

# Bukti korespondensi

## Paper

A Preliminary Study of Isolation and Characterization of Nanocrystalline Cellulose from Microcrystalline Cellulose by Acid Hydrolysis Process



Materials Science Forum


Submitted: 2021-11-19

Revised: 2022-01-04

Accepted: 2022-01-25

ISSN: 1662-9752, Vol. 1057, pp 11-18 © 2022  
Trans Tech Publications Ltd, Switzerland Online: 2022-03-31

# 1. Submission

1. Title ✓	Please note this information is in <b>read only status</b> . You can add comments only.
2. Edit Paper ✓	<b>Checklist</b> 
3. Edit Authors ✓	<b>Paper: A Preliminary Study of Isolation and Characterization of Nanocrystalline Cellulose from Microcrystalline Cellulose by Acid Hydrolysis Process</b>
4. Edit Keywords ✓	* <input checked="" type="checkbox"/> I am aware of the information on <u>Policy and Ethics</u> .
5. References ✓	* <input checked="" type="checkbox"/> I used personal login credentials to access my account on <a href="https://www.scientific.net">https://www.scientific.net</a> .
6. Copyright Agreement ✓	* <input checked="" type="checkbox"/> I downloaded the Word/LaTeX template from the Participant Home page and prepared the manuscript as given in the template.
7. Checklist ✓	* <input checked="" type="checkbox"/> I used <b>page settings A4</b> I only used the fonts <b>Times Roman (Times New Roman) and Helvetica (Arial)</b> for the entire document including special characters ( $\alpha$ $\gamma$ $\mu$ $\Omega$ $\geq$ $\pm$ $\bullet$ $\Gamma$ $\{11\}$ $0$ ) because using other fonts will cause incorrect display of text or may change a special character to a square. I wrote the paper in <b>English; I didn't use any other language (even in Figures)</b> .

## Template Manuscript Evaluation

### Reviewer's Information

E-Mail:	
Name:	

### Manuscript Information

Manuscript Number:	FP-023
Manuscript Title:	A Preliminary Study of Isolation and Characterization of Nanocrystalline Cellulose from Microcrystalline Cellulose by Acid Hydrolysis Process

### Evaluation Report

Kindly enter comments per section of the manuscript (**All comments should be detailed to provide adequate information for authors**)

General comment	<ul style="list-style-type: none"><li>- The manuscript already followed the template. Please fix some minor details.</li><li>- Use Fig. in the paragraph to refer Figure (Fig. 1, Fig 2, ...)</li><li>- Equations (refer with: Eq. 1, Eq. 2, ...) should be indented 5 mm (0.2"). There should be one line of space above the equation and one line of space below it before the text continues. (see guideline for details)</li><li>- Add a space under heading conclusion and acknowledgment</li></ul>
	<p>Change this citation sentence The hydrolysis process using sulfuric acid has been done by Yudhanto et al. (2018) isolated Agave cantala fiber using sulfuric acid with a concentration of 44 wt%. Litorado et al. (2020) isolated hemp fiber with variations of H<sub>2</sub>SO<sub>4</sub></p>

Methodology	-
Results	Units in tables should be given in square brackets [meV]. If square brackets are not available, use curly {meV} or standard brackets (meV).
Discussion	From figure 2 and the rest, change the size of font into 12
Bibliography/References	<p>- Remove the indentation and add a space between each reference, example:</p> <p>[3] G.R. Mettam, L.B. Adams, How to prepare an electronic version of your article, in: B.S. Jones, R.Z. Smith (Eds.), Introduction to the Electronic Age, E-Publishing Inc., New York, 1999, pp. 281-304.</p> <p>[4] R.J. Ong, J.T. Dawley and P.G. Clem: submitted to Journal of Materials Research (2003)</p>

## Manuscript Evaluation

### Reviewer's Information

E-Mail:	: n4wati@untirta.ac.id
Name:	Dr. Ir. Ni Ketut Caturwati, MT.

### Manuscript Information

Manuscript Number:	23
Manuscript Title:	<b>A Preliminary Study of Isolation and Characterization of Nanocrystalline Cellulose from Microcrystalline Cellulose by Acid Hydrolysis Process</b>

### Evaluation Report

Kindly enter comments per section of the manuscript (**All comments should be detailed to provide adequate information for authors**)

General comment	The Title is in accordance with the discussion
Introduction	fair
Methodology	Good

Others	-
Final Note	Accepted

Please rate the following: (1 = Excellent) (2 = Good) (3 = Fair) (4 = poor)

Originality	2
Contribution To The Field	3
Technical Quality	2
Clarity Of Presentation	3
Depth Of Research	2

### Recommendations

Kindly mark with an X


Accept As It Is	X
Requires Minor Corrections	
Requires Moderate Revision	
Requires Major Revision	

### Additional Comments


Please add any additional comments (Including comments/suggestions regarding online supplementary materials) if any.

