

Characterization of Microcrystalline from Pineapple Leaf (*Ananas comosus* L. Merr)

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Abstract. Microcrystalline cellulose (MCC) is a cellulose derivative obtained by treating the alpha cellulose contained in fibrous plants using acid solutions. Types of natural fibers from plants obtained from the pineapple leaf. Chemical composition of pineapple leaf showed that the content of cellulose is high at 69.5 to 71.5%. This research was conducted through the isolation of alpha cellulose with a NaOH solution, then hydrolyze with HCl solution, then crushed mechanically to obtained microcrystalline cellulose. Characterization of microcrystalline cellulose include, organoleptic test, loss of drying test, color identification reagents test, water solubility test, starch test, pH test, analysis and identification of functional groups microcrystalline cellulose by spectrophotometry Fourier Transform Infrared (FT-IR). Microcrystalline cellulose obtained is fine powder, white, odorless, pH 6.1, loss of drying 3.3%, giving a positive result in a identification test with iodized zinc chloride, solubility in water 0.16% and there is no starch in microcrystalline cellulose. The results of the analysis of functional groups microcrystalline cellulose obtained from pineapple leaf compared with Avicel using FT-IR showed similar wavenumber in the functional groups region and the fingerprint region. It was concluded that the pineapple leaf contain alpha cellulose and can be made into microcrystalline cellulose.

1. Introduction

Microcrystalline cellulose is a cellulose derivative obtained by treating the alpha cellulose contained in fibrous plants using acid solutions. Microcrystalline cellulose utilizing in pharmaceutical field as an excipients ingredient in the tableting formulation, as a binder [1]. The main content has the potential to be used as a raw material for making microcrystalline cellulose is fibrous material and which contains quite high cellulose. Every material has a different number of cellulose. Some of the natural resources used to produce cellulose such as found in cotton 90-95%, jute 80-90%, wood 40-50%, and bark 20-30% [2].

One of the cellulose contained in plants are pineapple leaves which are the type of natural fiber derived from plants. Pineapple (*Ananas comosus* L. Merr.) leaf shape resembles a sword pointed at one end with a blackish green color and the edges of the leaves are sharp thorns. Chemical composition of the leaves of pineapple (*Ananas comosus* L. Merr.) showed that the content of cellulose is high at 56%-82%; lignin from 4.4-4.7%; pentosan 17.0-17.8%; pectin 1.0-1.2%; fat and



wax 3.0-3.3%; ash 0.71-0.87%; other substances such as proteins, organic acids and others 4.5-5.3% [3, 11].

Cellulose can be made into microcrystalline cellulose with nitric acid solvent method with sodium nitrite and can also be made with sodium hydroxide solvent method. Nitric acid solvent method and sodium nitrite was prepared by dissolving cellulose in a strong alkaline solution to obtained almost pure cellulose, known as α -cellulose and by immersing α -cellulose with acid, then smoothed mechanically to obtained microcrystalline cellulose. The need for microcrystalline cellulose to Indonesia all from imports, particularly relevant when microcrystalline cellulose production conducted in the country. So that researchers interested in conducting research microcrystalline cellulose insulation from the leaves of pineapple (*Ananas comosus* L. Merr.) Using the solvent method of nitric acid and sodium nitrite [4].

The results of this study are expected to provide information on the utilization of the leaves of pineapple (*Ananas comosus* L. Merr.) As an alternative source of microcrystalline cellulose that can be used as a binder in the manufacture of tablets, giving value to the leaves of the pineapple (*Ananas comosus* L. Merr.) Which usually an agricultural waste.

2. Materials and Methods

2.1 Tools

Analytical balance (Shimadzu), Blender (Miyako), Hotplate (Cimarec), Fourier Transform Infrared Spectrophotometer (Shimadzu), Oven (Memmert), Cabinet Dryers, Filter Paper, Desiccator, pH meter (Hanna), Water Bath, Universal Indicator (Merck) and Other Glasses (Pyrex).

2.2 Materials

Pineapple leaves (*Ananas comosus* L. Merr.), Nitric acid (HNO_3), sodium nitrite (NaNO_2), sodium hydroxide (NaOH), sodium sulphite (NaSO_3), sodium hypochlorite (NaOCl), 37% hydrochloric acid (HCl), zinc chloride (ZnCl_2), potassium iodide (KI), iodine (I_2) and distilled water.

