was employed at 50 °C for a duration 1 hour, with a ratio of 1:20 g/ml. After duration of 60 minutes, the suspension underwent a hydrolysis process which was then halted by adding a

volume of cold DI water that was ten times more than the original suspension volume. The suspension was refrigerated overnight. The liquid portion was poured out and the acidity level of the solution was modified to a pH of 3 by several rinses with water and subsequent spinning with a force of 6000 times the acceleration due to gravity. During the subsequent phase, the suspension was maintained in distilled water for a period of 5-6 days, undergoing frequent filtering in order to get a pH level of 5. The resulting substance was dehydrated using an air oven and pulverised into a fine powder to acquire the CNC solid product. Fig. 5 showed the acid hydrolysis procedure and the isolated CNCs.

CNCs were prepared for different alkalization concentrations and termed CNC-1 (5% NaOH), CNC-2 (10% NaOH), and CNC-3 (15% NaOH) onwards.

3. Data Reductions

3.1 Fourier Transform Infrared (FTIR) Spectroscopy

The structural changes of banana cellulose through various chemical processes such as alkalization, bleaching, acid hydrolysis, etc. to CNC were studied by FTIR spectroscopy. The investigation was carried out using FTIR-8400 (Shimadzu). The samples have been dried at 60 °C. Then, the samples were mixed with potassium bromide. FTIR spectrum was recorded in the range of 4000–500 cm^{-1} (transmittance mode).

3.2 Scanning Electron Microscopy (SEM)

SEM was employed to examine the surface morphology of the prepared CNCs. Images have been captured for various magnifications. Analysis and the range of length and diameter of the prepared CNCs were measured by AURIGA, Zeiss, Germany.

3.3 Water absorption test

During this test, the specimens underwent immersion in distilled water, and their weight change percentages were tracked until they reached a state of equilibrium. Following this water immersion, samples were withdrawn after a 3-hour period, dried by gently wiping with filter paper to eliminate surface moisture, and then precisely weighed using an analytical balance with a resolution of 0.1 mg. The absorption of water was determined using the formula:

$$Q\ (\%) = \frac{W_2 - W_1}{W_1} \times 100 \quad (1)$$

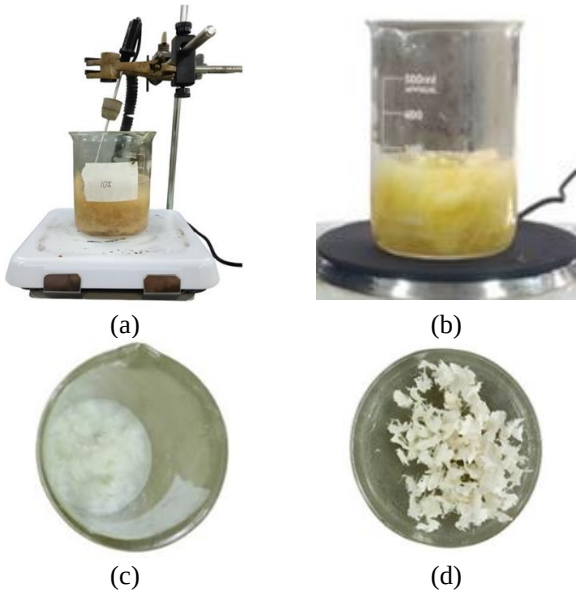
Here, 'W_1' represents the initial weight of the dry specimen, and 'W_2' is the weight of the specimen after absorbing water. The molar absorption is symbolized as 'Q'.