Revisi paper pada halaman berikut

Bukti accept setelah revisi

 \*Status description

Show By:  
10 

PAPER TITLE	MODIFIED	COMMENTS	STATUS*	REVIEWS	PROGRESS
A Preliminary Study of Isolation and Characterization of Nanocrystalline Cellulose from Microcrystalline Cellulose by Acid Hydrolysis Process	2022-01-25 14:59	add	Accepted	2/2	

V

## Copyright Agreement

Title: **Broad Exposure to Science and Technology 2021**

The undersigned author has submitted a manuscript titled: "**A Preliminary Study of Isolation and Characterization of Nanocrystalline Cellulose from Microcrystalline Cellulose by Acid Hydrolysis Process**"

(the "Paper") to be published in one of the periodicals by Trans Tech Publications Ltd., Kapellweg 8, CH-8806 Baech, Switzerland (the "Publisher").

1. The author grants to the Publisher the exclusive rights to the Paper during the full term of copyright, the exclusive rights comprised in the copyright of the Paper, including but not limited to the right to publish the Paper and the material contained therein throughout the world, in all languages and in all media of expression now known or later developed, and to license or permit others to do so.
2. Notwithstanding the above, the author retains the right to:
  1. Make copies of the Paper for author's use in classroom teaching at author's institution only.
  2. Use, after publication, the entire Paper or its parts in a book by the author, or a collection of author's own work.
  3. Make copies of the Paper for non-commercial internal distribution within the institution which employs the author for individual use only.
  4. Use up to one third of figures and tables of the Paper and up to 150 words of text for any purpose.
  5. Make oral presentations using the material from the Paper excluding handouts.
  6. Publish an extended, updated or rewritten version which includes at least 30% of new research data in another periodical.
  7. Deposit the Paper (postprint or preprint) in non-open access institutional repository with a link to Publisher's website only and proprietary rights other than copyright, such as patent rights.
  8. Upload only abstract of the published paper on any open-access repository including social network with a link to Publisher's website or DOI.

The author agrees that all copies made under any of the above conditions will include a notice of copyright and a citation to the Paper.

3. In the case of a Paper prepared under U.S. Government contract, the U.S. Government may reproduce, royalty-free, all or portions of the Paper and may authorize others to do so, for official U.S. Government purpose only, if the U.S. Government contract so requires. A copy of the contract must be attached.
4. If the Paper was written as a work made for hire in the course of employment, the Paper is owned by the company/employer who must accept the Copyright Agreement. In this case, the Publisher hereby licenses back to such employer the right to use the Paper internally or for promotional purpose only.
5. The author warrants that the Paper is author's original work. If the Paper was prepared in co-authorship, the author is obliged to inform the co-authors of the terms of the Copyright Agreement and obtain the consent to accept it on their behalf. Author guarantees that the Paper has not been submitted or published elsewhere. (If excerpts from copyrighted works included, Author should have a written permission from the copyright owners and credit to the sources in the Paper.) The author also states that, to the best of their knowledge, the Paper contains no libellous or unlawful statements, does not infringe on the rights of others, or contain material or instructions that might cause harm or injury.

### *Note to U.S. Government Employees*

A Paper prepared by U.S. federal government employee as part of his/her official duties is called a "U.S. Government work" and is in the public domain in the United States; in such case, Paragraph 1 above applies only outside the United States. Please attach a copy of any applicable policy of the author's agency. If the Paper was prepared jointly, and any co-author is not a U.S. Government employee, it is not a U.S. Government work. That co-author should be delegated by the other co-authors to accept the Copyright Agreement. If the Paper was not prepared as part of the employee's duties, it is not a U.S. Government work.

The acceptance of the terms of the Copyright Agreement is mandatory during the submission of a manuscript via the online submission system

By the acceptance, author agrees to receive login information for author access on [www.scientific.net](http://www.scientific.net) and to receive information via email for future books and specials related to materials science.

This service can be cancelled at any time.

### **Author's own work**

**Ferriawan Yudhanto**

**2021-11-19**





# A Preliminary Study of Isolation and Characterization of Nanocrystalline Cellulose from Microcrystalline Cellulose by Acid Hydrolysis Process

Ferriawan Yudhanto<sup>1,a\*</sup>, Harini Sosiati<sup>2,b</sup>, Venditias Yudha<sup>3,c</sup> and Edi Syafri<sup>4,d</sup>

<sup>1</sup>Department of Mechanical Technology, Vocational Program, Universitas Muhammadiyah Yogyakarta, Daerah Istimewa Yogyakarta, Indonesia

<sup>2</sup>Department of Mechanical Engineering, Faculty of Engineering, Universitas Muhammadiyah Yogyakarta, Daerah Istimewa Yogyakarta, Indonesia