2.3 Sampling

Sampling leaf pineapple (*Ananas comosus* L. Merr.) is done purposively by the characteristics of dark green and often a waste obtained from plantations in the village community Matangglumpangdua Peusangan Bireuen District of Aceh.

2.4 Reagent Preparation

2.4.1 Carbon Dioxide Free Distilled Water

Distilled water is boiled for 5 minutes and cooled should not absorb carbon dioxide from the air [5].

2.4.2 Nitric Acid Solution 3.5% (v / v)

Concentrated nitric acid were measured amount of 75.3 mL and diluted with distilled water to 1.4 liters.

2.4.3 Sodium Sulfite Solution 2% (w / v)

Sodium sulfite weighed as much as 20 grams and dissolved into distilled water up to 1 liter.

2.4.4 Sodium Hydroxide Solution 2% (w / v)

Sodium hydroxide weighed as much as 20 grams and dissolved into carbon dioxide free distilled water to 1 liter.

2.4.5 Sodium Hydroxide Solution 17.5% (w / v)

Sodium hydroxide weighed as much as 119 grams and dissolved into carbon dioxide-free distilled water up to 680 mL

2.4.6 Hydrochloric Acid Solution 2.5 N

Concentrated hydrochloric acid were measured amount of 100 ml and then diluted with distilled water up to 480 mL

2.4.7 Iodine Solution 0.05 N

Weighed as much as 0.2 grams of potassium iodide, dissolved in a little amount of water and added 0.13 grams of iodine. Shake until dissolved and diluted with water to 10 mL [6].

2.4.8 Iodized Zinc Chloride Solution

Weighed as much as 20 grams of zinc chloride and 6.5 grams of potassium iodide, dissolved in 10.5 mL of water. Then added 0.5 gram of iodine and shaken for 15 minutes. Filtered and stored in a place protected from light [6].

2.5 Sample Processing

Pineapple leaves washed and drained, chopped, then dried in the drying cabinet. Samples were dried powdered by using a blender and stored in a dry place. Weighed as much as 100 grams of dry powder, mixed with 1.4 liters of 3.5% nitric acid containing 15 mg of sodium nitrite, put in a beaker glass, heated in a water bath at 90°C for 2 hours, filtered with filter paper. The residue was added 1 liter of a mixture of sodium hydroxide solution 2% and 1 liter of 2% sodium sulfite, heated at 50°C for 1 hour, filtered and bleached with 1.2 liters of sodium hypochlorite 3.5% and 1.2 liter of water (1 : 1) boiled for 10 minutes, the mixture is washed and filtered to obtain alpha cellulose.

2.6 Separation of Alpha Cellulose

Cellulose pulp obtained from pineapple leaf powder added with 680 ml of 17.5% sodium hydroxide is heated at 80°C for 30 minutes. The result is then washed with water and filtered, the waste is combined with a mixture of 1.2 liters of sodium hypochlorite 3.5% and 1.2 liter of water (1: 1), heated at 100°C for 5 minutes, then washed with water until clear filtrate, filtered and squeezed, and then dried at 60°C in an oven to obtain alpha cellulose [7].

2.7 Manufacture of Microcrystalline Cellulose

Weighed as much as 10 grams of alpha cellulose was added glass beaker and hydrolyzed with 480 ml HCl 2.5 N by boiling for 15 minutes, then poured in cold water while stirring vigorously with a spatula and let stand 24 hours. microcrystalline cellulose produced from this process is washed with water to neutral pH, filtered and dried in an oven at 57°C - 60°C for 1 hour. The obtained microcrystalline cellulose, microcrystalline cellulose subsequently crushed. Results are stored at room temperature in the desiccator [7].

2.8 Examination Microcrystalline Cellulose

Microcrystalline Cellulose examination include: organoleptic test, loss of drying test, color identification reagents test, water solubility test, starch test, pH test, FT-IR spectrophotometry identification test. This test compared with the requirements of the British Pharmacopoeia [6] and the Indonesian Pharmacopoeia [8].

2.9 Characterization of Microcrystalline Cellulose

2.9.1 Organoleptic Test

Characteristics of the sample form that is placed on top of the white base, then observed shape or appearance, color, taste, and smell [6].