3.4 Crystallinity analysis

Isolated CNCs were examined to find out the crystallinity index. Such characterization was conducted through XRD analysis using XDR 7000, Shimadzu, Kyoto, Japan equipment. Operating voltage of 40 kV with Cu Kα radiation (l = 1.5406 Å) was maintained. The 2θ range of 10-80° for scanning was considered. The crystallinity index (Crl) was calculated using Segal's method as shown in Equation:

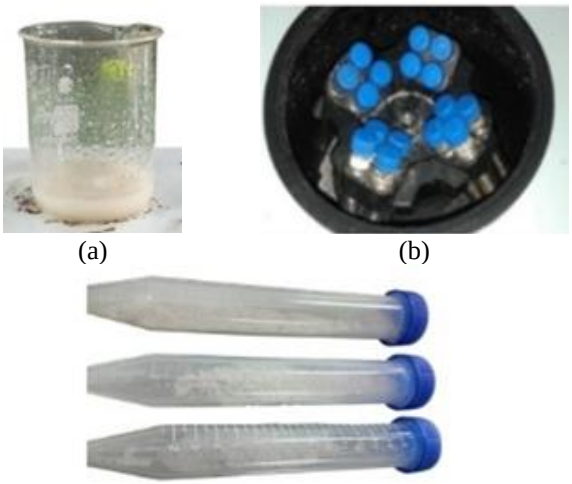
$$Crl(\%) = \frac{I_{200} - I_{am}}{I_{200}} \times 100\% \quad (2)$$

Where I_{200} is the maximum intensity of the diffraction at 200 peaks (2θ = 22.5°) and I_{am} is the minimum intensity of the diffraction at 2θ = 18.1°.



(a) (b) (c) (d)

Fig.4 (a) Bleaching treatment for 2h at 70° C, (b) Residue turned yellowish in color, (c) washed with neutral pH, and (d) oven dried chemically purified cellulose.



(a) (b) (c)

Fig.5 (a) Acid Hydrolysis, (b) Centrifugation, (c) Cellulose Nanocrystals (CNCs) for various alkalization concentrations stored in tube.

4. Result and Analysis

4.1 FTIR spectroscopy

The FT-IR spectra of CNC samples prepared from banana fiber is depicted in Figure 6. The broad peak has been observed in the range of 3500 to 3000 cm^{-1} and it can be said that it represented the -OH vibrational group of

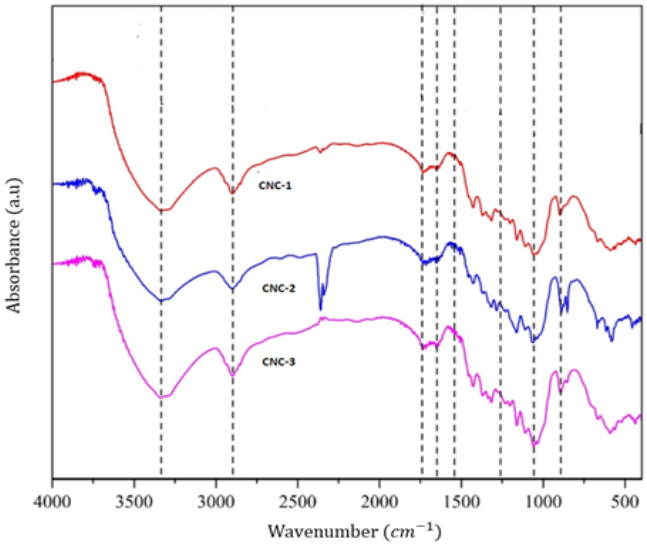
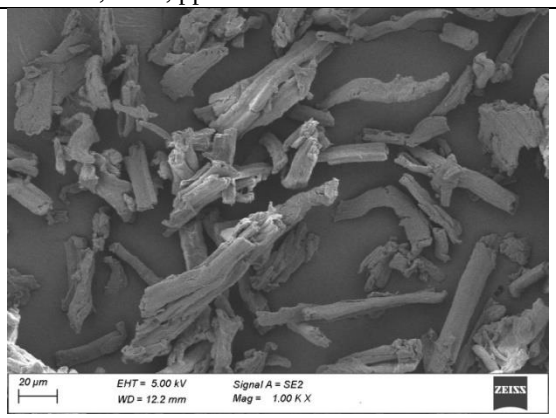


