

Bagasse Nanocellulose (*Saccharum Officinarum* L.): Process Optimization and Characterization

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ABSTRACT

This research aimed to determine the effect of sulfuric acid, the time of acid hydrolysis, and centrifuge rate in the synthesis process of nanocellulose from *Saccharum Officinarum* L. cellulose. Bagasse cellulose was isolated using three variations of acids, such as nitric acid, which was the mixture of sulfuric acid-nitric acid and the mixture of sulfuric acid-chloride acid. Cellulose, which was obtained from this process, was then hydrolyzed using sulfuric acid to produce nanocellulose. The process had used 40%; 45%; 50%; 55% and 60% of sulfuric acid concentration. The time variation of acid hydrolysis was 15'; 30' and 45'. The centrifuge rate of nanocellulose process was optimized to 1500; 3500 and 6000 rpm. Then nanocellulose formation was affected by sulfuric acid concentration, acid hydrolysis time, and centrifuge rate. The optimum sulfuric acid concentration was 50% and the hydrolysis time was 30' in temperature 80°C. Centrifuge rate had an important role in the nanocellulose particle separation process. At 1500 rpm, the colloid suspension could not be separated. On the other hand, at 3500 and 6000 rpm, the colloid suspension was separated. Variation of acids (nitric acid: sulfuric acid: chloride acid) in cellulose isolation process affected the size of nanocellulose. It was synthesized with sulfuric acids, such as 169.9 nm; 923 nm, and 1125 nm respectively. The result of FTIR spectrum analysis showed the disappearance of absorption at 1734 cm⁻¹; 1602.84 cm⁻¹; 1512.19 cm⁻¹; 1240.22 cm⁻¹ and 867.96 cm⁻¹ of area in nanocellulose spectrum after hydrolysis. It means that hydrolysis can remove lignin and hemicellulose from bagasse. The obtained crystallinity index of bagasse nanocellulose was 75.95%.

INTRODUCTION

Nanoscience and nanotechnology are the studies highlighted in many societies. Both scopes are research core currently noticed and developed. There is no doubt that nano research development will be a bridge between nanoscience and natural resources products. Nanotechnology plays a primary and important role in reviving the industry to a country with abundant resources [1][2].

Nanocellulose is a derivated cellulose in a nano-size [3]. It is produced by degrading the cellulose from various biomass sources like cassava bagasse [4], pineapple peel [5], and wastepaper

[6]. Nanocellulose is effective when it is isolated from pineapple peel waste juice to produce bacterial cellulose [7]. It is also suitable for corn [8]; palm oil mesocarp fiber [3]; potato peel waste [9]; palm sugar fiber [10]; sago palm [11], palm oil stem [12]; elephant grass [13]; sugarcane [14][15][16]; cassava peel [17] and wheat straw [18]. Nanocellulose comes from some sources, for example, algae, sea animals, cellulose bacterial, and microbial cellulose [19].

Nanocellulose is a biopolymer that is available in nature. It is a very plentiful and particularly important renewable biomaterial. The material can process many environment-friendly polymer products [20]. Cellulose in nanoscale is a remarkably interesting biomaterial because it has superior characteristics, such as high robustness and hardness in its structure, large surface area, non-toxic, combine with its lightness, biodegradable and renewable. Nanocellulose has a good combination of physic, mechanic, and biological characteristics. It has high biocompatibility, biodegradability, and low cytotoxicity. Industries are making an excellent nanocellulose to use in the pharmacy industry and to create a new biomaterial.[21].

Nanocellulose can remark through 2 approach models, such as bottom-up and top-down methods. In the bottom-up method, nanocellulose production will be done by biosynthesis, for example, fermentation that involves bacterial. In the top-down method, nanocellulose is created chemically by terminating and destructing the amorphousness area in cellulose [19]. Cellulose contains amorphousness and crystalline areas. The amorphousness area in cellulose has a lower density than the crystalline area. Also, it is more sensitive to acid.

Mechanic and or chemical destruction application involves acid hydrolysis, enzymatic treatment, high-pressure homogenization, and grinding. Synthesis routes from nanocrystalline cellulose production are giving three different types of nanocellulose, (1) stick cellulose microcrystal and whisker cellulose, (2) nanofibril cellulose including synonym form nano fibrillated cellulose and microfibrillated cellulose, also nanofiber cellulose, (3) bacterial cellulose including micro bacterial cellulose [19].

