Preparation and Characterization of Nanocrystalline Cellulose from Cassava Stem Wastes by Electromagnetic Induction

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Abstract
Cassava stems were one of the largest agricultural by products in Indonesia, especially in Lampung Province. It is known that cassava stems have a fairly high lignocellulose content, especially cellulose which reaches 39.29%. The high cellulose content in cassava stems has great potential to be used as raw material for Nanocrystalline Cellulose (NCC). The preparation of nanocrystalline cellulose consists of four main stages, namely: pre-hydrolysis, delignification, bleaching, and acid hydrolysis. The pre-hydrolysis stage was carried out by boiling a solution of CH3COOH and cassava stem powder for 60 minutes at a temperature of 105°C. Cassava stem powder was then delignified using a 25% NaOH solution heated to a temperature of 105°C for 1 hour. The bleaching stage used a 3.5% NaOCl solution at a temperature of 50°C for 60 minutes and was carried out twice. The last step is acid hydrolysis using 2.5N HCl solution for 15 minutes at a temperature of 105°C, then the electromagnetic induction treatment is varied with temperature variations of 30°C, 50°C, and 70°C for 60 minutes. The prepared nanocrystalline cellulose were tested for lignocellulose, XRD and PSA. From the test results, the best variation of nanocrystal cellulose preparation was an acid hydrolysis treatment with 70°C electromagnetic induction for 60 minutes, namely an increase in the percentage of cellulose 62.93%, crystallinity 90.68%, and an average particle size of 18.04µm with some particles measuring nanometer sizes. From the results of the research, it was concluded that electromagnetic induction increased crystallinity and decreased the size of nanocrystalline cellulose.

Keywords: cassava stems, nanocrystalline cellulose, α-cellulose, acid hydrolysis, electromagnetic induction

1. Introduction
Nanotechnology is currently an interesting topic for academics, industry and research institutions as a method of waste management, especially the use of biomass waste. Biomass waste contains natural polymer fibers in the form of crystals (microcrystals or nanocrystals) called cellulose. With nanotechnology, nanocrystals in cellulose have the potential to be used as fillers, binders, and disintegrants in tablets and capsules, as well as being useful as viscosity enhancers (Rowe et al., 2009, Peng et al., 2011). In general, nanocrystals are nanocellulose with a diameter of 5-20 nm (Samir et al., 2005) with a length of hundreds of nanometers, which consists of many crystalline parts.

The main substances for obtaining nanocrystalline cellulose were plants and their waste, but the use of plant waste maximizes the utilization of these plants and has a good impact on the environment. One of the plant wastes that is abundant in Indonesia and has a high cellulose content is cassava stem waste. The abundance of cassava stems is caused by the high production of cassava in Indonesia. Based on data from the Central Statistics Agency (BPS, 2018) the total production of cassava in Indonesia is 19.341.233 tons. Meanwhile, production in Lampung Province reached 6.683.758 tons. Cassava stems contain 39.29% cellulose, 24.34% hemicellulose, and 13.42% lignin (Lismeri et al., 2016). In another study, Sumada et al. (2011) obtained data that cassava stems contain 56.82% α-cellulose, 21.72% lignin, 21.45% Acid Detergent Fiber (ADF), and 0.05-0.5 cm long fiber.

Nanocrystalline cellulose in cassava stems could be obtained by using the hydrolysis of α-cellulose using strong acids, for example...
HCl and H$_2$SO$_4$. There have been many studies on isolation using various methods to produce nanocrystalline cellulose. Some research on nanocrystalline cellulose are: based on research on the isolation and characterization of nanocrystalline cellulose by Arjuna et al. (2018), the best results were obtained with a crystallinity index of 78.01% with a crystal size of 58.91 nm. Another research conducted by Sumiati et al. (2016), in this study, the best results were obtained using an HCl concentration of 8.75 ml/g with a crystallinity degree of 74%.

Research on the synthesis and characterization of microcrystalline cellulose from cassava stem waste has been carried out by Lismeri et al. (2020), in this study, the purity of α-cellulose was 99.68% and the crystallinity of cellulose microcrystals was 78.12%, but the size of the microcrystalline was still quite large and brown in color.