Fig.6 FT-IR spectra of banana fiber-based CNC

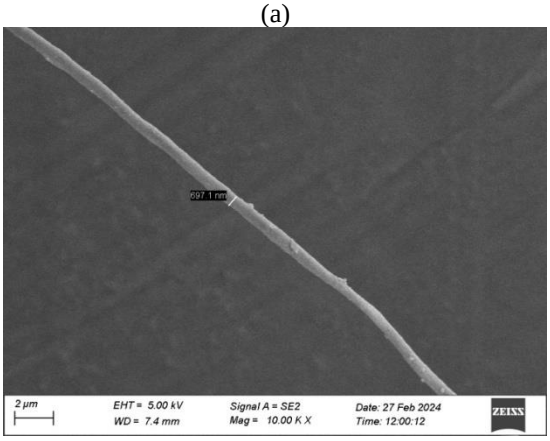
cellulose and the formation of hydrogen bonds between molecules. The hydrophilic properties of CNC were evidenced by the more pronounced peak in this range that occurred as the hydrolysis time increased, which reveals the presence of hydroxyl groups [16]. All samples kept dry to exclude the effect of -OH from water. The peak at 3226 cm^{-1} became more prominent when the cellulose underwent modifications in hydrogen bonding interactions, from CNC-1 to CNC-3 [17]. Increasing the duration of hydrolysis for the samples resulted in a more pronounced signal at 2783 cm^{-1}, indicating enhanced C-H bending and improved exposure of cellulose components [18]. Bleaching process was conducted to ensure the removal the hemicellulose and lignin. No peaks have been observed at 1667 cm^{-1}, 1458 cm^{-1}, and 1251 cm^{-1} in all CNC samples reveals the effectiveness of the process. A weak signal has been found at 1736 cm^{-1} represents the C=O stretching of the non-conjugated ketone, carbonyl, and ester groups. Due to the poor connection between lignin and hemicellulose, the peak at 97 cm^{-1} is also modest. Furthermore, the observed peaks at 1032 cm^{-1} and 876 cm^{-1} in the CNC synthesis can be ascribed to the stretching bond of C-OH and the symmetric glycosidic link of C-O-C, respectively. The peaks in question, as detailed in, provided information on both the crystalline structure and amount of cellulose [19]. The sulfonation peak is present in CNC-2 and CNC -3, but not in CNC -1. This may be attributed to the prolonged retention of sulfuric acid within the CNC subsequent to its reaction with banana fiber, resulting from a delayed hydrolysis process [20]. Based on the findings, the CNC samples maintained their initial morphological structure, and the greater peak intensity was attributed to the enhanced crystallinity of the cellulose.

4.2 Morphological analysis

The SEM images of the isolated CNC samples that demonstrate their morphology are displayed in Fig. 7. The SEM image of the CNC-1 sample reveals the presence of elongated structures with an uneven surface, which subsequently give rise to smaller, less consistent particles. The CNC-2 and CNC-3 samples exhibit rod-like characteristics, with a smooth surface and increased crystallinity. These traits are attributed to the high



(a)



(b)

Fig.7 SEM micrographs of CNC under different magnification (a) 1.00 Kx, (b) 10.00 Kx.

concentration alkali treatment, which results in well-defined nanostructures. The consistency in dimensions and form of CNCs is more noticeable at moderate levels of alkali concentration. The degradation of the fibers appeared to start at this time, as previously shown [21]. In addition, the CNC-3 sample had nanoparticles that were both brief and isolated from one another. These findings are in line with the outcome derived from the FTIR study. Extended reaction durations of sulfuric acid led to the disintegration of the CNC into shorter crystals and a decrease in hydrogen bonding within the cellulose chain, resulting in the formation of smaller CNC particles [22]. Particle distributions of the CNCs are depicted in Fig. 8. The mean particle size of the CNC samples decreased. The decrease in the size of the CNC particles was a result of eliminating more amorphous areas around the cellulose and breaking down the crystalline domains. Reduction in particle size of CNCs made them suitable for using various applications, i.e. drug carrier synthesis in diagnostic and therapeutic medicine, synthesis of medical and antimicrobial materials, and enzyme immobilization, etc. The EDX spectrums of the samples are showed in Fig. 9. The main components of CNC-1 are the carbon (C−41.66%) and oxygen (O−48.71%). Moreover sulfate groups have been observed in other peaks.

