The Effectivity of One-pot Concentrated Maleic Anhydride Hydrolysis for Betung Bamboo Pulp (*Dendrocalamus asper* sp)

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**Abstract :** Modification and synthesis of cellulose have attracted growing interest in the last decade due to applicable for many fields on advanced materials. Organic acid hydrolysis could be as an effective process for functionalization and size reduction of cellulose in one-pot reaction. The objective of this research is to study the effectivity of maleic acid anhydrous for functionalization and isolation of nanocellulose. Unbleached and Bleached Pulp of Betung Bamboo were used as cellulose resources. Concentrated maleic acid anhydride in 50% up to 60% (w/w) for 45 up to 90 minutes at 100 °C of reaction temperature were conducted to isolate cellulose nanocrystals from betung bamboo pulp. Fibrillation treatment on fiber solid residue using ultrasonication result micrometers scale less than 1 µm for unbleached (UBB) and bleached (BB) fibrillated cellulose. Functionalization of carboxyl groups is more effective on UBB than BB with surface charge up to -48.5 mV and specific groups of carboxyl are detected in 1716 cm⁻¹ and 1273 cm⁻¹. Thermal stability also has similar tendency by T onset in 313.69 °C and T max in 336.02 °C for UBB.

1. **Introduction**

One of the most potential lignocellulosic materials from nonwood source is Bamboo. Indonesia have more than 165 species of bamboo which naturally spread over the archipelago. Betung bamboo is one of the most abundantly planted in Indonesia, which commonly used as musical instrument, handcraft, structural, and textile so far. Betung bamboo consist of holocellulose (hemicellulose and alfa cellulose) about 83,33% and lignin about 30,21%, which could be a promising cellulose source [1]. High cellulose content of betung bamboo could be as an alternative substitution for wood cellulose which comercially produce during this time.

Microcellulose and nanocellulose demand are growing fast in the last decade which predict could reach USD 661.3 million in 2023, increase about 57% from 2018 with USD 284.7 million [2]. The driving force for this phenomena is environmental issue which encourage on sustainable material development. The excellencies of nanocellulose related on the properties which are good mechanical, chemical, physical, and optical that could improve the composite matric properties [3]. Therefore, because of the good performance, nanocellulose become widely develop nowadays especially for advance materials application such us medical, energy, waste water treatment, packaging, oil and gas refinery by further functionalization [4, 5]. Nanocellulose could be perfomed as cellulose nanocrystals
(CNC) with rod like morphology and cellulose nanofibril (CNF) with web-like appearance so have high aspect ratio depend on the isolation method [6].

The preparation for nanocellulose could be conducted by mechanical treatment, chemical treatment, oxidation, organosolv, and ionic liquid which might result different characteristics [6]. Mechanical treatments such as microfluidization, high pressure homogenization, grinding process could defibrilate and radial depolymerize which could produce high aspect ratio of CNF [7]. The effective method to isolate CNC is acid hydrolysis which could depolymerize the amorphous part of lignocellulosic materials and molecule diffusion in to cellulose that result crystalline nanocellulose (CNC). However, acid hydrolysis are use some mineral acids such as sulphuric acid, phosphoric acid, bromic acid, and nitric acid [8]. The disadvantages of the those mineral acids due to its corrosiveness that might high purification and after treatment cost. Furthermore, some weaknesses of sulphuric acid hydrolysis are sulphate groups that diffused in to cellulose limit for further process and low thermal stability because of attached sulphate groups [9]. Therefore, the use of organic acid should be study to minimize the use of minerals acid and to find eco-technology for nanocellulose isolation.

Some studies of nanocellulose isolation using organic acid have been done whether with dicarboxylic acid or tricarboxylic acid. Maleic anhydride and oxalic dihydrate could be as an effective acid and result in uniform nanocellulose which produce CNC and CNF in single process [9, 10, 11] with aspect ratio about 13.4 up to 27.3 for CNC and 92.5 up to 128.9 for CNF depend on acid concentration. [9, 10]. Citric acid, tricarboxylic organic acid, could produce CNC about 24% up to 32.2% by concentrated acid on 80% at high temperature [12,13]. Concurrent process in order to minimize the energy by one-pot reaction with slight catalyst have been done by Spinella et al. [14] and result CNC with yield about 20% with high carboxyl content.