Chemical destruction of cellulose from biomass is producing nanocellulose, which is acid hydrolysis [21]. The used acid for hydrolysis is sulfuric acid [4][14][18][22], bromide acid [23], and chloride acid. Acid hydrolysis is the primary process to produce nanocellulose. Several factors that determine the character of nanocellulose related to acid hydrolyses are material sources, the type of acid, acid concentration for hydrolysis, hydrolysis reaction time, and the temperature [24].

Saccharum officinarum L bagasse is sugarcane bagasse residue that comes from the sugar industry. The quantity of waste bagasse is overflowing every year. One sugar factory can produce 35 – 40 % of grinding cane weight [25]. Bagasse contains 40-50 % of cellulose, most of which are in crystal form. Other components in bagasse are hemicellulose which is 25-35 %, and the other is lignin which is about 18-24 % [26]. Bagasse is potentially developed as biomass to produce nanocellulose.

This research described nanocellulose synthesized from bagasse cellulose with a variation of acid concentration, hydrolysis reaction time, and centrifuge rate. Also, we analyzed the nanocellulose FTIR spectrum characteristics and XRD graphic by the physical characteristics and their polydispersity.

METHOD

Materials

Saccharum officinarum L bagasse (from sugar industry in Kediri, East Java), Nitric Acid (Merck), Sodium Hydroxide (Merck), Sulfuric Acid (Mallincrodt USA), distilled water, and Hydrogen Peroxide (Sigma).

Sample Preparation

Saccharum officinarum L bagasse was sugarcane bagasse residue obtained from a sugar industry in Kediri, East Java, Indonesia. The quantity of 4 kg fresh bagasse was washed and dried, then was chopped and grounded to produce a dry powder. Then, the dry powder was sifted with a 100-mesh sieve to produce a fine powder. After that, a fine powder was kept in a closed container at room temperature.

Cellulose Isolation

Bagasse cellulose isolation was done through three steps. First, nitric acid had added to the bagasse with a ratio of 20 mL each gram. Bagasse solution was heated at temperature 800°C for two hours. After that, the residue from the first step was added with Sodium Hydroxide 2 N. The ratio was 20 mL each gram. Then, it was heated in the same condition. In the last step, the residue was added to hydrogen peroxide 10% at the same condition [27][28]. Cellulose that had been produced was neutralized with distilled water and controlled pH.

Nanocellulose Synthesis

Saccharum officinarum L cellulose was hydrolyzed using sulfuric acid with various concentrations, such as 40 %, 45 %, 50 %, 55 %, and 60 % [29][30] at temperature 800°C. The effect of hydrolysis reaction time was observed at 15 minutes, 30 minutes, and 45 minutes. Sulfuric acid was added to cellulose, then was heated as if the reaction time optimized. Then, the reaction was stopped. Distilled water was added excessively (controlled pH). The solution was centrifuged at various rates, such as 1500, 3500, and 6000 rpm. Nanocellulose was separated and dialyzed until pH was neutral.

Particle Size Analysis

Particle size was decided by PSA nano-micro techwithmeasurement range 0.3-10000 nm. The measurement was done to all the samples in optimized variation.

Particle Crystallinity Test

Particle crystallinity was tested by XRD. Theanalysis was done in peak area in diffraction graphic and crystallinity index value was calculated with this equation:

$$\text{CrI (\%)} = (\text{Sc/St}) * 100$$

where Sc is theCrystalline domain area and St is thetotal domain area. Thesample tested for XRD analysis was bagasse, bagasse cellulose, and bagasse nanocellulose.

Functional Group Identification

Theinfrared spectrum from each material was calculated in 4000 – 400 cm⁻¹ area using FTIR or Fourier Transform Infrared. Theanalysis was done to know the successfulness of hydrolysis to separate the lignin bond and hemicellulose to produce bagasse cellulose and nanocellulose.

RESULT AND DISCUSSION

Cellulose Isolation from *Saccharum officinarum L*

Saccharum officinarum L bagasse was lignocellulose biomass, where the main components were cellulose, lignin, and hemicellulose [1][31]. Bagasse contained 40-50% of cellulose, 25-35 % of hemicellulose, and other compounds [32][33][34]. The other component to form lignocellulose was a few ashes, waxes, and minerals[34][35].The treatments are varied. The researchers had conducted the treatments by adding the acid solution, nitric acid, and its combination.Lignocellulose may occur due to hydrolysis process to produce cellulose and lignin [36]. The addition of acid may terminate the intrachain bond between hemicellulose and cellulose in bagasse [37]. Hydrolysis occurred in the ester bond between hemicellulose and lignin. It can also appear in hydrogen bond between hemicellulose and cellulose [5].