Based on this research, the authors are interested in making modifications to the research method that has been carried out by Lismeri et al. to obtain a better quality of crystalline cellulose. The modification of the method that the author will do is to reduce the size of the raw material from 16 mesh to 120-200 mesh and add electromagnetic induction to the hydrolysis process.

Electromagnetics was a tool that used changing an electric field into a magnetic field so that it could be used in the manufacture of nanocrystalline cellulose. The presence of a magnetic field results in an increase in molecular activity, causing the grouping of molecules to split (Fuhaid et al., 2011). It is hoped that the electromagnetic can weaken the bonds of these molecules so that they can be more easily broken so that it will reduce the energy used.

2. Methodology

2.1. Tools and Materials

The tools used in this research are disk mill, 120 mesh and 200 mesh sieves, hot plate, static and clamps, desiccator, storage bottle, magnetic stirrer, thermometer, funnel, filter paper/cloth, aluminum foil, measuring cup, beaker glass, dropper, ph meter/ ph paper, drying oven and pan, petri dish, cloth/tissue, stopwatch, analytical balance, spatula/spoon, label paper and plastic zip pack, set of electromagnetic tools, set of freeze dryer (FD-10-MR), a set of lignocellulosic test kits, a set of X-ray Diffraction (XRD X’pert PRO PANalytical) tools and Particle size Analyzer (PSA Beckman Coulter LS 13 320). The materials used in this study were cassava stems, distilled water, 0.1N CH$_3$COOH, 25% NaOH, 3.5% NaOCl, 2.5 N HCl, ether, 96% ethanol and iodine.

2.2. Electromagnetic Specification

The electromagnetic specifications used in this study were: number of turns of wire: 800 turns, wire diameter: 0.7 cm, height of the socket: 7 cm, diameter of the socket: 15 cm, current capacity: 10 amperes, additional metal core 0, 32 cm, and magnetic induction: 2.6674 x 10$^{-5}$ Wb/m$^2$.

2.3. Pre-hydrolysis Process

Pre-hydrolysis was carried out by boiling cassava stem powder using 0.1 N acetic acid with a sample to solvent ratio of 1:20 for 60 minutes at 105°C (Haafiz et al., 2013). After that the sample is separated from the solvent by filtering and squeezing then the sample is rinsed repeatedly until the pH is neutral (Umar, 2011). Then it is dried in an oven.

2.4. Delignification Process

The isolation stage was continued with alkaline heating using 25% w/v sodium hydroxide (NaOH) at 105°C and boiled for 60 minutes with a ratio of 1:20 between the sample and sodium hydroxide (Haafiz et al., 2013). In this process a brown pulp (α-cellulose) is formed which is isolated as a residue. The pulp was then rinsed to neutral and dried.

2.5. Bleaching Process

This process was carried out by immersing plant fibers in a 3.5% NaOCl solution with a sample and solvent ratio of 1:20 for 60 minutes (repeated up to 2 times). After that, the solution was then filtered and the residue obtained was rinsed repeatedly using distilled water until the pH was neutral. Furthermore, the pulp was dried using an oven at 50°C for ±6 hours. The dry pulp obtained is referred to as α-cellulose (Widia et al., 2017).

2.6. Hydrolysis Process

α-cellulose obtained was further hydrolyzed. α-cellulose was then added with 2.5 N HCl solution and treated using an electromagnetic induction device with a ratio of 1:20 with a time of 60 minutes and a
temperature variation of 30, 50, 70°C. Then the mixture was boiled at 105 °C for 15 minutes. The solution was filtered and the residue rinsed repeatedly until the pH was neutral. The results obtained are then stored in the freezer for further drying using a freeze dryer. The result is cellulose nanocrystal.

3. Results and Discussion

3.1. Preparation Results

In this study, the raw substance that was given treatment in the process of making nanocrystalline cellulose experienced a color change from brownish-yellow to yellowish-white. The color changes that occur are shown in Figure 1.