<sup>3</sup>Department of Mechanical Engineering, Faculty of Industrial Technology, Institut Sains & Teknologi AKPRIND, Daerah Istimewa Yogyakarta, Indonesia

<sup>4</sup>Department of Agricultural Technology, Politeknik Pertanian Payakumbuh, Sumatera Barat, Indonesia

<sup>a\*</sup>ferriawan@umy.ac.id (corresponding author), <sup>b</sup>hsosiati@ft.umy.ac.id, <sup>c</sup>venditias.y@akprind.ac.id, <sup>d</sup>edisyafri11@gmail.com

**Keywords:** Nanocrystalline cellulose, Acid hydrolysis process, Physical characterization

**Abstract.** This research focuses on the isolation of MCC (microcrystalline cellulose) into NCC (nanocrystalline cellulose) by acid hydrolysis process. The sulfuric acid hydrolysis (44 wt.% H<sub>2</sub>SO<sub>4</sub>) aims to fibrillation from MCC into NCC material. NCC has good properties such as high-surface-area, high-aspect-ratio, weight light, and reactive materials. The morphology of NCC was characterized by SEM (Scanning Electron Microscope) and TEM. The physical characterization was tested using FTIR, XRD, and TGA. The morphological result showed that the particle size of NCC was more homogeneous with a diameter size of 25±3 nm with 310±5 nm in length. The physical properties of NCC better slightly than MCC, indicated by the increasing crystallinity index value from 74.8 to 76.4%, and it has a high thermal resistance of 330°C.

## Introduction

In the last decade, the use of renewable material in nanosized was implemented by researchers as a filler composite material. The acid hydrolysis method is a common method used to extract nanocellulose from lignocellulose resources. The hydrolysis process using sulfuric acid has been done by isolated Agave cantala fiber using sulfuric acid with a concentration of 44 wt.% [1], isolated hemp fiber with variations of H<sub>2</sub>SO<sub>4</sub> concentration from 41-50 wt.% [2], and isolated Mulberry pulp with a 47 wt.% [3].

The hydrolysis process using sulfuric acid has been done by Yudhanto et al. (2018) isolated Agave cantala fiber using sulfuric acid with a concentration of 44 wt.%, Listyanda et al., (2020) isolated hemp fiber with variations of H<sub>2</sub>SO<sub>4</sub> concentration from 41-50 wt.%, and Reddy et al., (2014) isolated Mulberry pulp with a 47 wt.% [1-3]. The isolation aims to degrade microcellulose into nanocellulose through the fibrillation process. Nanocrystalline cellulose has a 10-25 nm diameter and a length of 100-500 nm [4]. Nano-sized NCC causes them to have a high aspect ratio and surface area values, so it is widely applied to nanocomposite films as reinforcement or filler [5, 6]. Another physical property is the high crystallinity index of NCC (nanocrystalline cellulose), which is an essential factor for the increased mechanical strength when applied as reinforcement or filler to nanocomposite films and nano-membranes with PVA (Polyvinyl Alcohol) matrix [7, 8].

The MCC is a purified, partially depolymerized cellulose from fibrous plant material with long crystalline polymer chains (C<sub>6</sub>H<sub>10</sub>O<sub>5</sub>)-n. The NCC was successfully extracted from commercial MCC

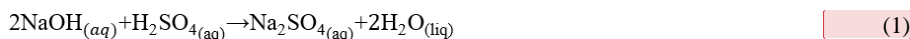
**Commented [f1]:** The citation sentence has been changed according to reviewer suggestion

(microcrystalline cellulose) powder by sulfuric acid hydrolysis in the present study. The main goal of this work focus on the morphological and physical properties of NCC. The raw material starts from MCC powder for shortening the purification process of lignocellulosic fiber, which takes a long time. The use of MCC is expected to speed up the process of obtaining the NCC.

## Experimental

**Materials.** The materials used for this study include MCC commercial was obtained with code 1.02330.0500, H<sub>2</sub>SO<sub>4</sub> (analytical purity), NaOH (analytical purity) obtained from Merck and Co., Inc. The commercial MCC has a diameter range of 10-20 μm. The morphological MCC and NCC were observed by SEM (Scanning Electron Microscope) dan TEM (Transmission Electron Microscope) to compare their size and shape. The Physical characterization was analyzed by FTIR (Fourier Transform Infrared), XRD (X-ray Diffraction), and TGA (Thermal Gravimetric Analysis) test.

