

Analysis of Powder Properties and Pharmacopeial Specifications of Bagasse Cellulose Isolated from *Saccharum Officinarum* L in Indonesia

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Abstract

Saccharum Officinarum L is native to Asian countries, including Asia. Indonesia is the third-largest sugarcane producer in the ASEAN region. The amount of sugarcane production represents the potential amount of bagasse waste obtained. Bagasse has not been used properly and optimally in Indonesia. Bagasse contains 43.6% cellulose; hemicellulose 33.8%; lignin 18.1%; ash 2.3% and wax 0.8%. The purpose of this study was to isolate bagasse cellulose with 5% of nitric acid. We determined the organoleptic powder properties and pharmacopeial specifications of bagasse cellulose as pharmaceutical raw materials. As a result, the test parameters as pharmaceutical raw materials for bagasse cellulose met the requirements compared to Japan Pharmacopeia XV except for the ash content value. Bagasse cellulose powder had a white color, a water content of 5.93%, and pH 6.7. It was insoluble in water-ethanol and ether and had Pb-Cr-Cd metal content <10 ppm. The microbial limits were below the specified requirements. The ash content was 0.6, the bulk density was 0.136, the tapped density was 0.158, Carr's index was 13.92, the Hausner ratio was 1.162, and the angle of repose was 56.4. Based on the angle of repose value, bagasse cellulose powder had fairly good flow properties.

Keywords: Powder Properties, Pharmacopeial Specifications, Bagasse Cellulose, *Saccharum Officinarum* L

Introduction

Sugarcane (*Saccharum Officinarum* L) is a plant native to Asian countries with tropical and subtropical climates, such as Thailand, Philippines, Indonesia, Brunei Darussalam, Laos, and Vietnam^{1,2}. The largest production of *Saccharum Officinarum* L is located in Brazil, controlling about 43% of global production, while in the ASEAN region, Thailand is the largest sugarcane producer. During the period 1980 to 2013, there was an increase in sugarcane production in the ASEAN region (an average of 4.28% per year). In 1980 sugarcane production in ASEAN countries was only 5,080,234 tons, but at the end of 2013 sugarcane

production in ASEAN was recorded to have increased by 16,378,700 tons^{2,3}.

Indonesia ranks the 10th largest in the world and the 3rd largest in ASEAN as a sugarcane producing country after Thailand (49.51%) and the Philippines (17.77%) with a percentage of 16.05%. Sugarcane plantations in Indonesia can be found scattered in West Java, East Java, Central Java, DI Yogyakarta, North Sumatra, and South Sumatra⁴. Sugarcane production in Indonesia in 2006 was approximately 64,169 tonnes⁵. Indonesia can produce an average of 27.88 million tons of sugarcane in the period 2009 - 2013³.

Sugarcane is a plant that is the primary source of raw materials for sugar and monosodium glutamate (MSG). Sugarcane is processed by extracting the juice by squeezing the sugarcane stalks to get the extraction in the form of bagasse waste. The amount of sugarcane

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production in both the world, ASEAN, and especially Indonesia represents the large potential amount of bagasse waste produced.

Bagasse is the residue of the sugarcane milling process that has been squeezed or extracted from the sugar-making industry and alcohol^{6,7}. Bagasse is a fibrous residue from sugarcane that has gone through the juice extraction process (taking the water phase from the sugarcane stalks)⁸. The main component of bagasse is fiber (43-52%) as a byproduct of sugarcane juice extraction⁶. From one sugar factory, industrial byproducts can be obtained in the form of bagasse up to 35-40% of the weight of milled sugarcane⁹. Referring to the 2013 projection of sugarcane production in Indonesia, which reached an average of 27,880,000 tons, it can be calculated that the amount of bagasse produced in Indonesia is 11,152,000 tons on average. The amount of bagasse waste is estimated to increase because sugar production in Indonesia until 2020 is estimated to increase by the Ministry of Agriculture³.

The existence of bagasse as waste can disturb the environment if there is no proper and optimal treatment or utilization. Generally, bagasse waste is used directly as fuel for the sugar industry, animal feed, and organic fertilizers⁵. The economic value of utilization is still low and is considered not a productive way of handling waste.

