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THE ISOLATION OF α-CELLULOSE & THE PREPARATION OF MICROCRYSTALLINE CELLULOSE FROM SOURSOP LEAVES (*Annona muricata* L.)

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ABSTRACT

Powder characteristics are very important to consider in the tablet manufacturing process because they can affect the characteristics of the resulting tablets. This study aims to determine the characteristics of microcrystalline cellulose powder isolated from soursop leaves. This research method includes preparation of soursop leaves raw material, isolation of α-cellulose using 17.5% NaOH solution with 3.5% nitric acid (containing 20 mg sodium nitrite), manufacture of microcrystalline cellulose using 1 M HCl solution, and characterization of microcrystalline cellulose including quality checks. powder (organoleptic, identification, solubility), and pH testing. The results showed that the yield of microcrystalline cellulose (MCCDSr) was 96.59% against α-cellulose. The results of the characterization of microcrystalline cellulose powder including organoleptic, identification, solubility, melting point, and pH showed similar characteristics with Avicel® PH 101 as a comparison. Based on the research that has been done, the characteristics of microcrystalline cellulose powder from soursop leaves are similar to Avicel® PH 101.

Keywords: soursop leaves, delignification, hydrolysis, cellulose, microcrystalline cellulose.

INTRODUCTION

Indonesia is an enormous country with positive development in the homegrown drug item piece of the pie consistently. Notwithstanding, in the assembling of drug items, the drug business in Indonesia is still exceptionally reliant upon imported unrefined components, until January 2013 it was recorded that practically 96% of the natural substances utilized by the Indonesian drug industry were as yet imported[1].

Indonesia is a country wealthy in plentiful normal assets, where the abundance of regular assets is straightforwardly connected with the variety of synthetic mixtures contained in it so it has extraordinary potential for drug improvement[1]. As is notable, Indonesia is one of the nations that have the biggest backwoods on the planet which has a wide assortment of verdure. In Indonesia, there are additionally many kinds of plants that can be utilized as

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meds, flavors, etc[2]. Indonesia is an extremely wide archipelagic nation, has roughly 35,000 huge and little islands with an exceptionally high variety of vegetation. Indonesia is assessed to have 100 to 150 groups of plants, and of these, the vast majority of them can be utilized as modern plants, organic product trees, zest plants, and restorative plants. Because of WHO records, over 20,000 types of therapeutic plants are utilized by individuals around the world[3]. One of these natural resources is Soursop leaves. Use of plants as traditional medicinal ingredients require scientific research to find out the truth of its usefulness[4].

Soursop (*Annona muricata* L.) has for quite some time been referred to and is ordinarily utilized as a medication for ages by individuals of Indonesia. Soursop trees can develop without extraordinary consideration in the nursery or yard. In antiquated times, the soursop plant was just known locally for outside treatment, particularly skin sicknesses. Be that as it may, starting around 2010, soursop organic product is known to be effective for treating diarrhea, intense bile, and urinary stones. The leaves are additionally valuable for treating cerebral pains, sleep deprivation, liver sickness, diabetes, and hypertension, and as a mitigating, antispasmodic, diarrhea, ulcers, bubbles, seizures, skin break out, and head lice[5].

Soursop leaves contain alkaloids, tannins, and several other chemical constituents including *Annonaceous acetogenins*[5]. Soursop leaves contain 87.58% dry matter, 8.93% ash, 16.9% protein, 28.3% crude fiber, 4.76% crude fat and 2.09% calcium. There is also 4.08% hemicellulose, 34.71% cellulose, 14.64 lignin, and 0.25% silica in soursop leaves flour[6]. Cellulose is perhaps the most bountiful sustainable regular asset in Indonesia and is the primary part of lignocellulose from cell walls in plants alongside hemicellulose, lignin, gelatin, and wax[7]. Unadulterated cellulose or α-cellulose can be acquired from plant filaments, then treated with sodium hydroxide to isolate lignin (delignification). Lignin can be taken out by substance implies utilizing sodium hydroxide, or by microbiological techniques utilizing cellulosic growths[8,9].