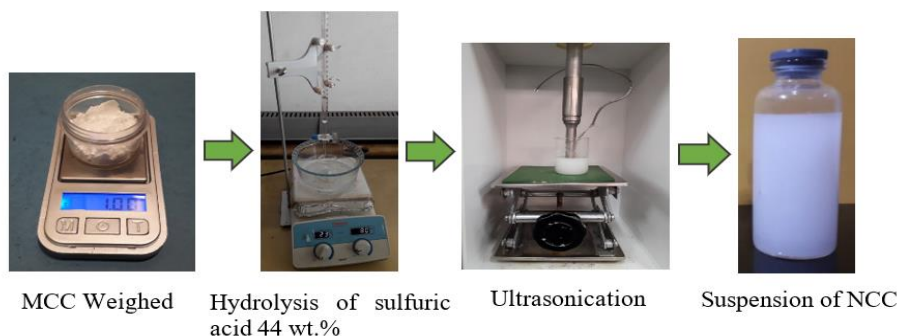
**Methods.** The MCC powder was weighed and put in a glass beaker filled with distilled water in a ratio of 1:50 by weight volume. Next, sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) is dripped into a burette tube with a concentration of 44 wt.%, slowly rotated using a magnetic stirrer with a constant rotation speed of 100 rpm for 1 hour with a preheat temperature of 60°C [9]. After the hydrolysis process, the NCC was then neutralized by adding Sodium hydroxide (NaOH) in an ice bath (5°C), namely salt-hydrolysis and continued neutralization using the centrifugation method was conducted at 4000 rpm for 15 minutes. Finally, the NCC suspension was rinsed with distilled water until pH neutral. The chemical reaction for stopping sulfuric acid is shown in equation 1.



**Commented [f2]:** Equations has been indented 5 mm (0.2"). It has been spaced above the equation and one line of space below.

The ultrasonication was used for the next fibrillation of the MCC with a power of 240 watt for 60 minutes to obtain NCC suspension. This NCC was centrifuged to obtain NCC suspension, the scheme of this research was shown in Fig. 1.

**Commented [f3]:** Use Fig. in the paragraph to refer Figure



**Figure 1.** Schematic flow of MCC isolation into NCC with sulfuric acid hydrolysis-ultrasonic process

## Characterization

**Morphology of MCC and NCC.** Morphology of MCC was observe by SEM JEOL type JSM 6510 with operating voltage range of 5-40 kV. The sample were coated with gold using sputtering technique. The nanosized of NCC was confirm by TEM (JEOL JEM-1400 series).

**FTIR Test.** FTIR analysis aims to detect possible changes in the functional group of the sample (MCC and NCC materials) in the wavenumber range of 500 to 4000 cm<sup>-1</sup>. The test equipment model

is IR Prestige-2 with a thin pellet-shaped test sample. The samples were mixed with KBr (1:100, w/w), after the sample was mixture then pressed into thin pellets that were analyzed.

**XRD Test.** XRD test is performed to analyze the CI (Crystallinity Index) of the materials. XRD patterns were collected by an X-ray diffractometer model Rigaku-miniflex 600 (40 kV, 15 mA). The instrument was operated at scanning rate of  $2^\circ/\text{min}$  from  $2\theta = 3-40^\circ$  with Cu  $K\alpha$  radiation ( $\lambda=1540$  nm). Calculation of the CI value following the Segal's empirical method [10], with the equation 2 as follows:

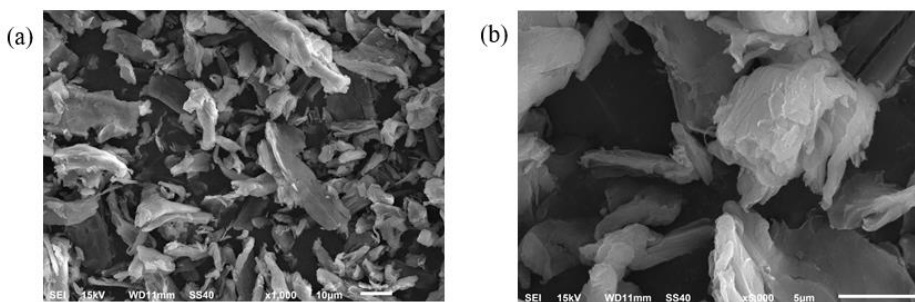
$$CI = (I_{002} - I_{\text{amorphous}}) / I_{002} \quad (2)$$

The maximum intensity of crystalline from plane 002 at  $2\theta = 22.5^\circ$  was noted  $I_{002}$  and  $I_{\text{amorphous}}$  is the intensity of diffraction attributed to amorphous cellulose. The higher CI was indicated that cellulose crystallinity is better.

**TGA Test.** The TGA test aims to degradation of MCC and NCC materials because of high temperature, the analysis obtained in the form of  $T_{\text{onset}}$  (the initial temperature of degradation) and  $T_{\text{max}}$  (maximum temperature of degradation). Thermalgravimetric analysis was carry out using a Mettler Toledo. Each sample (MCC and NCC) was heated from temperature of  $30^\circ\text{C}$  to  $600^\circ\text{C}$  under nitrogen atmosphere at heating rate of  $10^\circ\text{C}/\text{min}$ .