![Figure 1](image_url) Changes in the Color of Substances in The Producing of Nanocrystalline cellulose. (a) Raw Materials; (b) Pre-hydrolysis Results; (c) Delignification Results; (d) Bleaching Results; (e) E0; (f) E30; (g) E50; (h) E70

The color change that occurred was caused by degraded lignin compounds during the treatment of making nanocrystalline cellulose. Lignin is a constituent compound that gives cellulose a brownish color, so that if lignin is removed it will cause the color of cellulose to be brighter. The effect of prehydrolysis on lignin dissolution was proposed by Wibisono et al., (2011), in this study it was explained that the ability of acetic acid to bind lignin will result in the release of α-cellulose bound to lignin so that the pulp content with high α-cellulose content is obtained.

Lignin degradation occurred again when the delignification process was carried out. The decrease in lignin during the delignification process is caused by the reaction of breaking the bond between lignin and hemicellulose because it is degraded by OH- ions, Na+ ions that have been cut off with OH- ions will bind to lignin and will dissolve in water (Nura’ et al., 2017). This is corroborated by Hamisan (2009) which states that the dark brown delignification solution is an indication that compounds having chromophore groups with conjugate bonds undergo a dissolution process.

The use of NaOCl in the bleaching process will increase the brightness of the α-cellulose produced, this result is caused by the dissolution of lignin which is still bound to the cellulose after the pre-hydrolysis and delignification process. Dissolution of residual lignin during the bleaching process occurs due to the degradation of lignin into short chains due to NaOCl solution so that it is easily dissolved in the solution (Fengel, 1995).

After the pre-hydrolysis, delignification and bleaching processes were carried out, cellulose was obtained which was then hydrolyzed with various variations of electromagnetic induction temperature. Each variation is hydrolysis without electromagnetic (E0), hydrolysis with electromagnetic 30°C (E30), hydrolysis with electromagnetic 50°C (E50) and hydrolysis with electromagnetic 70°C (E70). Each of these variations was then tested with several tests to determine the characterization of the nanocellulose produced.

3.2. Characterization of Nanocrystalline Cellulose

The characterization test aimed to compare the results of the nanocrystalline cellulose obtained from the research results with commercial MCC. From the tests that have been carried out on each variation of nanocrystalline cellulose and comparing them with commercial MCC samples, the characterization results are obtained in Table 1.

| Characteristic | Standard MCC | E0 | E30 | E50 | E70 |
|---------------|--------------|----|-----|-----|-----|

![Table 1](image_url)
Based on the characteristic test, it was found that all of the test characteristics on the hydrolysis variation of nanocrystals complied with the requirements set for MCC. According to the Indonesian Pharmacopoeia Edition III (1979), a good MCC is in the form of a powder, white, tasteless and odorless. In addition, the water content, solubility in various solutions, pH and starch content in nanocrystalline cellulose were according to MCC standards. The difference between nanocrystalline cellulose with MCC characteristic requirements in characteristic testing is color. The color on the terms or commercial MCC is white, while E0, E30, and E50 have a yellowish white color. While the E70 is brownish yellow.

Nanocrystalline cellulose had a yellowish white to brownish yellow color caused by hydrolysis carried out at high temperatures and for a long time. In the study of Vanhatalo et al. (2014) which used a hydrolysis temperature between 120-160°C and a time of 0-1440 minutes showed that an increasing hydrolysis temperature would result in a decreasing trend in brightness level, so it can be concluded that studies with a hydrolysis temperature lower than 120°C or more than 160°C will have similar trend. Browning of nanocrystalline cellulose is caused by glucose undergoing dehydration and condensation reactions (caramelization) in high-temperature acid solutions (Lichtenthaler, 2011).

From the research results, the brightness level of nanocrystalline cellulose is only affected by the parameters of temperature and hydrolysis time, while electromagnetic induction has no significant effect on the brightness level of nanocrystalline cellulose. This is based on the comparison of the brightness level of nanocrystalline cellulose E0 with E30. Based on observations, the brightness level of E0 nanocrystalline cellulose has no difference compared to E30 nanocrystalline cellulose. So it can be concluded that electromagnetic induction has no significant effect on the brightness level of nanocrystalline cellulose.