Bagasse has been used as a composite, textile material, pulp, and paper industry because of its cellulose content¹⁰. Bagasse contains 43.6% cellulose; hemicellulose 33.8%; lignin 18.1%; ash 2.3% and wax 0.8%^{11,12,13,14,15,16}. Another component in bagasse is hemicellulose, as much as 25-35%, which is an amorphous polymer and mainly consists of xylose, arabinose, galactose, and mannose¹³. The remainder is mostly lignin (18-24%). Based on the cellulose composition, bagasse has the potential for the development of great benefits and high economic value.

Cellulose and nano cellulose are biopolymers that are abundant in nature and are renewable biomaterials that can be processed into various environmentally friendly polymer products²⁰. Cellulose is applicable in the pharmaceutical industry, in the pharmaceutical, cosmetic, food, and beverage industries. The application has been studied by many researchers¹⁷. Cellulose is

used as a filler in capsules, disintegrants, and binders in tablets^{18,19}.

Cellulose has versatile uses in many industries such as cattle food, wood, paper, fiber and clothing, cosmetics, and pharmaceuticals as excipients¹⁷. The functions of cellulose in the pharmaceutical industry are very diverse. Cellulose is applicable as a filler for capsules at a concentration of 5-30%. Besides, cellulose is suitable as a binder in tablets with a concentration of 5-40% (wet granulation) and (10-30%) dry granulation. Cellulose is proper as a disintegrant tablet with a concentration of 5-20% and as a glidant tablet with a concentration of 1-25%¹⁸.

Cellulose is available in nature. Lignocellulosic biomass is the form of cellulose. It is the combination of lignin, cellulose, and hemicellulose^{14, 15, 21}. The primary source of cellulose is a natural material. It contains plant cell walls, acetic acid bacteria, some animals (tunicate), some algae, and oomycetes. Cellulose can be isolated from pineapple peel juice to produce bacterial cellulose²², palm oil mesocarp fiber²³, potato skin waste²⁴, palm sugar fiber²⁵, sago²⁶, cassava pulp^{27, 28} and bagasse^{29,30}.

To sum up, the researchers used bagasse in this study because of its abundance in nature, lack of economic value, and a large percentage of cellulose. This study aimed to isolate cellulose from bagasse waste. The obtained cellulose was examined as a pharmaceutical raw material according to pharmacopeial specification parameters such as pH, moisture content, solubility, test powder properties such as bulk density, tapped density, Carr's index, Hausner ratio, and angle of repose, microbial test, an assay of metal weight content³¹.

Materials

The materials were bagasse from the home industry, Hydrochloric Acid (Merck), Nitric Acid (Merck), Sodium Hydroxide (Merck), Sulfuric Acid (Mallincrodt USA), Aquades, Hydrogen Peroxide (Sigma), and Alpha Cellulose (Sigma).

Methods

Sampling

Bagasse was available from the remaining sugarcane. It has been extracted from the brown sugar industry in

Kediri, East Java, Indonesia. The sugarcane age had entered the planting period of 6-8 months. Sampling was done by a simple random sampling technique.

Preparation of Sugarcane Powder

A total of 28 kg of fresh bagasse, washed and dried, then chopped and milled to produce a coarse powder of bagasse. The obtained bagasse coarse powder was then processed with a 100 mesh sieve. The powder obtained was stored in a closed container and stored at room temperature.

Bagasse Cellulose Isolation

The bagasse was first hydrolyzed with nitric acid at 80°C for 2 hours. The washed residue was added

with 2 N sodium hydroxide (alkaline hydrolysis) and hydrogen peroxide for the bleaching process under the same conditions¹⁵. The bagasse cellulose was tested organoleptically, moisture content, pH, solubility, aerobic bacteria content, fungal content, metal content, bulk density, tapped density, and angle of repose.

Results and Discussion

Preparation of Bagasse Coarse Powder

Bagasse waste used in this research was from the brown sugar home industry. The home industry produces byproducts in the form of bagasse as much as 3675 kg per day. The color of the bagasse before preparation was light to dark creamy with a distinctive aroma of sugarcane. The sample used in this study was a fresh sample or freshly squeezed juice (Figure 1)



Figure 1 Bagasse waste at the location of the brown sugar home industry in Kediri, East Java, Indonesia

The fresh bagasse was dried in the sun and baked. The dry bagasse was crushed using a grinder to become sugarcane dregs powder. The bagasse powder used in this study was the powder that passed the 100-mesh sieve. The bagasse powder was creamy in color and had a fiber texture. The powder obtained was then tested for moisture content (Table 1) and stored in a closed jar with silica gel.