Microcrystalline cellulose is by and large viewed as a tablet filler in the direct pressure technique in drugs[10]. Microcrystalline cellulose has dry restricting properties and can work as a disintegrant. Microcrystalline cellulose as a smasher should have low oil

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properties because microcrystalline cellulose has a low coefficient of contact. Having a low coefficient of contact doesn't make microcrystalline cellulose not utilized in that frame of mind with a combination of deteriorating specialists and ointments[11].

The wellspring of MCC is cellulose tracked down in woody plant filaments, corn cobs, cotton, hemp, bagasse, and straw. MCC can be made by synthetic or enzymatic strategies. In the compound planning of MCC, when α-cellulose is hydrolyzed with dilute mineral acid, and new product (microcrystalline cellulose) is then framed. Dilute mineral acid commonly used is HCl with an optimal concentration of 1 M[8,9,12,13].

As of late, Indonesia can meet its own medication needs, practically 90% of medication needs come from homegrown items, however, the drug business in Indonesia is still exceptionally reliant upon imported unrefined components, which is practically 96% of the complete natural substances utilized, one of which is the import of unrefined components for cellulose and microcrystalline cellulose[12]. Consequently, different elective wellsprings of unrefined components from regular materials are required in Indonesia to limit the requirement for imports.

There are not many studies related to the cellulose content and the manufacture of MCC from soursop leaves. According to research conducted by Londok & Mandey (2014), soursop leaves contain 34.71% cellulose. So the novelty in research on making MCC from soursop leaves is still very high[6].

Based on the research above, the researchers were interested in isolating α-cellulose from soursop leaves plants and preparation of microcrystalline cellulose from soursop leaves (MCCDSr) by chemical hydrolysis method using dilute acid (HCl). The obtained MCCDSr was then tested for physical properties to determine the characterization of the sample and compared with a standard comparison (Avicel PH 101).

METHODS

Tools and materials

The tools used in this study were: Soursop Leaves, 70% Ethanol (Ikapharmindo), Digital Balance (CAS), Stirring Rod, Neck Flask 1, Glass Beaker (Pyrex), pH Paper (Merck), Oven, Erlenmeyer (Pyrex), Dropper Pipette, Stirrer Hotplate, Glass Funnel (Pyrex), Filter Paper, 4 L Glass Jar, Thermometer,

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And other glassware. The ingredients used include Soursop Leaves, Aquadest, Sodium Hydroxide, Nitric Acid, Sodium Nitrite, Zinc, Potassium Iodide.

Research Path

1. Simplicia Preparation

Soursop leaves were gathered however much 5 kg, then wet arranging was completed to isolate debasements or unfamiliar materials contained in simplicia. Soursop leaves are then washed with running water, depleted, then, at that point, cleaved, dried by circulating air through in the air safeguarded by direct daylight, then, at that point, crushed with a blender, and sieved with a 40 cross section strainer (Lestari, 2022 & Rizki, *et al*. with modification)[12,13].

2. Extraction

Simplicia powder from soursop leaves was extracted by maceration method using 70% ethanol organic solvent until simplicia was submerged with solvent (1:5). Maceration is carried out until the filtrate looks almost colorless, then aerated until a thick extract is obtained[14]. Extraction serves to separate the material from the mixture using a suitable solvent. The extraction process is stopped when balance is reached the concentration of the compound in the solvent with the concentration inside the cell plant[15].

3. Isolation of α-Cellulose

Every 150 g of soursop leaves powder is mixed with 3.5% nitric acid (containing 20 mg sodium nitrite) as much as 2 L in a container. The next procedure refers to our previous study[8].

4. Preparation of MCC of Soursop Leaves

α-Cellulose of Soursop leaves obtained were then hydrolyzed with hydrochloric acid, each as much as 1 gram of soursop leaves cellulose was added with 20 ml of 1 M hydrochloric acid at a temperature of 105°C for 100 minutes. Then filtered and washed with distilled water until the pH is neutral (same as the pH of the distilled water used for washing). After that, it is dried and the desired MCC is obtained[12,17].

5. MCCDSr qualitative test using Zinc Iodinate

Solution of zinc iodinate was prepared by dissolving 20 g of zinc chloride and 6.5 g of potassium iodide in 10.5 ml of water, followed by the addition of 0.5 g of iodine, and shaking for 15 minutes. About 10 mg of cellulose was added to the watch glass and then

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dissolved in 2 ml of zinc chloride solution (USP, 2007). The compound formed will produce a purple color[9].