### 3.3. Lignocellulosic

Lignocellulose testing on nanocrystalline cellulose aimed to determine the effect of the hydrolysis process and electromagnetic induction on the levels of hemicellulose, cellulose and lignin contained in nanocrystalline cellulose. Tests were carried out on three samples of materials, namely alpha cellulose from bleaching (α-cellulose), E0, and E70. Based on the tests that have been carried out, the percentage results for each test material are attached in Table 2 below.

| Characteristic | Standard | MCC | E0 | E30 | E50 | E70 |
|---------------|---------|-----|----|-----|-----|-----|
| Form          | Powder  | ✓   | ✓  | ✓   | ✓   | ✓   |
| Odor          | Odorless| ✓   | ✓  | ✓   | ✓   | ✓   |
| Color         | White   | ✓   | ☒ | ☒   | ☒   | ☒   |
| Water         | Insoluble| ✓   | ✓  | ✓   | ✓   | ✓   |
| Alcohol 96%   | Insoluble| ✓   | ✓  | ✓   | ✓   | ✓   |
| HCl 2N        | Insoluble| ✓   | ✓  | ✓   | ✓   | ✓   |
| NaOH 1N       | Insoluble| ✓   | ✓  | ✓   | ✓   | ✓   |
| Eter          | Insoluble| ✓   | ✓  | ✓   | ✓   | ✓   |
| pH            | 5.5-7.5 | ✓   | ✓  | ✓   | ✓   | ✓   |
| Starch        | None    | ✓   | ✓  | ✓   | ✓   | ✓   |
| Water Content | <5%     | ✓   | ✓  | ✓   | ✓   | ✓   |

The results of the lignocellulose test showed that the hydrolysis treatment would increase the cellulose content and decrease the hemicellulose and lignin content. In accordance with research conducted by Sun et al. (2002), the use of an acid solution in hydrolysis will cause damage to the lignin and hemicellulose bonds, then the lignin and hemicellulose are dissolved in the hydrolyzed acid solution. In the hydrolysis process, electromagnetic induction will accelerate the process of breaking lignin and hemicellulose so that it will increase the cellulose content. This is evidenced by the lignocellulosic test on the variation of E70 cellulose nanocrystal which obtained the highest cellulose value of 62.93%.

### 3.4. X-Ray Diffraction

XRD testing aims to determine the crystallinity of a sample or material. From the XRD test results that have been carried out on the sample, the XRD data is then processed using the Origin Pro application, the graph in Figure 2.

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### Table 2. Lignocellulosic Results

| No | Substance | Hemi | Cell | Lig | Wat | Ash |
|----|-----------|------|------|-----|-----|-----|
| 1  | α Selulosa| 20.39| 33.59| 42.42| 3.18| 0.40|
| 2  | E0        | 16.87| 47.10| 32.42| 2.19| 1.39|
| 3  | E70       | 14.35| 62.93| 20.73| 1.52| 0.44|

The results of the lignocellulose test showed that the hydrolysis treatment would increase the cellulose content and decrease the hemicellulose and lignin content. In accordance with research conducted by Sun et al. (2002), the use of an acid solution in hydrolysis will cause damage to the lignin and hemicellulose bonds, then the lignin and hemicellulose are dissolved in the hydrolyzed acid solution. In the hydrolysis process, electromagnetic induction will accelerate the process of breaking lignin and hemicellulose so that it will increase the cellulose content. This is evidenced by the lignocellulosic test on the variation of E70 cellulose nanocrystal which obtained the highest cellulose value of 62.93%.
The XRD graph displayed a pattern of fluctuating lines resulting from the refraction of X-ray by crystals on the particles of the test material. The more and higher the line of refraction, the higher the crystallinity of the material. From the calculations that have been carried out using the value of the peak position, the area of the crystalline and amorphous fractions and the FWHM obtained from the XRD test graph of each test material, the crystallinity is listed in Table 3.

Table 3. Crystallinity

| Sample   | Crystallinity |
|----------|---------------|
| α-selulosa | 48.99%        |
| E0       | 74.75%        |
| E30      | 76.40%        |
| E50      | 79.23%        |
| E70      | 90.68%        |
| MCC Comercial | 84.31%    |

From the results of calculations, cellulose from bleaching initially only had a crystallinity of 48.99%, then increase to 74.75% after being hydrolyzed. This proves that hydrolysis affects the crystallinity of nanocrystalline cellulose. The increase in crystallinity occurs because the hydrolysis process will break the long chains of cellulose, so that the amorphous part of the cellulose microfibril is cut off and leaves the crystalline part (Ma et al., 2016). Thus, the less amorphous the crystallinity level will increase.