## Results and Discussion

**Morphology of MCC and NCC.** The morphology of MCC powder was shown in Fig. 2a and 2b, an irregular length and shape with a range diameter of 10-20  $\mu\text{m}$ . The combination of chemical (acid hydrolysis) and mechanical (ultrasonication) treatments aims to change the dimension from micro into nano scale. The NCC has shaped like a long needle shape. NCC looks like a homogeneous single crystal with a higher aspect ratio value than MCC. The average diameter and length obtained were of  $25 \pm 3$  nm and  $310 \pm 5$  nm, respectively. The comparison of length and diameter is called the aspect ratio (L/D), which is 12.4. These results were observed from TEM photos, as shown in Fig. 3a. The results of the diameter and length distribution of NCC were then calculated using image-J software, as shown in Fig. 3b. This homogenous nano-size increases bind with nanocellulose as reinforcement with the polymer matrix. The addition of NCC into thermoplastic polymers such as PLA (Polylactic acid) or PVA is expected to increases the mechanical strength of bio-plastic films.



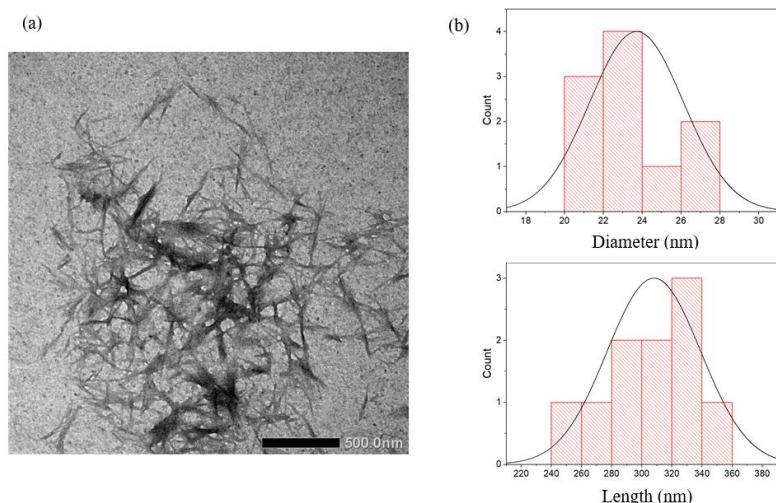
**Figure 2.** SEM Photo of MCC of (a) 1000 and (b) 5000 x magnification

**Commented [f4]:** Equations has been indented 5 mm (0.2"). It has been spaced above the equation and one line of space below.

**Commented [f5]:** Use Fig. in the paragraph to refer Figure

**Commented [f6]:** Use Fig. in the paragraph to refer Figure

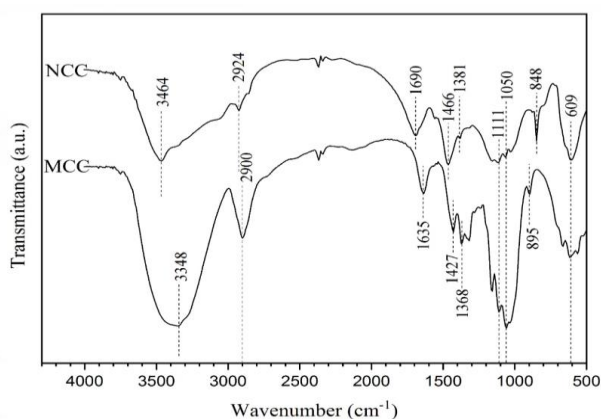
**Commented [f7]:** Use Fig. in the paragraph to refer Figure



**Figure 3.** Nanocrystalline Cellulose (a) TEM photo, (b) Distribution of diameter (D) and length (L)

**FTIR analysis.** Fig. 4 shows the FTIR spectra obtained from MCC and NCC materials. There are offered three leading region bands, in the range of 3348-3464  $\text{cm}^{-1}$ , 2900-2924  $\text{cm}^{-1}$ , and 1050-1111  $\text{cm}^{-1}$  [10]. These three areas vibration of the intramolecular bonds stretches such as the O-H compound, C-H bending, and C-O stretching pyranose ring group, a natural cellulose constituent [11]. The absence of wavenumber 1739  $\text{cm}^{-1}$ , 1371  $\text{cm}^{-1}$  and 1250  $\text{cm}^{-1}$ , indicates disappeared residual acetate group elements (hemicellulose and lignin) in the NCC material [12,13]. The change of band located at 1630  $\text{cm}^{-1}$  to 1690  $\text{cm}^{-1}$  was shows hydrophilicity properties. The water content in cellulose has been reduced due to the loss of hemicellulose content. The band located at 1427-1466  $\text{cm}^{-1}$  was H-C-H bonds or  $\text{CH}_2$  symmetric bending indicating cellulose type II. The cellulose type I showed at the peaks of 895  $\text{cm}^{-1}$ , 1050  $\text{cm}^{-1}$ , and 1111  $\text{cm}^{-1}$ , changes in the peaks of these wavenumbers indicate a difference in the regeneration of crystalline cellulose I to cellulose II [14]. It is also supported by the appearance of new peaks in the spectrum of NCC material.