2.9.2 Loss of Drying Test

Weighed as much as 1.25 gram sample is inserted in a crucible porcelain, dried in an oven at 100°C - 105°C to constant weight is obtained. Percentage of loss of drying can be determined by the weight ratio of the sample weight before drying to the weight after drying [6].

2.9.3 Color Identification Reagent Test

Weighed as much as 10 mg of sample is attached to the watch glass and dispersed in 2 mL solution of zinc chloride iodized. Microcrystalline cellulose samples will become blue violet [6].

2.9.4 Water Solubility Test

1.25 gram sample is weighed, shaken with 20 mL of distilled water for 10 minutes, filtered, evaporated filtrate water bath at a temperature above 100°C - 105°C for one hour. Weight remainder should be no more than 12.5 mg (0.25%) [6].

2.9.5 Starch Test

A total of 10 mg of powder was added 90 ml of distilled water, heated for 15 minutes, filtered while hot, cooled filtrate, added 0.1 mL of 0.05 M iodine. Samples Microcrystalline cellulose samples does not contain starch that is not blue coloration [6, 8].

2.9.6 pH Test

Weighed as much as 2 grams of powder, mixed with 100 ml of distilled water for 5 minutes, and measured pH with a pH meter [7, 8].

2.9.7 Infrared Spectrophotometer Identification Test

Weighed \pm 2 mg of sample and 200 mg of potassium bromide, put in a mortar and ground until homogenous mixture is measured using a spectrophotometer infrared fourier transform.

3. Results and Discussions

3.1 Leaves Pineapple (*Ananas comosus* L. Merr.) Microcrystalline Alfa Cellulose Results

In this study, the sample used is the leaves of pineapple (*Ananas comosus* L. Merr.). Obtained from the pineapple leaf plantations in the village community Matangglumpangdua Peusangan Bireuen District of Aceh. Making microcrystalline cellulose from pineapple leaf using nitric acid and sodium nitrite. Yield results that in alpha cellulose can be as much as 15.48% of the 100 gram sample pineapple (*Ananas comosus* L. Merr.) leaves. Microcrystalline cellulose to yield as much as 7.25 grams (7,25%).

Theoretically, the chemical composition of the leaves of pineapple showed that the content of cellulose is high at 69.5 to 71.5% [3]. While the results obtained yield as much as 15.48% alpha cellulose. This can happen due to several factors, among others, due to the sample used is too little, the fertility of the soil or land use, and can also occur because of the type (variety) from pineapple leaves are used theoretically different from the varieties of pineapple leaves are used in the research.

Table 1. Microcrystalline Cellulose Examination Results

No	Parameter	Results	Specification
Organoleptic Test			
1	• Appearance	Fine Powder	Fine Powder
	• Colour	White	White
	• Smell	Odorless	Odorless
	• Taste	Tasteless	Tasteless
2	Loss of Drying Test	3,3%	Weight Losing Not More Than 6% [9]
3.	Water Solubility Test	0,16%	Weight of Dissolved Not More Than 0.25% [6]
4.	Starch Test	Not Formed Blue Color	Not Formed Blue Color [6]
5.	pH Test	6,1	5,0 - 7,5 [9]
6.	Color Identification Reagent Test	Violet Blue Color	Violet Blue Color [6]

According to the table above, the characteristics of the test results it can be concluded microcrystalline cellulose from pineapple leaves meet the requirements.