From Table 3, high temperatures will increase the level of crystallinity of nanocrystalline cellulose. This is in accordance with Nura' Ini's research (2017), hydrolysis with the same time of 75 minutes at different temperatures produces different levels of crystallinity. At a temperature of 120°C obtained crystallinity of 74.3% while at a temperature of 160°C of 79.1%. In this study, the best crystallinity value was obtained by hydrolysis treatment with electromagnetic 70°C, where the crystallinity value was 90.68%. Compared with the same treatment at a lower temperature of 50°C, the crystallinity value increased by 14.38%. The higher the hydrolysis temperature used, the more effective the hydrolysis process that occurs, because hydrolysis requires high temperatures to occur optimally.

The effect of electromagnetic on crystallinity can be seen from the comparison of the results of the cellulose nanocrystal test at variations of E0 and E30. At a temperature of 30°C (room temperature) hydrolysis occurs at a small rate, because a large number of hydrolysis reactions occur when the solution temperature reaches 70°C. So that in the E30 treatment, the dominant parameter that affects the decrease in size and increase in crystallinity is electromagnetic waves. Judging from the test results, the test material in the E30 treatment had a crystallinity of 76.40%. This result is better than the E0 hydrolysis treatment (room temperature) which is 74.75%. So it can be concluded that the electromagnetic effect on the increase in crystallinity. The effect of electromagnetic induction is stated by Siregar (2007) where the particles affected by magnetic induction will undergo splitting into smaller parts.

3.5. PSA (Particle Size Analyzer)

PSA testing aims to determine the crystal size of the cellulose produced in the study. Figure 3 is a graph of the results of the PSA test on the test material of this study.
Figure 3. Particle Size Analyzer Results. (a) Cassava Stem Powder; (b) MCC Commercial; (c) E0; (d) E30; (e) E50; (f) E70

Just like the XRD test chart, the PSA chart also displays an up and down line pattern. The line pattern shows the distribution of the cellulose crystal size of the test material in the ratio of volume (%) with a range of 0-100% and diameter (µm) between 0.375-2000 µm. The higher the graph, the higher the volume of cellulose crystals at a certain size. And if the graph is getting to the right, then the size of the cellulose crystals will be getting bigger. From the PSA test results, the average size is shown in Table 4 below.

| Sample               | Size  |
|----------------------|-------|
| Cassava Stem Powder  | 166.2 µm |
| α-selulosa           | 83.78 µm |
| MCC Commercial       | 57.66 µm |
| E0                   | 22.22 µm |
| E30                  | 49.33 µm |
| E50                  | 46.65 µm |
| E70                  | 18.04 µm |

Based on the data from the particle size test of cellulose crystals obtained in this study, the variation of hydrolysis with or without electromagnetic resulted in a decrease in the particle size of cellulose crystals. Hydrolysis treatment without electromagnetic can reduce the particle size of alpha cellulose from 83.78 µm to 22.22 µm, or 73.47%. According to Ma et al. (2008), the decrease in cellulose particle size was caused by the erosion of the amorphous part of the microfibrils, so that the more amorphous eroded, the smaller the size of the cellulose crystals of the test material. The effect of hydrolysis in decreasing the crystal size of cellulose was also proven in a study conducted by Zhang et al. (2007). In this study, pure cellulose with a size of 0.465 mm was hydrolyzed with an acid solution. After hydrolysis, the average particle size was 560 nm. So it can be concluded that hydrolysis can reduce the size of cellulose particles.

The Particle Size Analyzer (PSA) test results showed that an increase in temperature would tend to reduce the particle size of cellulose crystals. In the E30 variation, the particle size of cellulose crystals was 49.33 µm, E50 obtained the particle size of cellulose crystals of 46.65 µm, whereas when using E70, the particle size of cellulose crystals was smaller, which was 18.04 µm. The decrease in particle size resulting from the hydrolysis process was also obtained in a study conducted by Lismeri et al., (2018). In this study, PSA testing on the hydrolysis test material with a time of 90 minutes and a temperature of 70°C at 228.8 nm while at
the same time but at a temperature of 50°C at >400 nm. This happens because at high temperatures the hydrolysis will be more optimal so that the amorphous degradation process will be higher (Nura’ Ini et al., 2017).