**Commented [f8]:** Use Fig. in the paragraph to refer Figure



**Figure 4.** FTIR Spectra of MCC and NCC materials

**XRD Analysis.** The crystallinity index affects the mechanical strength of the composite when used as a reinforcement in the polymer matrix. The cellulose, which contains impurities such as lignin and hemicellulose, has a CI value of 45-80%. Another crystallinity index of raw materials for several types of plant fiber is shown in Table 1. The higher value of crystallinity index for natural fibers, the crystalline cellulose, indicates a good quality fiber.

**Table.1** Crystallinity Index of various raw natural fibers

Raw Fibers	Crystallinity Index [%]
Ramie [2]	79.75
Agave cantala [9]	64.50
Agave sisalana [17]	79.32
Agave angustifolia [18]	59.00
Agave americana [19]	50.10
Bamboo [20]	45.57
White straw [21]	54.42
Salacca midrib [22]	62.40

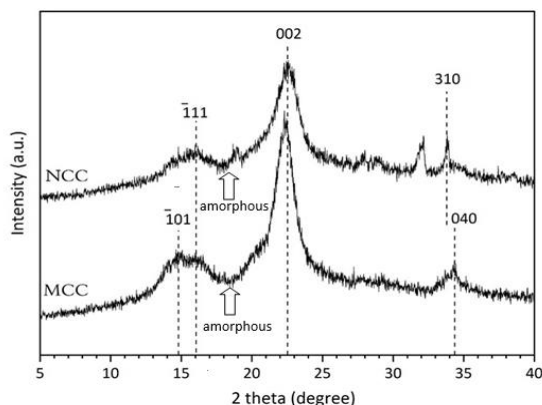
**Commented [f9]:** Units in tables have been given in square brackets [ ]

Fig. 5 shows XRD patterns of MCC and NCC. The crystallinity index calculates using Segal's equation and the data was presented in Table 2. The CI result indicated that NCC increased slightly by 2.2% after the acid hydrolysis process. The diffraction peaks at around  $2\theta = 16^\circ, 22.5^\circ,$  and  $34.2^\circ$ , from the JCPDS#030289 for native cellulose representing the [111], [002] and [040] crystallographic planes of typical cellulose type I [24]. A high-crystallinity index of NCC increases the intermolecular bond of the OH- (hydroxyl) group in the NCC polymer chain. Thus, it impacts strengthening the intramolecular bond between NCC and the polymer.

**Commented [f10]:** Use Fig. in the paragraph to refer Figure

The NCC isolated from natural fibers has been widely applied to the manufacture of membranes and nanocomposite films as a reinforcement in the PVA (polyvinyl alcohol) matrix. For example, Rochardjo et al. (2021) used the NCC isolated from ramie fiber as filler to reinforce the PVA matrix to manufacture nano-membranes by the electrospinning process. The addition of NCC in the membrane can increase the tensile strength and elongation break by 112% and 50%, respectively [8].

PVA nanocomposite film material reinforced with NCC isolated from Agave cantala increased tensile strength and elongation break by 76.7% and 138%, respectively [15]. The NCC suspension, which high-crystallinity and high-surfaces area, causes an excellent intramolecular bond with the PVA polymer [16].



**Figure 5.** XRD diffraction patterns of MCC and NCC materials on the  $2\theta = 5^\circ-40^\circ$

**Table 2.** Comparison of crystallinity index of MCC and NCC materials

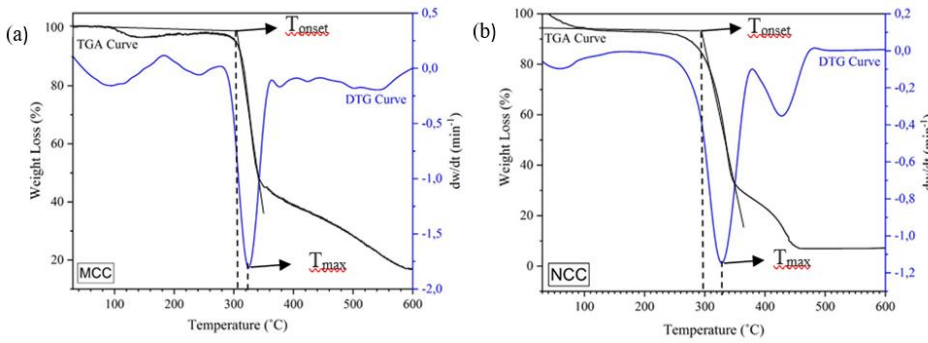
Material	I <sub>amorphous</sub> [cps]	I <sub>002</sub> [cps]	CI [%]
MCC	786	198	74,8
NCC	573	135	76,4