Figure 1. Cellulose of *Saccharum officinarum L* Bagasse

The usage of alkali (NaOH) aimed to optimize the delignification process after the lignin-cellulose ether bond separation. It was the result of acid hydrolysis. The lignin-hemicellulose ether bond was weak and unstable to alkali consequently. It is easy to be disrupted by alkali. Free alkali and lignin might bond to create a lignin-alkali complex that could dissolve, makes it easier to remove. Sodium hydroxide also could dissolve hemicellulose and be removed. The step of cellulose isolation was ended with bleaching using hydrogen peroxide. Cellulose, from the three acid variations, was a white color powder. It was suitable with standard cellulose (Figure 1). The percentage of cellulose with a nitric acid solution was 41.400 %.

Nanocellulose Isolation from *Saccharum officinarum L* Cellulose

Sulfuric acid was added to cellulose fiber and heated at first to induce the fibers. It cut off the inter-fiber bonding longitudinally. The ion hydronium entered to amorphous area from the microfibrillar cellulose chain. It terminated the hydrolytic at glycoside bond to produce crystal cellulose in smaller sizes. The smaller size cellulose is nanocellulose. The amorphous area in microfibrillar cellulose was easy to be attacked by acid because it was looser. It would be a starter in cellulose degradation reaction with acid hydrolysis. On the other hand, the crystal area will be maintained by the complex structure that bonds tightly. Sulfation reaction produced crystal cellulose with the sulfuric ester in the OH group from crystal cellulose [38][19]. Polymerization degree decreased because the non-crystal area in microfibrillar cellulose will vanish when acid hydrolysis occurred [1]. Nanocellulose, which had produced, was a white colloid suspension that could be dried (Figure 2).



Figure 2. Nanocellulose of *Saccharum officinarum* L Bagasse

The Effect of Sulfuric Acid Concentration on Bagasse Nanocellulose Synthesis

Cellulose hydrolysis produced nanocellulose, and it was affected by several factors, such as hydrolysis temperature, hydrolysis time reaction, acid type and concentration, and centrifuge rate [24]. The short time reaction would produce cellulose with a higher polymerization degree because some fibers had not terminated. Concentrated acid could cause cellulose as it was hydrolyzed completely and produced monomer. Nevertheless, the lower acid concentration will produce cellulose with a higher polymerization degree. Hydrolysis with sulfuric acid could produce nanocellulose. The results were rich in negative sulfuric ester group and obtained from esterification condensed (sulfation). Sulfation occurred between OH and cellulose with sulfuric acid. Then, nanocellulose produced had dispersible characteristics as a colloid suspension in water [19].

Nanocellulose colloid particles had not dispersed completely at 40% concentration, compared to the 45% and 50% concentration with similar time reactions (Figure 3). The comparison was obtained from nanocellulose hydrolysis. On the other hand, there was no nanocellulose colloid suspension formed in hydrolysis time reaction of 30 minutes at 55% and 60% concentration. Cellulose might be hydrolyzed completely in concentrated acid. Hydrolysis stopped by adding distilled water excessively and then the process continued with centrifugation.

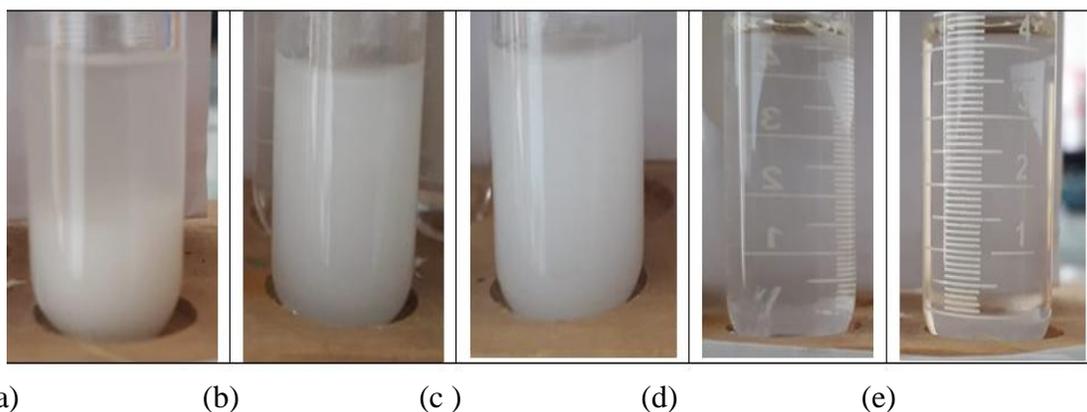


Figure 3. Hydrolysis results of cellulose *Saccharum officinarum* L with sulfuric acid at concentration

variations of 40% (a); 45% (b); 50% (c); 55% (d) and 60% (e).