The electromagnetic effect on this research can be seen from the comparison of the results of the cellulose crystal testing on the E0 and E70 treatments. From the test results, E70 has the smallest average size of 18.04 µm. While the hydrolysis treatment without electromagnetic has a size of 22.22 µm. These results indicate that there is an electromagnetic effect on the decrease in the crystal size of cellulose. However, the variation of hydrolysis with electromagnetic at temperatures below 70°C has a larger cellulose crystal size than without electromagnetic. This is possibly caused by the electromagnetic at certain parameters tends to change some structures to become amorphous. This phenomenon occurs in the study of Sasue et al. (2017) which uses electromagnetic power at 300 watts, after testing the results obtained that electromagnetic waves will increase the amorphous level in certain areas so that the size of cellulose crystals becomes larger. Based on this, it can be concluded that the use of electromagnetic induction as a method of reducing the size of cellulose crystals must be regulated by certain parameters.

PSA testing showed that the size of the cellulose crystals produced still varied in the nanometer to micrometer size distribution. The distribution of particles in nanometer size is in the size range of 0.375-0.954 µm or 375-954 nm. The largest percentage of nano-sized crystalline cellulose particles is found in the E70 variation, which is 4.819%. So it can be concluded that the hydrolysis treatment with electromagnetic can reduce the size of cellulose crystals into nanoparticles, but in a very small percentage. Based on the test results, it can be concluded that all the results of the treatment variations of nanocrystalline cellulose in this study have characteristics close to the standards set for MCC, and the results of research with variations of 70°C electromagnetic hydrolysis treatment have better sizes and crystallinity levels compared to commercial MCC. The smaller the crystal size, the more and easier the nanocrystalline cellulose are distributed in medicine or food when used as a filler, so that the product is denser. Meanwhile, the higher the crystallinity of a cellulose nanocrystal used as a drug or food filler, the stronger and stiffer the product will be.

4. Conclusion

From this research, the best cellulose nanocrystal product was obtained, namely the E70 variation with a crystallinity degree of 90.68% and a particle size of 375 nm as much as 0.099% where the use of electromagnetic induction in the hydrolysis process has an influence on the size and degree of crystallinity of nanocrystalline cellulose. However, electromagnetic induction did not affect the brightness of the nanocrystalline cellulose, because the brightness of the nanocrystals was affected by the increase in temperature and the length of time the hydrolysis process took place.

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References

Arjuna, A., Natsir, S., Khumaerah, A.A., and Yulianty, R. (2018) Waste cabbage fibers modification into nano-crystalline cellulose via acid hydrolysis method, Galenika Journal of Pharmacy, 4, 119-125.

Badan Pusat Statistik (2018) Cassava Production by Province 2014-2018, Central Bureau of Statistics Website Data.

Farmakope Indonesia (1979) 3rd edition, Ministry of Health of the Republic of Indonesia.

Fengel, D. (1995) Wood: Chemistry, Ultrastructure, Reactions, First Printing. Gadjah Mada University Press, Yogyakarta.

Fuhaid, N., Sahbana, M.A., and Arianto, A. (2011) The Effect of Electromagnetic Fields on Fuel Consumption and Exhaust Emissions in Gasoline Motors, Journal of PROTON, 3, 1-9.

Haafiz, M.K.M., Eichorn, S.J., Hassan, A., and Jawaid, M. (2013) Isolation and Characterization of Microcrystalline Cellulose from Oil Palm Biomass
Residue, Journal of Carbohydrate Polymers, 93, 628–634.

Hamisan, A.H. (2009) Delignification of Oil Palm Fruit Bunch using Chemical and Microbial Pretreatment Methods, International Journal of Agriculture Research, 4, 250-256.

Lichtenthaler, F.W. (2011) Carbohydrates: Occurrence, Structures and Chemistry Ullman’s Encyclopedia of Industrial Chemistry, 7th edition, Wiley-VCH, Weinheim.

Lismeri, L., Zari, Poppy M., N. Tika, and Darni, Y. (2016) Cellulose Acetate Synthesis from Cassava Stem, Journal of Chemical and Environmental Engineering, 11, 82-91.