As the best known of authors, limited information about nanocellulose isolation especially acid hydrolysis from unbleached pulp beside mechanical treatments. Lignin content on the unbleach pulp might inhibited acid diffusion to the cellulose, nevertheless nanocellulose could occurred in high sulfuric acid concentration for more than 64% [15], which further severe affect on purification steps. The only study of organic acid hydrolysis from unbleached wood pulp, with lignin content 17.2%, result high aspect ratio of CNF about 280 which potential for reinforcement agent. [10]. Furthermore, nanocellulose from unbleached pulp have higher decomposition temperature than bleached pulp [10, 15] that might be applied for wide range temperature of composites processes. However, nanocellulose which isolated by sulfuric acid have lower decomposition temperature about 200 °C [15, 16] than by organic acid due to sulphate groups.

\[
\text{Cellulose} + \text{Maleic anhydride} \rightarrow \text{Cellulose esterification}
\]

Figure 1. Esterification reaction of cellulose and maleic anhydride.

The motivation of this study is to find eco-technology for nanocellulose isolation. Therefore, the use of unbleached pulp is necessary as compliment for effective process. However, bleached pulp also use
for this study as references for commercial product. The objective of this research is to study the effectiveness of maleic acid anhydride to isolate nanocellulose from unbleached and bleached betung bamboo pulp. Single process of nanocellulose isolation and esterification could obtain by organic acid hydrolysis with predicted reaction as illustrated in figure 1.

2. Materials and Methods

2.1. Materials
Unbleached and bleached betung bamboo pulp were used as cellulose sources. Betung bamboo about 5 years old was cultivated from Bogor Botanical Garden than krafting pulped (sulphate method) in Research Center of Pulp and Paper Ministry of Industry, Bandung. Unbleached betung bamboo were composed by 87.5% of cellulose, 2.5% of hemicellulose, and 4.6% of lignin. Sodium chlorite, acetic acid, and maleic acid anhydride were purchased from merck, Indonesia. All chemicals were used as received without further purification.

2.2. Preparation of bleached betung bamboo pulp
Prior to hydrolyze, betung bamboo pulp was bleached for comparison on the effect of concentrated maleic anhydride hydrolysis. As much 10 g of oven dried unbleached betung bamboo pulp with moisture content about 78% was immersed on 600 mL of distilled water. Sodium chlorite was added for 16 mL than acetic acid for 0.8 mL. Bleaching process was conducted for 60 minutes at 80 °C with manual stirring every 15 minutes. Bleached pulp was stored at 4 °C for further process.

2.3. Nanocellulose Isolation
Organic acid hydrolysis using maleic anhydride was conducted by reflux process. Maleic acid anhydride was diluted in conditioned distilled water at 80 °C. Concentration of maleic anhydride were 50% and 60% (w/w). The ratio of oven dried unbleached pulp (UBB) and bleached pulp (BB) to organic acid solution was 1 : 10. Pulp was then hydrolyzed in the reaction flask under temperature at 100 °C for certain reaction time. After acid hydrolysis, UBB and BB at certain ratio were precipitated with 200 mL of cold distilled water then vacuum filtered to separate hydrolyzate (unreacted organic acid) and restrained solid residu. Subsequently, restrained solid residue were centrifuged on 10,000 rpm for 15 minutes for several times until neutral with pH about 6.2-6.5. Solid residue were sonicated for 45 minutes to obtain fibrillated cellulose. Sample were coded as present in table 1.

| Sample code | Maleic anhydride concentration (%) | Reaction time (minutes) |
|-------------|-----------------------------------|------------------------|
| UBB_45_50   | 50                                | 45                     |
| UBB_60_50   | 50                                | 60                     |
| UBB_90_50   | 50                                | 90                     |
| UBB_45_60   | 60                                | 45                     |
| BB_45_50    | 50                                | 45                     |
| BB_60_50    | 50                                | 60                     |
| BB_90_50    | 50                                | 90                     |
| BB_45_60    | 60                                | 45                     |

2.4. Yield and solid content
Yield of cellulose was measured using gravimetry method. Dried cellulose after freeze drying process were calculated by ratio of sample weight before and after treatment as perform in equation 1.

\[
\text{Yield} = \frac{\text{sample weight after hydrolysis}}{\text{initial weight}} \times 100\%
\]  

(1)

2.5. Dispersion stability and zeta potential analysis
Dispersant stability could be as an indicator on effectivity for size reduction and functionalization. Stability of the ultrasonicated nanocellulose was observed for 10 minutes, 24 and 48 h. Surface charge of treated nanocellulose was analyzed using Horiba Nano Partica sz-100 at ambient temperature. The concentration of solution was determined in 0.1 g/L.