The moisture content of bagasse powder was more than the water content after the storage period after the

first observation. It was known based on the weight when wet. Several factors influenced the weight difference, such as the quality of the initial bagasse, the sap content in the fresh sugarcane stalks, and the juice squeezing process. It can leave the sap residue even though it is squeezed by a machine. The drying and storage period caused the remaining water to evaporate more so that the water content decreased. The moisture content of bagasse powder (6.61%) met the quality standard, which was less than 10%.

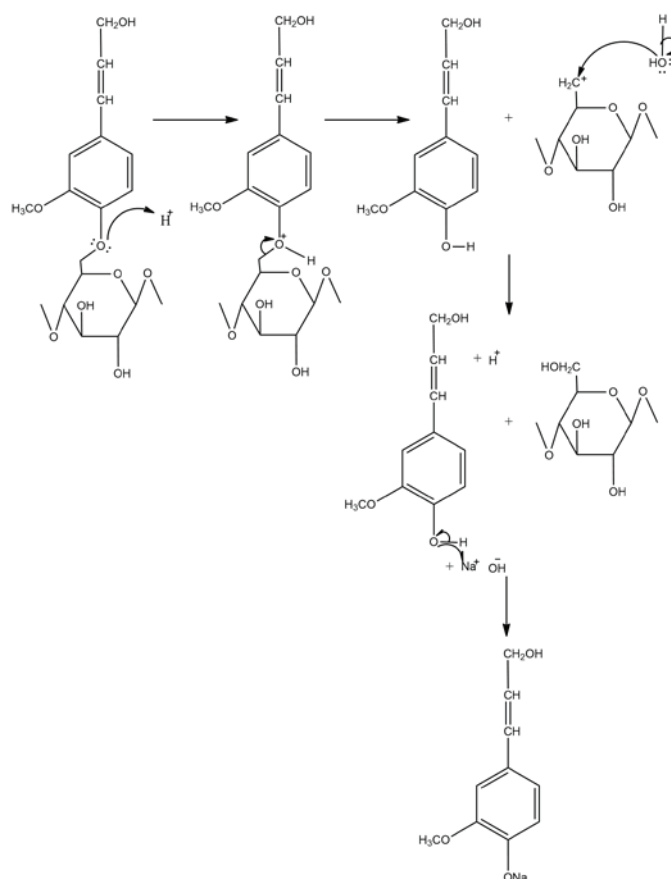
Table 1. Water Content Test of Bagasse Powder

No.	Water Content of Bagasse Powder (%)			Mean	SD
	1	2	3		
1	6.75	6.53	6.56	6.61	0.11

Isolated Bagasse Cellulose

Bagasse is lignocellulosic biomass whose main constituents are cellulose, lignin, and hemicellulose^{14,16}. Lignin is a heteropolymer with a complex structure and composed of several components. It is water-insoluble, impermeable, and resistant to microbial attack and oxidative stress³¹. Hemicellulose is a compound with a complex structure. It consists of several polysaccharides, such as the pentose (xylose, arabinose) and hexose (mannose, glucose, galactose). The hemicellulose average molecular weight is about 30,000 g / mol and is a component of the co-lignin building blocks of cell walls²³.

The addition of 5% nitric acid to bagasse powder aims to open the gaps in the removal of lignin and hemicellulose to produce cellulose^{33, 34, 35}. The addition of acid can break the intrachain bonds between hemicellulose and cellulose in bagasse³⁶. Hydrolysis occurs in ester bonds between hemicellulose and lignin. It also arises in hydrogen bonds between hemicellulose and cellulose³⁷. The hydrolysis process in acidic and hot conditions can soften the lignin structure that coats or protects hemicellulose so it provides pore space for acid to enter³⁶.

**Figure 2 Mechanism of Bonds Termination of Ether between Lignin and Cellulose**

The use of alkalis (NaOH) aimed to optimize the delignification process after breaking the lignin-cellulose ether bond due to acid hydrolysis (Figure 2). The lignin-hemicellulose ester bond was weak or less stable to alkalis, so it was easily disturbed by alkalis (Figure 4). Alkali and lignin that were free can bind to form a soluble lignin-alkali complex so they were

easily detached. Sodium hydroxide can also dissolve hemicellulose, making it easy to remove.

Isolation of bagasse cellulose by hydrolysis of 5% nitric acid produced cellulose which was organoleptically by standard cellulose in the form of white powder (Figure 3). The yield of cellulose obtained was 41.93 with a standard deviation value of 0.01.