**Commented [f11]:** Units in tables have been given in square brackets [ ]

**TGA analysis.** Fig. 6a shows the TGA (thermal gravimetric analysis) and DTG (differential thermogravimetric analysis) curves of MCC material. It shows the initial ( $T_{onset}$ ) and maximum ( $T_{max}$ ) degradation temperature values. The  $T_{onset}$  for NCC decreased about 10°C lower than MCC, and it indicated that a bit of sulfate ion still reacted on the surfaces of NCC. This statement is supported by Listyanda et al. (2020) using ramie fiber as NCC with variation time of hydrolysis process. A more extended interaction between negative sulfate ion and nanocellulose can reduce thermal stability due to dehydration reaction [23]. The  $T_{max}$  values between MCC and NCC did not make a significant difference. The  $T_{max}$  of NCC show that it is 5°C higher than the MCC material (Fig. 6b). That thermal condition indicated that the sulfate ion in the NCC suspension has been successfully removed by combining salt-hydrolysis and centrifugation processes on the cold temperature. The detailed thermal stability showed in Table 3.

**Commented [f12]:** Use Fig. in the paragraph to refer Figure

**Commented [f13]:** Use Fig. in the paragraph to refer Figure



**Figure 6.** TGA and DTG curve of (a) MCC and (b) NCC materials

**Table 3.** Initial and maximum degradation temperature of MCC and NCC material.

Material	$T_{onset}$ [°C]	$T_{max}$ [°C]
MCC	305	325
NCC	295	330

**Commented [f14]:** Units in tables have been given in square brackets [ ]

## Conclusion

Nanocrystal cellulose has been isolated from commercial MCC with acid hydrolysis to distinct the morphology and physical properties. The acid hydrolysis process for a concentration of 44 % wt. for an hour and preheat 60°C successfully decreases the diameter and length of NCC to 25±3 nm, 310±5 nm, respectively. Moreover, the NCC has good physical properties by high-value crystallinity index (76.4%), high-aspect ratio (12.4), and high-thermal resistance (330°C). NCC's properties make this material potentially used as filler to reinforced nanocomposite.

**Commented [f15]:** A space under heading conclusion has been added



## Acknowledgement

This research was carried out under research grants of Tri Dharma Improvement Program for Higher Education of LP3M, Universitas Muhammadiyah Yogyakarta (UMY). Contract No. 550/PEN-LP3M /II/2020, Batch 1.