Data Analysis

This study uses data analysis with qualitative and quantitative descriptive analysis. Qualitative descriptive analysis was used to describe the research results from laboratory tests. While the results of the data in the form of numbers obtained were analyzed using Microsoft Excel.

RESULTS AND DISCUSSION

Soursop leaves are chopped, and cleaned by washing with running water and then drying in direct sunlight in the open and repeated for a week. Soursop leaves are mashed using a blender to get a smooth soursop leaves fiber. The preparation is intended to facilitate the isolation of cellulose so that optimal yield is obtained[19].

Extraction

The process of thickening the extract and collecting the dregs from the rest of the extraction can be seen in Figure 1 below:

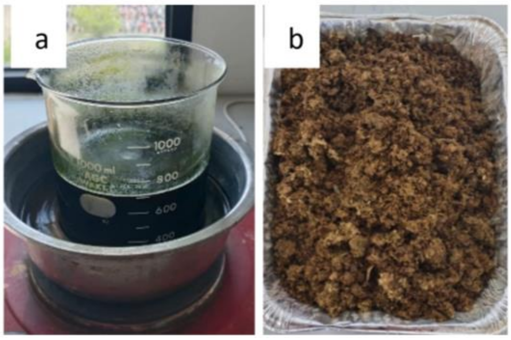

Figure 1. (a) Thickening of the extract; (b) Drying and collection of residue (dregs) after extraction

Table 1. Yield of Extract

	Weight (gram)	Yield (%)
Simplicia Powder	397,43	100
Extract	51,35	12,92

Isolation of α-Cellulose

In the α-cellulose isolation process, when alkaline heating (chemical delignification) uses 17.5% w/v Sodium Hydroxide (NaOH) solution, brown cellulose pulp or slurry is formed which settles in sodium hydroxide solution. The bleaching process aims to remove residual lignin in the pulp. The bleaching process will make the pulp color brighter or whiter. The solution was filtered and the residue was rinsed with distilled water until the pH returned to neutral (same as the pH of the water for rinsing). Then the pulp is dried in an oven at 50° C for 12-24 hours.

Table 2. Yield of α-cellulose from simplicia

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powder

	Data 1 (gram)	Data 2 (gram)	Mean (gram)	Rendemen (%)
Simplicia Powder	100	100	100	100
α-Cellulose	17,73	18,07	17,90	17,90

Preparation of MCC of Soursop Leaves (MCCDSr)

The dried pulp was boiled with 1 M HCl solution. During the hydrolysis process, there was a partial separation of the constituents of the cellulose microfibrils where the amorphous form would break up and leave a crystalline form, namely regions of regularly arranged cellulose molecules. This process aims to cut the polymer into smaller (micro) sizes with a small degree of polymerization where n≈220 so that microcrystalline cellulose is produced[20]. The filtered microcrystalline cellulose residue was then dried using an oven at 50° C for 12-24 hours. The yield produced from the manufacture of microcrystalline cellulose was 96.59% of α-cellulose.

The results of α-cellulose from the chemical delignification process and MCCDSr results from the chemical hydrolysis process can be seen in Figure 2 below:

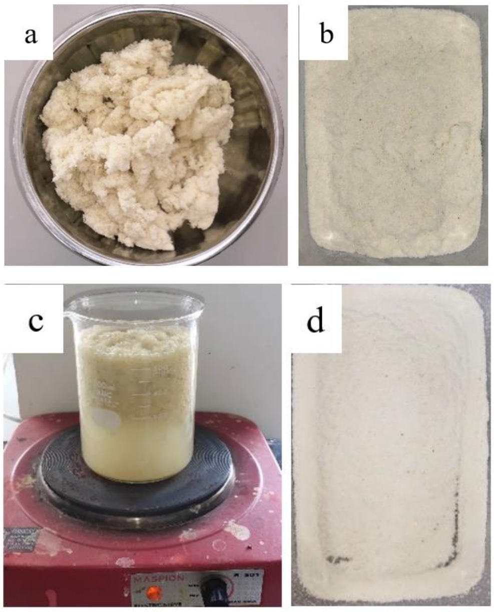

Figure 2. (a) α-cellulose after blending (unfiltered); (b) α-cellulose after filtering; (c) Hydrolysis process; (d) hydrolyzed MCCDSr.