Lismeri, L., Irmalinda, G., Darni, Y., and Herdiana, N. (2018) Application of Cellulose Fiber from Cassava Stem Waste as Composite Film Based on Low Density Polyethylene (LDPE), Proceedings of the 7th National Leather, Rubber and Plastic, Yogyakarta, 29 August 2018.

Lismeri, L., Utami, Rhiki S.U., Darni, Y., Hanif, M., and Riyanto, A. (2018) Production of Reducing Sugars From Cassava Stem by Hydrolysis Using Dilute Acid and Electromagnetic Field Induction, Journal of Chemical and Environmental Engineering, 13, 8-14.

Lismeri, L., Agustina, E., Darni, Y., Agustin, N., and Damara, N. (2020) Preparation and Characterization of Cellulose Microcrystals from Cassava Stem Waste, Journal of Technology and Industrial Innovation, 1, 028-036.

Ma, X., Zheng, X., Yang, H., Wu, H., Cao, S., and Huang, L. (2016) A Perspective on Lignin Effects on Hemimicrocelluloses Dissolution for Bamboo Pretreatment, Journal of Industrial Crops and Products, 94, 117-121.

Nura’ Ini, E. and Putra, S.S.H. (2017) Production of Microcrystalline Cellulose (MCC) from Sengon Wood Sawdust Waste Through Sonication and Hydrothermal Processes, Undergraduate thesis, Institut Teknologi Sepuluh Nopember, Surabaya.

Rowe, R.C., Sheskey, P.J., and Quinn, M.E. (2009) Handbook of Pharmaceutical Excipient Sixth Edition, Pharmaceutical Press, Washington.

Samir, M.A., Alqani, F., and Dufresne, A. (2005) Review of Recent Research into Cellulosic Whiskers, Their Properties and Their Application in Nanocomposite Field, Journal of Biomacromolecules, 6, 612-626.

Sasue, R., Sanglan, H.F., Mosey, H.I.R. (2017) Analysis of the Effect of Microwave Radiation on Starch Crystal Structure, Journal of MIPA UNSRAT Online, 6, 59-62.

Siregar, H.P. (2007) The Effect of Electromagnetic-Based Energy Saving Device Coil Diameter on Diesel Motor Performance, Journal of Mechanical Engineering, 9, 1-8.

Sumada, K., Tamara, P.E.T., and Alqani, F. (2011) Isolation Study of Efficient α-Cellulose from Waste Plant Stem Manihot esculenta crantz, Journal of Chemical Engineering, 5, 434 – 438.

Sumiati, M., Wahyuni, D., and Malino, M.B. (2016) Analysis of the Relationship of Acid Concentration during Hydrolysis, Degree of Crystallinity and Properties of Crystalline Cellulose from Wood Sawdust, Journal of Prisma Fisika, IV, 64-68.

Sun, Y. (2002) Enzymatic Hydrolysis of Rye Straw and Bermudagrass for Ethanol Production, Raleigh: Ph.D. Thesis, NC State University.

Umar, S.T. (2011) Utilization of Hemp Fiber for Cellulose Production, Datinilitbang-BPP Kemenhan RI, Tersedia di: http://www.balitbang.kemhan.go.id/?q=content/pemanfaatan-serat-ramiuntuk-pembuatan-serat-selulosa, Retrieved 15 December 2019.

Vanhatalo, K.M. and Dahl, Olli P. (2014) Effect of Mild Acid Hydrolysis Parameters on Properties of Microcrystalline Cellulose, Journal of Bioresources, 9, 4729-4740.

Wibisono, I., Hugo, L., Antaresti, and Aylianawati. (2011) Making Pulp from Reeds, Journal of Widya Technic, 10, 11-20.

Widia, I. and Wathono, N. (2017) Review Article Microcrystalline Cellulose:
Isolation, Characterization, and Applications in the Pharmaceutical Field, *Journal of Farmaka*, 15, 127-143.

Zhang, Jianguo., Elder, Thomas J., Pu, Yunqiao., Ragauskas, Arthur J. (2007) Facile Synthesis of Spherical Cellulose Nanoparticles, *Journal of Carbohydrate Polymers*, 69, 607-611.