The Effect of Acid Hydrolysis Time on Bagasse Nanocellulose Synthesis

Acid hydrolysis time optimization to nitrocellulose synthesis was 15 minutes, 30 minutes, and 45 minutes. The colloid suspension had formed at 15 minutes and 30 minutes hydrolysis time based on physical observation (Figure 4). The hydrolysis result at 45 minutes physically was clear or transparent. Turbidity level at 15 minutes hydrolysis time was more turbid than at 45 minutes. The long hydrolysis time (45 minutes) caused cellulose to hydrolyze almost completely to a smaller size or monomer formation.

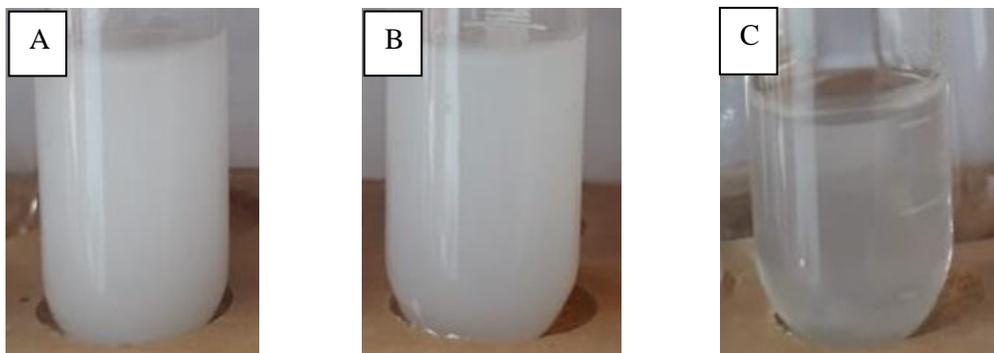


Figure 4. The effect of acid hydrolysis time on bagasse nanocellulose synthesis A) 15' B) 30' and C) 45'

The Effect of Centrifugation Rate on Bagasse Nanocellulose Synthesis

Centrifugation was done to all the hydrolysis results with 40-60% concentration. All the samples were not separate into two phases at 1500 rpm. Nanocellulose colloid could be separated well in nanocellulose hydrolysis result with 40%, 45%, and 50% sulfuric acid at 3500 and 6000 rpm. On the other hand, the compounds did not separate at 55% and 60% concentrations (Table 1). Sulfuric acid hydrolysis result with 55% and 60% concentration was clear and transparent. The homogeneity was larger than other results. The hydrolysis with higher acid concentration may produce smaller size nanocellulose and cause a termination of glycoside bond until its monomer formation was created.

Table 1. The Effect of Centrifugation Rate on Nanocellulose Separation

Rate (rpm)	Sulfuric Acid Concentration				
	40	45	50	55	60
1500	Not Separate	Not Separate	Not Separate	Not Separate	Not Separate
3500	Separate	Separate	Separate	Not Separate	Not Separate
6000	Separate	Separate	Separate	Not Separate	Not Separate

The Effect of Sulfuric Acid Concentration and Hydrolysis Time to Particle Size

Polydispersity index indicates the variation in particle distribution size. The high

polydispersity shows different particle sizes. Cellulose chain termination may cause nanocellulose formation that less uniform or less homogenous when acid hydrolysis ran suddenly [19]. Different particle sizes also caused aggregation between nanocellulose particles. Sulfuric acid hydrolysis results with 40%, 45%, and 50% concentration had good polydispersity indexes, such as 0.16, 0.37, and 0.24. It was based on polydispersity index measurement. The best polydispersity index was lower than 0.3 due to the effect of sulfuric acid concentration [5].

Nanocellulose has 160-400 nm length (L) and 20-30 nm diameter [16]. According to particle measurement result using PSA, bagasse nanocellulose particle could be obtained from sulfuric acid hydrolysis with 50% concentration and 30 minutes hydrolysis time which was 169.9 nm (Figure 6). On the other hand, the result of sulfuric acid hydrolysis with 40% and 45% concentration was micro cellulose with a size of 4.2 μm and 1.049 μm (Table 2).