4.3 Water absorption characteristics

Water absorption percentages are demonstrated in Fig. 10. The value of water absorption was measured over several hours. After 3 hours of experimentation, the maximum water absorption was recorded at 51.58%, which was obtained for the sample with 5% NaOH. For the sample with 15% NaOH, the minimum water absorption was found to be 29.94%, and

for the sample with 10% NaOH, water absorption of 34.48% was recorded. The measured value for untreated banana cellulose oriented CNCs was 60%.

4.4 Crystalline structure analysis

Crystallinity index of the prepared CNCs are listed in Table 1. An increase in crystallinity was observed for CNC samples subjected to extended hydrolysis durations. This phenomenon can be attributed to the dissolution of amorphous regions encasing the crystalline components during acid hydrolysis, resulting in the liberation of individual crystals [22]. Improvement in the crystallinity could increase the tensile properties and the flexural characteristics [23]. Thus it can be said that the CNC produced through 15% NaOH fiber pre-treatment will provide better mechanical strength. Furthermore, it is observed that crystal size become lower with increasing alkali concentration and also hydrolysis time. XRD patterns for the various CNCs are illustrated in Fig. 11.

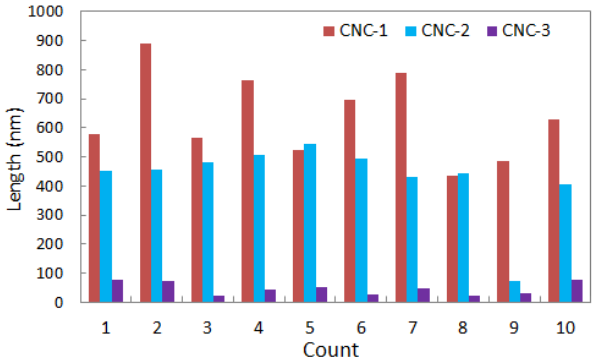


Fig.8 Particle distribution of banana fiber-based CNCs

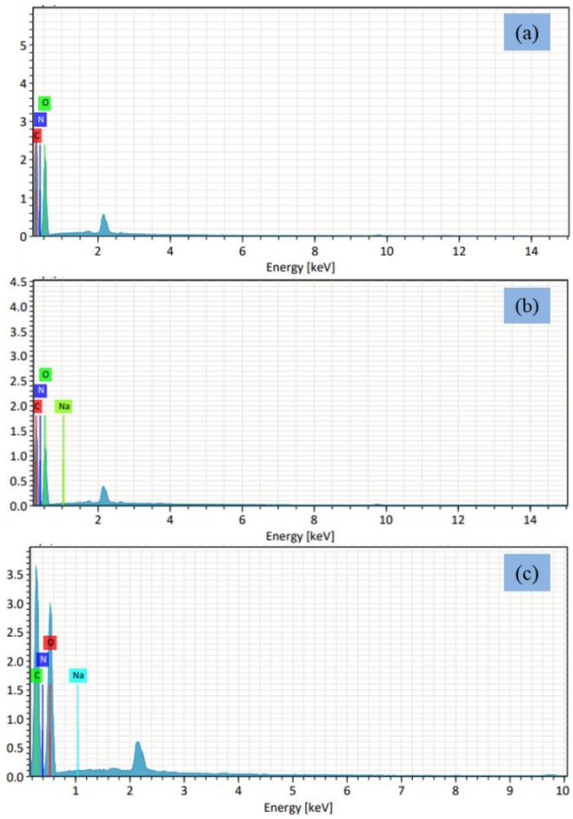


Fig.9 EDX spectrum of (a) CNC-1, (b) CNC-2; and (c) CNC-3.