2.6. Morphology
Morphology characterization was observed using Scanning Electron Microscope (SEM) Jeol JSM 5310-LV. Prior to analyze, isolated nanocellulose was freeze dried for several days to remove water content. After drying, samples were placed on metal pan than gold coated before observation. Scanning electron wave was conducted in 20 kV.

2.7. Chemical component
Chemical component of nanocellulose were analyzed using FTIR-UATR Spectrum Twoo Perkin Elmer. Sample was placed on reflector than scanned on wavelength 400 – 4000 cm\(^{-1}\). Scanning was conducted for 15 scans.

2.8. Thermal analysis
Thermal stability was analyzed using Thermal gravimetry analysis (TGA) 4000 Perkin Elmer. As much as 8 mg of sample was placed on porcelen crucible. The thermal analysis in TGA furnace was conducted under nitrogen flow condition in 40 mL/minutes. Before characterization, sample was holded at 25 °C for 1 minutes than heated from 15 °C up to 500 °C with heating rate at 10 °C/minutes.

3. Result and Discussion
3.1. Yield and solid content
The principal of acid hydrolisis on nanocellulose isolation is to depolimerize amorphous part (i.e lignin, hemicellulose) of lignoselulosic materials. Hemicellulose could be depolimerizer in to sugar (i.e glucan, xylan, mannan) so that could keep crystalline part of cellulose. Organic acid could produce both CNC and CNF in single route of process [9,10,12]. Hydrolisis process would produce cellulose nanocrystals (CNC) and fiber solid residue (FSR), than solid residu could be produce cellulose nanofiber (CNF) by mechanical treatment. Unbleached pulp (UBB) which contain lignin, hemicellulose, and cellulose has lower yield of solid residu than bleached pulp (BB) as present in table 2. These result possibly incorporate with higher hemicellulose of UBB which depolimerized in to sugar than BB. Bleaching process might be decomposed hemicellulose, so could affect for overall yield. Based on the yield data, concentrated maleic acid hydrolysis could not be as effective as sulphuric acid for nanocellulose isolation because of lower acidic level (pKa) which is about 1.9 than sulphuric acid which in -3. These result different from Bian et al, 2017 [10] that reported CNC yield isolated by organic acid hydrolysis from unbleached kraft wood pulp with low lignin content about 3.9% was reached 5.94% higher than bleached kraft wood pulp which in 3.02%. while higher lignin content up to 17% give lower yield due to protection of lignin for cellulose. Some factors affect the hydrolysis process such us cellulose characteristics and optimum reaction conditions which might be different between wood pulp and betung bamboo pulp. Table 2 shown that acid concentration more affected for yield of UBB than
reaction time, similar to sulfuric acid hydrolysis which are more effective to isolate nanocellulose on high concentration up to 64% for unbleached pulp because of lignin could inhibit the acid diffusion [15].

Table 2. Yield of FSR and microfibrillated cellulose characterization

| Sample       | Yield (%) | Solid content (g/L) | Surface charge (mV) |
|--------------|-----------|---------------------|---------------------|
| UBB_45_50    | 77.16     | 0.23                | -48.5 ±0.2          |
| UBB_60_50    | 77.63     | 0.50                |                     |
| UBB_90_50    | 78.81     | 0.60                |                     |
| UBB_45_60    | 61.39     | 0.47                |                     |
| BB_45_50     | 97.40     | 0.40                | -38.7 ±0.1          |
| BB_60_50     | 81.97     | 0.40                |                     |
| BB_90_50     | 64.55     | 0.40                |                     |
| BB_45_60     | 65.78     | 0.47                |                     |