Table 3. Yield of MCCDSr from α-Cellulose

	Data 1 (gram)	Data 2 (gram)	Mean (gram)	Rendemen (%)
α-Cellulose	17,73	18,07	17,90	100
MCC	17,13	17,44	17,29	96,59

Table 4. MCC Powder Quality Comparison

	MCCDSr	Avicel® PH 101	USP 37-NF 32
Organoleptic	Form: crystal Color: white Smell: no smell Flavor: no taste	Form: crystal Color: white Smell: no smell Flavor: no taste	Form: crystal Color: white Smell: no smell Flavor: no taste
Qualitative Identification	Violet-Blue	Violet-Blue	Violet-Blue
Solubility	Not soluble in water Not soluble in 95% alcohol Not soluble in HCl 2N Difficult to	Not soluble in water Not soluble in 95% alcohol Not soluble in HCl 2N Difficult	Not soluble in water Not soluble in 95% alcohol Not soluble in HCl 2N Difficult

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	dissolve in NaOH 1N Insoluble in Ether	to dissolve in NaOH 1N Insoluble in Ether	to dissolve in NaOH 1N Insoluble in Ether

Organoleptic Test

The result of an organoleptic evaluation of the quality of microcrystalline cellulose from the appearance of shape, color, smell, and taste revealed that it had the same qualities as the comparison and literature (Avicel PH 101), namely crystal powder that was white, odorless, and tasteless. Organoleptic analysis revealed that the isolated substance was genuine microcrystalline cellulose.

Based on Figure 2a, it can be seen that the α-cellulose produced has a cotton-like texture, somewhat similar to the results of previous studies using kapok fruit skin[8], and with this texture makes the sieving process difficult because between The powder is very light and agglomerates. So that the process of refining and sifting must be done repeatedly in order to obtain the results of α-cellulose as shown in Figure 2b above. According to Werner's Nomenclature of Color by Syme (1821), the color on Avicel PH 101 is White No. 1 (Snow White), the color of the α-cellulose sample of the MCC sample is White No. 4 (Yellowish White)[21].

Qualitative Test

Based on Figure 2a, it can be seen that the α-cellulose produced has a slightly yellowish-white color. From the qualitative tests carried out, the results of the color change in the microcrystalline cellulose samples due to the administration of iodinated zinc chloride, microcrystalline cellulose which was originally white turned into a violet-dark blue color dispersed, the same thing happened to Avicel® PH 101 as a comparison and has been in accordance with the literature. From the identification results, it can be concluded that the isolated compound is true microcrystalline cellulose.

The results of the solubility test of microcrystalline cellulose powder showed that microcrystalline cellulose was insoluble in the four tested solvents, namely water, 95% alcohol, 2 N of HCl, 1 N of sodium hydroxide, and ether. Microcrystalline cellulose is difficult to dissolve in solvents because of the strong hydrogen bonds between hydroxyl groups in adjacent bond chains in the crystalline structure of microcrystalline cellulose[22,23]. The results of the

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microcrystalline cellulose solubility test were the same as Avicel® PH 101 and were in accordance with the literature (Table 4). This indicates that the isolated compound is true microcrystalline cellulose.

In pH testing, microcrystalline cellulose has a pH of 7.5 while Avicel® PH 101 has a pH of 7.4. The pH test results of microcrystalline cellulose were in accordance with the pH range listed in the literature, namely pH 5.0-7.5.

CONCLUSION

Based on the research conducted, the extract yield was 12.92%, and from the chemical delignification process of 100 grams of simplicia powder using 1M HCl, it was obtained that α-Cellulose was 17.90 grams or with a percentage of 17.90%, while the hydrolysis process α-Cellulose obtained MCCDS as much as 17.29 grams or with a percentage of 96.59%. Based on the research that has been done, the characteristics of microcrystalline cellulose powder from soursop leaves are similar to Avicel® PH 101.

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