Table 1 Crystallinity index of banana cellulose-based CNC

Specimen	Crystallinity Index (%)
CNC-1	52.83
CNC-2	57.62
CNC-3	62.27

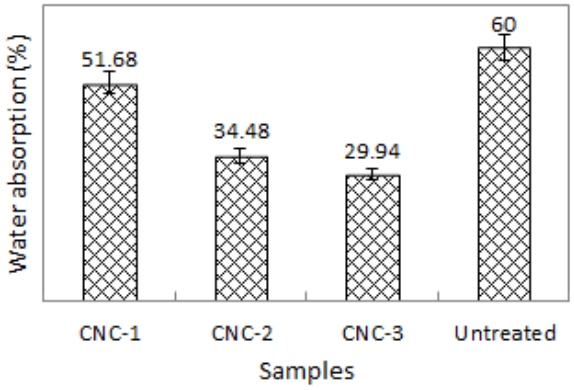


Fig.10 Water absorption characteristics of CNCs.

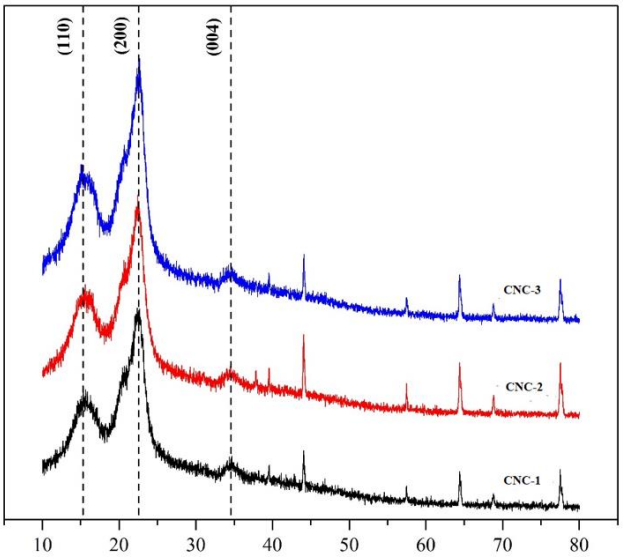


Fig.11 XRD patterns of CNCs for various concentration of NaOH.

This reduction in crystallite size can be explained by the acid-induced degradation of the cellulose layer, leading to the hydrolysis of smaller cellulose crystals [24]. This finding underscores the significant impact of chemical pre-treatment on the structural characteristics of isolated CNCs. It is reported that, with the decreasing

5. Conclusion

This study reports the acid hydrolysis process for isolating CNCs from banana cellulose and investigates the effect of alkalization with various concentrations of NaOH on its characterization. Acid hydrolysis method was employed to isolate CNCs using H_2SO_4 from pre-treated cellulose. Isolated CNCs were characterized by FTIR, XRD, SEM, and EDX. Morphological investigation confirmed rod-like CNC particle shapes. FTIR spectra showed that hemicellulose and lignin were efficiently removed from banana cellulose. The crystallinity of the CNC increased to 62.27%. Through this meticulously conducted experiment, it was determined that the water absorption properties of banana nanocellulose were significantly enhanced by 5%

NaOH treatment, in comparison to other chemical treatments. The pivotal role played by NaOH in modifying the inherent hydrophilicity of banana nanocellulose was underscored. Such investigation could provide insightful impact in the field of biomedical engineering, electrochemistry, electronic equipments, etc. Further investigation could be conducted on the influence of alkalization time and temperature on the characterization of isolated CNCs from banana cellulose.

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