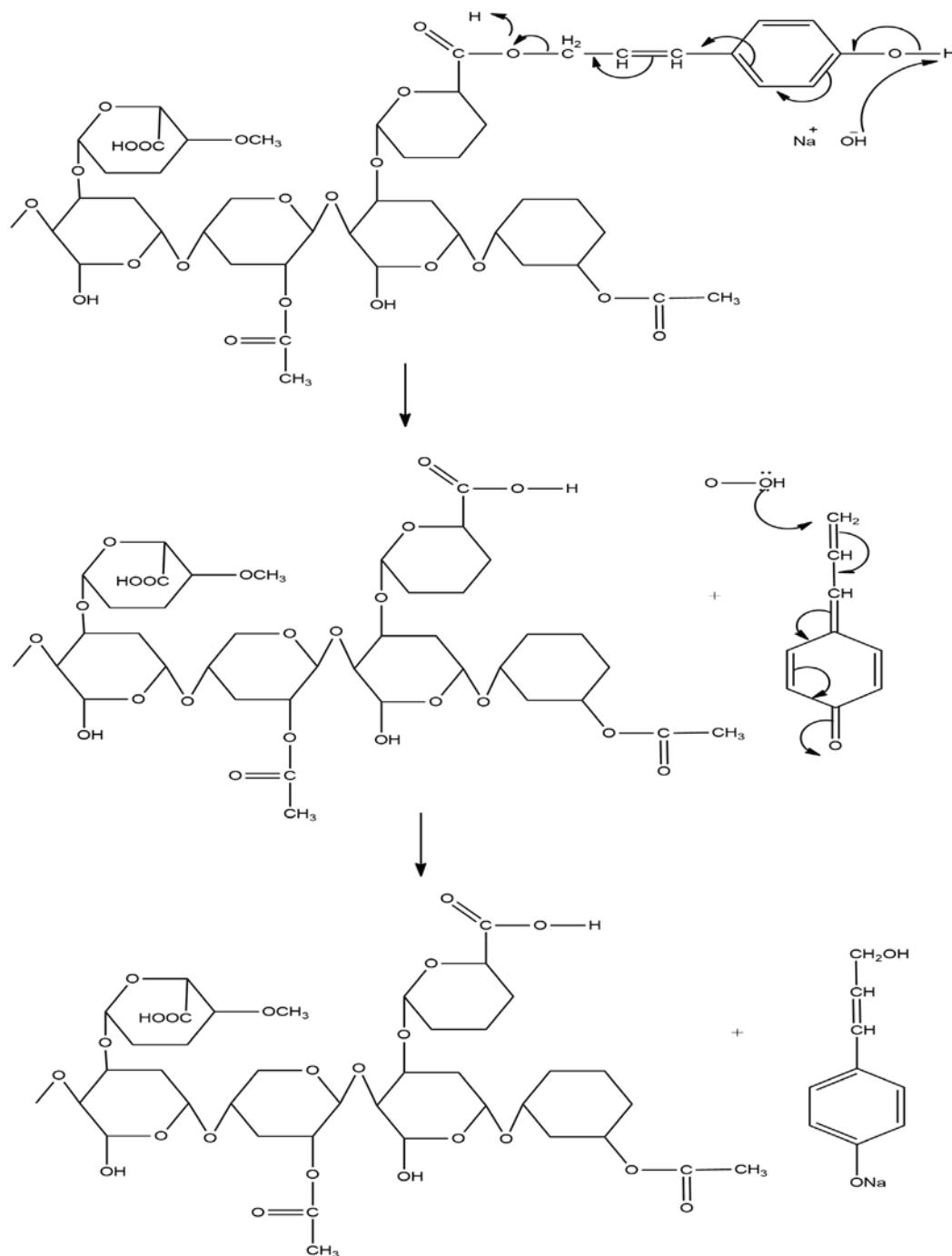


Figure 3 Mechanism of Bonds Termination of Ester between Lignin and Hemicellulose

Test on the pH of Bagasse Cellulose

We measured the change in pH during the isolation process of bagasse cellulose (Table 2). The pH of bagasse cellulose powder is 6.7. The pH value of cellulose

is in the range of 5-7.5, stated by the pharmacopeial specifications for cellulose powder listed in the Japan pharmacopeia XV. Thus, the bagasse cellulose obtained met the specified pH requirements.