Commented [f16]: A space under heading Acknowledgement has been added

## References

- [1] Yudhanto F., and Heru SB Rochardjo, Application of taguchi method for selection parameter bleaching treatments against mechanical and physical properties of agave cantala fiber, IOP Conference Series: Mater. Sci. and Eng. Vol. 352. No. 1. IOP Publishing, (2018).
- [2] Listyanda, R.F., Kusmono, Wildan, M.W. and Ilman, M.N., Extraction and characterization of nanocrystalline cellulose (NCC) from ramie fiber by sulphuric acid hydrolysis, AIP Conf. Proc. Vol. 2217. No. 1. AIP Publishing LLC, (2020).
- [3] Reddy, J.P. and Rhim, J.W., Characterization of bionanocomposite films prepared with agar and paper-mulberry pulp nanocellulose, Carb. Poly. 110 (2014): 480-488.
- [4] Kusmono, K., A preliminary study of extraction and characterization of nanocrystalline cellulose (NCC) from ramie fiber, J. of Mater. Process. and Char. 1.1 (2020).
- [5] Frone, A.N., Panaitescu, D.M., Donescu, D., Spataru, C.I., Radovici, C., Trusca, R. and Somoghi, R., Preparation and characterization of PVA composites with cellulose nanofibers obtained by ultrasonication, BioRes. 6.1 (2011): 487-512.
- [6] Fortunati, E., Puglia, D., Luzi, F., Santulli, C., Kenny, J.M. and Torre, L., Binary PVA bionanocomposites containing cellulose nanocrystals extracted from different natural sources: Part I." Carb. poly. 97.2 (2013): 825-836.
- [7] Sultana, T., Sultana, S., Nur, H.P. and Khan, M.W., Studies on Mechanical, Thermal and Morphological Properties of Betel Nut Husk Nano Cellulose Reinforced Biodegradable Polymer Composites, Jour. of Comp. Sci. 4.3 (2020): 83.
- [8] Rochardjo, H.S., Fatkhurrohan, A.K. and Yudhanto, F., Fabrication of Nanofiltration Membrane based on Polyvinyl Alcohol Nanofibers Reinforced with Cellulose Nanocrystal using Electrospinning Techniques, Int. Jour. of Tech. 12, 2 (2021).
- [9] Yudhanto, F., Jamasri and Rochardjo, H.S.B., Physical and thermal properties of cellulose nanofibers (CNF) extracted from agave cantala fibers using chemical-ultrasonic treatment, Int. Rev. of Mech. Eng. 12 (2018): 597-603.
- [10] French, A.D. and Cintrón, M.S., Cellulose polymorphy, crystallite size, and the Segal crystallinity index, Cellulose 20.1 (2013): 583-588.
- [11] Xie, J., Hse, C.Y., Cornelis, F., Hu, T., Qi, J. and Shupe, T.F., Isolation and characterization of cellulose nanofibers from bamboo using microwave liquefaction combined with chemical treatment and ultrasonication, Carb. Poly. 151 (2016): 725-734.
- [12] Kargarzadeh, H., Ahmad, I., Abdullah, I., Dufresne, A., Zainudin, S.Y. and Sheltami, R.M., Effects of hydrolysis conditions on the morphology, crystallinity, and thermal stability of cellulose nanocrystals extracted from kenaf bast fibers, Cellulose 19.3 (2012): 855-866.
- [13] Fatkhurrohan, Rochardjo, H.S.B., Kusumaatmaja, A. and Yudhanto, F., Extraction and effect of vibration duration in ultrasonic process of cellulose nanocrystal (CNC) from ramie fiber, AIP Conf. Proc. Vol. 2262. No. 1. AIP Publishing LLC, (2020).
- [14] Yang, Y.P., Zhang, Y., Lang, Y.X. and Yu, M.H., Structural ATR-IR analysis of cellulose fibers prepared from a NaOH complex aqueous solution, IOP conf. series: mat. Sci. and eng. Vol. 213. No. 1. IOP Publishing, (2017).

Commented [f17]: References have been change according to MSF template

- [15] Yudhanto, F., Jamasri, J., Rochardjo, H. and Kusumaatmaja, A., Experimental Study of Polyvinyl Alcohol Nanocomposite Film Reinforced by Cellulose Nanofibers from Agave Cantala, *Int. Jour. of Eng.* 34.4 (2021): 987-998.
- [16] Yudhanto, F. and Rochardjo, H.S.B., Physical and Mechanical Characterization of Polyvinyl Alcohol Nanocomposite Made from Cellulose Nanofibers, *Mater. Sci. For.* Vol. 988. Trans Tech Publications Ltd, (2020).
- [17] Sosiati, H., Muhaimin, M.M., Wijayanti, D.A. and Triyana, K., Microscopic characterization of cellulose nanocrystals isolated from sisal fibers." *Mater. Sci. For.* Vol. 827. Trans Tech Publications Ltd, 2015.
- [18] Rosli, N.A., Ahmad, I. and Abdullah, I., Isolation and characterization of cellulose nanocrystals from Agave angustifolia fibre, *BioRes.* 8.2 (2013): 1893-1908.
- [19] Krishnadev, P., Subramanian, K.S., Janavi, G.J., Ganapathy, S. and Lakshmanan, A., Synthesis and Characterization of Nano-fibrillated Cellulose Derived from Green Agave americana L. Fiber." *BioRes.* 15.2 (2020): 2442-2458.
- [20] Das, M. and Chakraborty, D., Influence of alkali treatment on the fine structure and morphology of bamboo fibers, *Jour. of App. Poly. Sci.* 102.5 (2006): 5050-5056.
- [21] Kaushik, A., Singh, M. and Verma, G., Green nanocomposites based on thermoplastic starch and steam exploded cellulose nanofibrils from wheat straw, *Carb. Poly.* 82.2 (2010): 337-345.
- [22] Yudha, V., Rochardjo, H.S.B., Jamasri, J., Widyorini, R., Yudhanto, F. and Darmanto, S., Isolation of cellulose from salacca midrib fibers by chemical treatments, *IOP Conf. Series: Mat. Sci. and Eng.* Vol. 434. No. 1. IOP Publishing, (2018).
- [23] Listyanda, R.F., Wildan, M.W. and Ilman, M.N., Preparation and characterization of cellulose nanocrystal extracted from ramie fibers by sulfuric acid hydrolysis, *Heliyon* 6.11 (2020).
- [24] Rochardjo, H.S.B., Jamasri, Yudhanto, F., Extraction of Natural Fibers by High-Speed Blender to Produce Cellulose Sheet Composite, *Int. Rev. of Mech. Eng.* 13.12 (2019): 691-699.