Table 2. The Effect of Sulfuric Acid Concentration on Nanocellulose Size

No.	Sulfuric Acid Concentration (%)	Particle Size (nm)	PDI
1	40	4200	0.16
2	45	1049	0.37
3	50	169.9	0.24

Measurement and polydispersity index could not be performed at 45 minutes of hydrolysis time. There were no nanocellulose that can be separated. Nanocellulose had polydispersity index well at hydrolysis time 30 minutes with sulfuric acid concentration 50%. The particle size of this was 169.9 nm so it was called nanocellulose (Figure 5). Additionally, hydrolysis process at 15 minutes was not optimal. When the process stopped, the result was microcellulose (Table 3).

Table 3. The Effect of Hydrolysis Time on Particle Size

No	Hydrolysis Time (min)	Particle Size (nm)	PDI
1	15	1047	0.38
2	30	169.9	0.24
3	45	The colloid is not forming	-

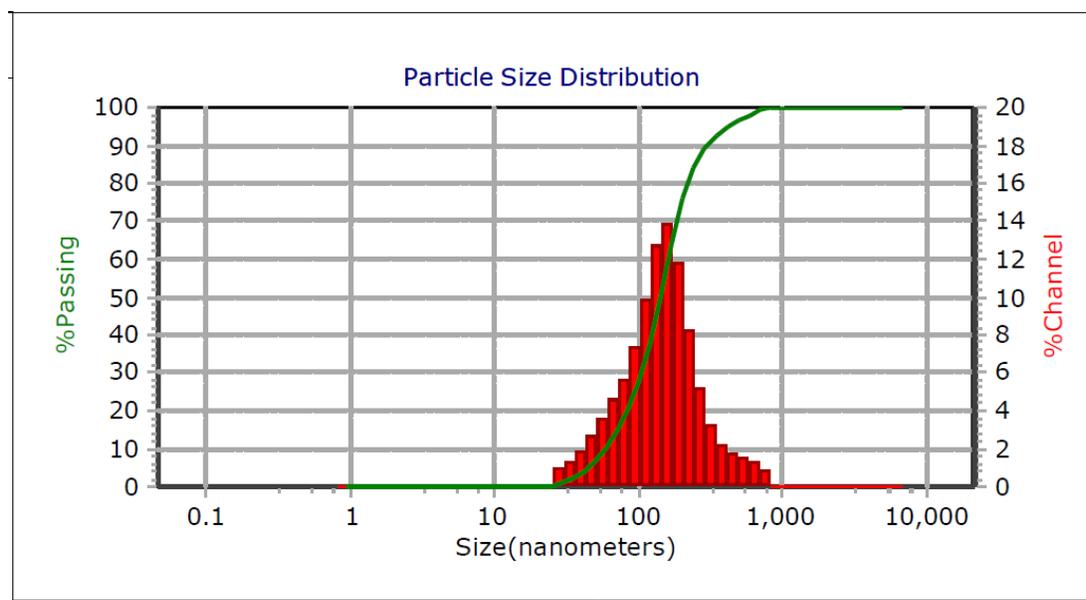


Figure 5. Particle size distribution of nano cellulose synthesized with sulphuric acid 50% (30 minutes)

FTIR Spectrum Analysis

FTIR spectrum for bagasse compared with FTIR spectrum for cellulose and nanocellulose had described different peak at 1734 cm^{-1} area and 1602.84 ; 1512.19 cm^{-1} ; 1240.22 cm^{-1} and 867.96 cm^{-1} (Figure 6 and Table 4). Nanocellulose infrared spectrum absorption was not differ with cellulose infrared spectrum absorption. Both showed that the delignification process and hemicellulose termination was done successfully.