Table 2. The pH Value at the Isolation Stage of Bagasse Cellulose

Stage	pH	Physical Change
Bagasse + Nitric Acid 5%	Very Acid	Orange
Residue Washed with Distilled Water	6.6	Orange
Residue + NaOH	11.8	Red brown
Residu Washed with Distilled Water	7.2	Dark cream
Residue + H ₂ O ₂	2.8	White
Residue Washed with Distilled Water (Bagasse Cellulose)	6.7	White

Test on the Water Content of Bagasse Cellulose

Water content is the amount of water contained in a material, such as soil, simplicia, rocks, and agricultural materials. It is one of the quality standard requirements for a simplicia or extract. The acceptable water content of the simplicia or extract is not more than 10%³⁸. According to Japan Pharmacopeia XV, the water content of cellulose is ≤ 6.5 (%). The water content of bagasse cellulose obtained in this study was in line with the requirements, namely 5.93%.

Table 3. Water Content of Bagasse Cellulose

No.	Water Content of Bagasse Cellulose (%)			Average	SD
	1	2	3		
1	5.93	5.93	5.93	5.93	0.00

Test on the Powder Properties of Bagasse Cellulose

The obtained values of bulk density and tap density of bagasse cellulose were 0.136 g / cm³ and 0.158 g / cm³. There was no significant difference (large) compared to the cellulose requirements in the pharmacopeia where

bulk and tap densities were 0.15-0.39 g / cm³ and 0.21-0.48 g / cm³ [18].

The value of the Hausner ratio, Carr's index, and angle of repose determine how the flow properties of the cellulose are obtained. The obtained bagasse cellulose

had a Hausner ratio, Carr's index, and angle of repose, which were not significantly different from commercial cellulose. Based on the Carr's index value, bagasse cellulose had good flowability. It was based on the value of angle of repose met pharmacopoeial requirements, namely $<62^{39,18}$.

The difference was the effect of the used source of the material for the manufacture of commercial cellulose. Standard cellulose is another plant that is not fibrous (generally wood). The difference in source material affects the physical and chemical characteristics of the isolated cellulose.

Table 4. Powder Properties Test on Bagasse Cellulose

Cellulose	Bulk Density	Tap Density	Hausner Ratio	Carr's Index	Angle of Repose
Commercial	0.217	0.263	1.212	17.49	53.1
Pharmacopoeia	0.15-0.39	0.21-0.39	-	-	<62
HNO ₃	0.136	0.158	1.162	13.92	56.4

Pharmacopoeial Specifications of Bagasse Cellulose

Based on all the parameters listed in the Japan pharmacopoeia XV, the test parameters of cellulose bagasse hydrolyzed from 5% nitric acid were completely appropriate except for the value of the ash content. The value of bagasse cellulose ash content was

greater than the standard because the raw material for the manufacture of isolation comes from a more fibrous plant. Manufacturing generally uses wood pulp, which is relatively less fibrous than bagasse (Table 5). The ash content can be eliminated by increasing the washing process at each isolation stage.

Table 5. Comparison of Pharmacopoeial Specifications of Bagasse Cellulose with Standard Cellulose of Japan Pharmacopoeia XV

Test	Selulosa Bagasse	Selulosa JP XV
Identification	+	+
Color	White	White
Microbial Limits a. Aerob b. Fungi and Yeast	a. 200 cfu/g b. No Growth	a. 10^3 cfu/g b. 10^2 cfu/g
pH	6.7	5.0-7.5
Water Content	5.93%	$\leq 6.5\%$
Ash Content	0.6	$\leq 0.3\%$

Cont... Table 5. Comparison of Pharmacopeial Specifications of Bagasse Cellulose with Standard Cellulose of Japan Pharmacopeia XV

Solubility		
a. Water	a. Not Dissolved	-
b. Ethanol	b. Not Dissolved	
c. Ether	c. Not Dissolved	
Water Soluble Substance	Not Dissolved	≤15.0 mg
Heavy Metal	Pb = <0.0016 ppm Cr = <0.0168 ppm Cd = <0.002 ppm	≤10 ppm

Conclusion

Based on a series of parameter tests on bagasse cellulose isolated with 5% nitric acid, it can be concluded that bagasse cellulose generally met the requirements as a raw material for cellulose pharmaceutical preparations except for the ash content value. In the isolation stage, the washing intensity can be increased by removing the remaining ash content. Based on the angle of repose value and the powder properties test results, the bagasse cellulose powder has quite good flow properties.

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Conflict of Interest : The authors declare that there is no conflict of Interest or nil.

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