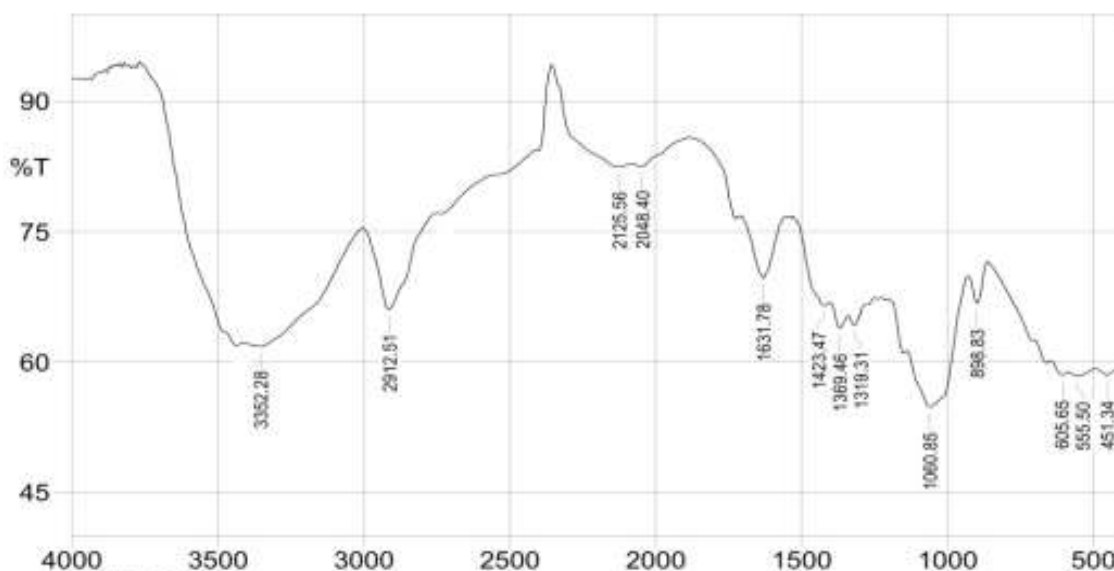


Figure 1. Fourier Transform Infrared Spectrum of Microcrystalline Cellulose

From the figure above results obtained fourier transform infrared spectrum of microcrystalline cellulose from pineapple leaves, where the wave number 3352.28 cm^{-1} shows the OH stretching vibration. Absorption band at wavenumber 2912.51 cm^{-1} showed the presence of C-H aliphatic stretching vibration supported by an absorption band at wavenumber 1423.47 cm^{-1} and 1369.46 cm^{-1} , which showed the presence of C-H aliphatic bending vibration. In wavenumber 2125.56 cm^{-1} and 2058.40 cm^{-1} indicate the presence of O-H vibration. Absorption band 1631.78 cm^{-1} shows the C = O bending vibration [10]



Figure 2. Overlapping Spectrum of Microcrystalline Cellulose from Pineapple Leaves with Standard Avicel

Fourier transform infrared overlapping spectrum of microcrystalline cellulose from pineapple leaves with standard avicel there is a similar shape the spectrum and wave number value in the functional groups region and the fingerprint region. Thus, powder isolates isolated from pineapple leaf is microcrystalline cellulose.

Table 2. Results of Data Comparison between Microcrystalline Cellulose from Pineapple Leaves and Avicel

Nomor	Wave Number of Microcrystalline Cellulose from Pineapple Leaves	Wave Number of Microcrystalline Cellulose from Standard Avicel	Interpretation
1.	3344,57 cm ⁻¹	3352,28 cm ⁻¹	O-H Stretching
	2897,08 cm ⁻¹	2912,51 cm ⁻¹	C-H Aliphatic Stretching
2.	1431,18 cm ⁻¹	1423,47 cm ⁻¹	C-H Aliphatic Bending
	1357,89 cm ⁻¹	1369,46 cm ⁻¹	C-H Aliphatic Bending
	2133,27 cm ⁻¹	2125,56 cm ⁻¹	O-H Stretching
3.	2056,12 cm ⁻¹	2058,40 cm ⁻¹	O-H Stretching
	1635,64 cm ⁻¹	1631,78 cm ⁻¹	C=O Bending

4. Conclusions

Pineapple leaf is a waste on the farm, so that should be a process for the utilization of pineapple leaf waste into a product that has a sale value. Pineapple leaf successfully processed into microcrystalline cellulose used in the pharmaceutical, cosmetics and food. Microcrystalline cellulose from pineapple leaves obtained is fine powder, white, odorless, pH 6.1, loss of drying 3.3%, giving a positive result in a identification test with iodized zinc chloride, solubility in water 0.16% and there is no starch in microcrystalline cellulose. The results of the analysis of functional groups microcrystalline cellulose obtained from pineapple leaf compared with Avicel using FT-IR showed similar wavenumber in the functional groups region and the fingerprint region. It was concluded that the pineapple leaf contain alpha cellulose and can be made into microcrystalline cellulose

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