Ultrasonication process could be as a defibrillation treatment to produce fibrillated cellulose from FSR. Solid content of fibrillated cellulose on the supernatant is present in table 2. Solid content of UBB samples were decreased by longer reaction time, while BB samples were tend to be constant. UBB which still consist of some lignin were easier to deaggregate than BB in low power level of sonication wave, about 40%. Amorphous structure of UBB might be facilitate the sonic wave to diffuse among particles. Cavitation from US wave could break down the cell wall structure of lignin and disolve in the solution [17]. Furthermore, higher acid concentration result in higher solid content both for UBB and BB. These result could be corelated with smaller particles which resulted in higher acid concentration.

3.2. Dispersion stability and surface charge

Dispersion of supernatant in regular times could be indicated the reduction of cellulose size and substituion of carboxyl groups. Figure 2 represent the dispersion stability of cellulose from treated UBB and BB. Treated UBB samples were turbid after 10 minutes of ultrasonication than become translucent after 24 h and still have same appearance after 48 h as shown in figure 2 (a,b), while UBB_45_60 with higher acid concentration result turbid solution even in 24 h than less turbid after 48 h. Contrary, treated BB result in turbid after 10 minutes than translucent at 24 h and less translucent after 48 h as seen in figure 2 (d,e,f). Acid concentration up to 60% clearly affect on dispersion stability of treated UBB and BB, which might be correspond to higher solid content of supernatant as represent on table 2. These phenomena could be correlated with higher carboxyl content for translucent to turbid supernatant [12, 18]. Moreover, UBB has more stable particles than BB by reaction time and acid concentration comparison.

Zeta potensial analysis which indicate for surface charge of CNF is present in table 2 for treated UBB and BB. In the same hydrolysis condition, UBB_45_50 have higher negative surface charge which indicate that higher carboxyl groups exist in the solution than BB_45_50. In case of BB_45_50, the surface charge is -38.7 mV, as reported by Chen et al. [9] which result about -30.1 up to 31.3 mV with 50% of maleic anhydride concentration. Previous study reported by Bian et al. [10] with result surface charge about -46.8 mV for bleached and -42.1 mV for unbleached wood kraft pulp. The result might be different with this study where unbleached pulp result higher surface charge, -48.5 mV than bleached pulp, in this case could be considered because of different mechanical treatments and concentration. These result also emphasize for higher dipersion stability of UBB than BB, figure 2, because of higher carboxyl repulsion among particles of UBB.
3.3. Morphology

Resulted treated cellulose, UBB and BB, are in various diameters as shown in figure 3 (a,c). Figure 3 (b,d) in high magnification show that fibrillated cellulose could be obtained in less than 1 µm both for treated UBB and BB. In this case, treated cellulose is in micro scale that further could denote as microfibril cellulose (MFC). Nevertheless, less length of the MFC fiber would be resulted in low aspect ratio with separately fibers. Sonication process might be shorten the length of MFC, while diameter slightly reduce due to partial dissociation of cellulose [12].

Figure 2. Dispersion stability of functionalized cellulose from UB pulp with hydrolysis condition UBB_45_50 (a), UBB_90_50 (b) UBB_45_60 (c) and BB pulp with hydrolysis condition BB_45_50 (d), BB_90_50 (e), BB_45_60 (f).

Figure 3. SEM morphology analysis of treated UB_45_50 with magnification in 9000x (a), 30.000x (b) and treated BB with magnification in 9000 (c), 22.000x (d)
3.4. Chemical component