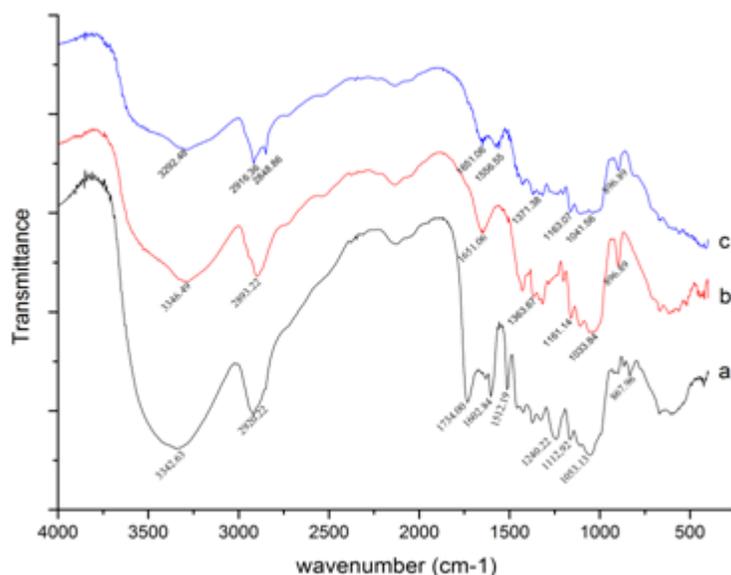


Figure6. FTIR spectrum of A) bagasse (without treatment), B) bagasse cellulose isolated with nitric acid, and C) bagasse nanocellulose

Table 4. Functional Groups Interpretation

Group	<i>Saccharum officinarum</i> L Bagasse	Cellulose	Nanocellulose
O-H	3342.63	3346.49	3292.48
C-H	2920.22	2893.22	2916.36 and 2848.86
O-H absorb water	-	2652.06	1651.06 and 1556.55
C-H bond and C-O	-	1363.67	1371.38
C-O-C	1112.92 and 1053.13	1161.14 and 1033.84	1163.07 and 1041.56
Glycoside Bond	-	896.89	896.89
C=O lignin and C-O lignin/hemicellulose carboxylate group	1734.00 and 1602.84	-	-
C=C from lignin aromatic ring	1512.19	-	-
Siringil group in lignin	1240.22	-	-
C-H bond in lignin	867.96	-	-

XRD Graphic Analysis

According to a value of 2θ that had required, the three samples, such as bagasse, cellulose bagasse, and nanocellulose bagasse, had a similar and unique crystal peak profile (around 12; 22 and 340) (Figure 7). The differences between the three peaks were the intensity that showed the three samples had different crystallinity. The relationship between crystallinity degree with a geometry aspect ratio ($1/d$) play a significant role as a parameter to control the characteristic of nanocellulose material. The crystallinity index values of bagasse, cellulose bagasse, and nanocellulose bagasse were 53.05%, 59.27%, and 75.95%. The hydrolysis treatment with acid had been proved to increase crystallinity.

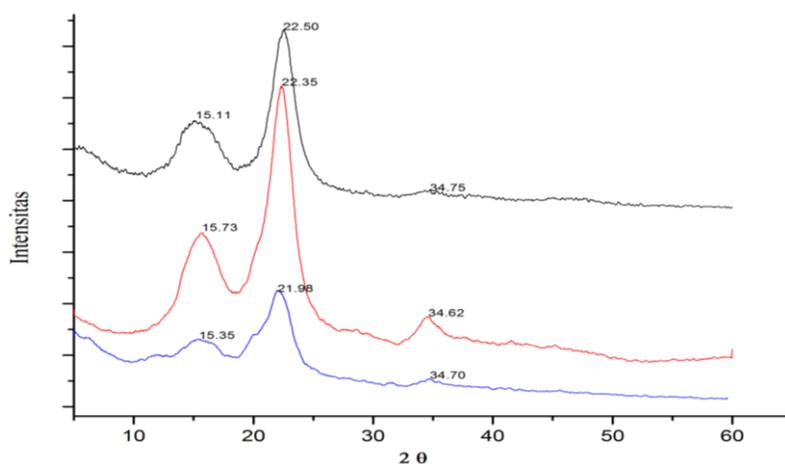


Figure 7. XRD Graph of *Saccharum officinarum* Bagasse (Blue line), Cellulose (Red line and Nanocellulose (Black line)

SUMMARY

The formation of nanocellulose is affected by sulfuric acid concentration, acid hydrolysis time, and centrifuge rate. The optimum sulfuric acid concentration was 50%, alongside hydrolysis time was 30 minutes in temperature 800 C. Centrifuge rate had an essential role in the nanocellulose particle separation process. At 1500 rpm, the colloid suspension could not be separated. On the other hand, at 3500 and 6000 rpm, the colloid suspension was separated. The result of FTIR spectrum analysis showed the disappearance of absorption at 1734 cm^{-1} area and 1602.84 ; 1512.19 cm^{-1} ; 1240.22 cm^{-1} , and 867.96 cm^{-1} in nanocellulose spectrum after hydrolysis. It explains that hydrolysis was a success at removing lignin and hemicellulose from bagasse. The Crystallinity index of bagasse nanocellulose that was obtained was 75.95%. The crystallinity index of nanocellulose higher than bagasse and cellulose.

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