The compliance of esterification reaction which substituted hydroxyl group of cellulose with carboxyl group of maleic acid could be expected by FTIR analysis. Figure 4 (a,b) show the FTIR spectrogram for treated UBB and BB in various hydrolysis condition. Specific functional groups of cellulose are detected by stretching of hydroxyl groups in 3330 cm\(^{-1}\), stretching of C-H groups in 2900 cm\(^{-1}\), and β-glycosidic linkage in 896 cm\(^{-1}\) whether for treated UBB and BB. Carboxyl groups, which contribute for carboxylation, are detected in 1716 cm\(^{-1}\) and 1273 cm\(^{-1}\) for treated UBB, while carboxyl groups are detected in 1724 cm\(^{-1}\) for treated BB. Beside that, stretching O(C=O)CH in 1372 cm\(^{-1}\) is detected for treated BB, figure 4 (b), which correspond to maleic acid groups. Furthermore, absorbed hydroxyl groups, which corelate to absorbed water, are detected for BB and treated BB in 1644 cm\(^{-1}\). These result could be confirmed that BB are more hydrophilic than UBB that still have lignin content which are more hydrophobic than cellulose. Overall, beside amorphous part depolymerization, esterification process also occurred from maleic acid hydrolysis even in weak vibration in specific carboxyl groups because of maleic anhydride has low acidity level. Two carboxyl groups in UBB\_45\_60, figure 4 are confirm for higher turbidity on figure 2 (c) than others.

![Figure 4. FTIR Spectrograph of treated UBB and BB in various treatment of treated UB (a) and BB (b)](image-url)
3.5. Thermal analysis

Thermal stability of UBB and BB could be observed using TGA and DTG thermograph as shown in figure 5 and table 3. Thermal stability is corresponded to the decomposition of hydrocarbon materials in to char, tar, and gas against to increasing temperature. DTG analysis, figure 5 (b), show that whether untreated UBB and BB have two decomposition peaks, first area under 100 °C which correlated to absorbed water and second area between 300-400 °C which correspond to cellulose. Figure 5 (a) show that UBB have low decomposition area because hemicellulose could be firstly decomposed than cellulose and lignin, which confirm that overall FSR yield, table 2, lower than BB. Furthermore, UBB has the lowest decomposition temperature at 5% of weight loss (T5) at 122.18 °C. However, after acid hydrolysis T5 increase at 272.64 °C which correlated to hemicellulose degradation even T_onset and T_max decrease about 8%. T_onset and T_max, table 3, are decrease after maleic acid hydrolisis both for UBB and BB, however UBB_45_50 has higher value than BB_45_50. These result because of carboxyl groups formation by esterification process might decrease decomposition temperature of CNF [13]. Whereas, sulphate groups on nanocellulose isolated by sulphuric acid hydrolysis have T_onset about 200- 250 °C [16, 19], which are lower than nanocellulose isolated by organic acid hydrolysis. Lignin content in UBB facilitate for high thermal stability due to higher decomposition temperature than hemicellullose and cellulose.

Figure 5. TGA (a) and DTG (b) Thermogram of untreated and treated betung bamboo pulp

| Sample     | T_onset (°C) | T_max (°C) | T5 (°C) | T30 (°C) | Char residue at 450 °C (%)
|------------|--------------|------------|---------|----------|-----------------------------|
| UBB        | 341.91       | 367.67     | 122.18  | 363.96   | 21.05                       |
| UBB45_50   | 313.69       | 336.02     | 272.64  | 336.51   | 16.29                       |
| BB         | 334.99       | 365.11     | 298.79  | 359.7    | 17.65                       |
| BB45_50    | 308.69       | 330.08     | 264.75  | 331.89   | 16.41                       |

4. Conclusion

Concentrated maleic anhydride hydrolysis could be as an effective process for functionalization of cellulose but not for isolation of nanocellulose with maleic acid anhydride concentration up to 60% and reaction time up to 90 minutes. Fibrillation treatment on fiber solid residue using ultrasonication result micrometers scale less than 1 µm for unbleached and bleached fibrillated cellulose which denote as microfibrillated cellulose. One of the advantages by using unbleached pulp beside could cut off for bleaching process also maintain the thermal stability due to lignin content. Functionalization by carboxyl
groups also result in unbleached which surface charge up to \(-48.5\) mV and specific groups of carboxyl are detected in 1716 cm\(^{-1}\) and 1273 cm\(^{-1}\) so that has good dispersion stability. Functionalized unbleached pulp also give higher the \(\Delta H\) mal stability than bleached pulp with \(T_{\text{onset}}\) in 313.69 °C and \(T_{\text{max}}\) in 336.02 °C. All things considered, functionalized MFC from unbleached betung bamboo pulp is potential for reinforcement agent for non polar matric which could facilitate for better dispersion. This study suggest for further study especially to obtain the optimum condition for high yield CNC and CNF derived from non wood natural fiber.

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