

Comparison of microcrystalline characterization results from oil palm midrib alpha cellulose using different delignization method

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Abstract. Oil palm midrib is one of the waste generated by palm plants containing 34.89% cellulose. Cellulose has the potential to produce microcrystalline cellulose can be used as an excipient in tablet formulations by direct compression. Microcrystalline cellulose is the result of a controlled hydrolysis of alpha cellulose, so the alpha cellulose extraction process of oil palm midrib greatly affect the quality of the resulting microcrystalline cellulose. The purpose of this study was to compare the microcrystalline cellulose produced from alpha cellulose extracted from oil palm midrib by two different methods. First delignization method uses sodium hydroxide. Second method uses a mixture of nitric acid and sodium nitrite, and continued with sodium hydroxide and sodium sulfite. Microcrystalline cellulose obtained by both method was characterized separately, including organoleptic test, color reagents test, dissolution test, pH test and determination of functional groups by FTIR. The results was compared with microcrystalline cellulose which has been available on the market. The characterization results showed that microcrystalline cellulose obtained by first method has the most similar characteristics to the microcrystalline cellulose available in the market.

1. Introduction

Indonesia is one of the largest country exporter of palm products and its derivatives, which reached 48% of the world's market share [1]. Oil palm plantations produce sizable solid waste in the form of midrib, resulting from cuts in the harvest and pruning the leaves are routinely performed every six months [2]. So far this waste has not been used optimally, only used as fodder [2], compost, even just as firewood. So that needs treatment to overcome the accumulation of waste and increase the economic value of the oil palm midrib.

Palm frond containing cellulose (31.7%), hemicellulose (33.9%), lignin (17.4%), and silica (0.6%) [3]. The amount of cellulose can vary in different natural sources Depending on the species and age of the plant [4]. Cellulose can partially depolymerization forming microcrystalline cellulose (MCC) is conventionally prepared with an excessive amount of mineral acids and it is known commercially as Avicel[®] [4, 5]. Microcrystalline cellulose is used in several area such a pharmaceutical (as binders, adsorbents, flowability), cosmetic (as substitutes, thickeners, binders) food (as stabilizers, anti-caking agents, fat substitutes and emulsifiers), beverage (as gelling agents, stabilizers , anti-caking agents and suspending agents) and polymer composites industries [4]. It can be produced from diverse sources that is high in cellulose. Microcrystalline cellulose can be extracted from oil palm fibers (such as raw stalk fiber, empty fruit bunches, and spikelet) [6], rice straw [7], corn cobs [8], sugar palm bunches



[9], the back of a water gourd [10], muli bamboo [11], and fodder grass [12] with different isolation methods. Highly purified microcrystalline cellulose from plant cell walls can be isolated with several methods such as acid and alkali hydrolysis, steam explosion, extrusion and radiation-enzymatic [4]. Microcrystalline cellulose has been regarded as the best of excipients in direct compression manufacturing of tablets, reducing sedimentation of dry syrups and suspensions, capsules dry binder, stabilizer, and lubricant good crushers [13, 14]. Microcrystalline cellulose is fine-particle form for powdered or a colloidal form that is a water-soluble polymer.

Based on the above description, the authors are interested in isolating α -cellulose from oil palm midrib by two different methods and turn it into microcrystalline cellulose. Microcrystalline cellulose Obtained by both methods was Characterized separately, Including organoleptic test, color test reagents, dissolution test, pH test and determination of functional groups by fourier transmission infrared (FTIR) and the result compare with commercially microcrystalline cellulose. So that will be produced microcrystalline cellulose alternatively and can address the problem of solid waste from oil palm plantations.

2. Method

This study consisted of two stages, the first stage includes the isolation of alpha cellulose waste from oil palm midrib, isolation using two different methods. The second stage includes the manufacture of microcrystalline cellulose from alpha cellulose and microcrystalline cellulose produced characterized.

2.1 Material

Palm midrib that has been cleaned of leaves taken from Tebing Tinggi, North Sumatra. Avicel Ph-102, hydrochloric acid, nitric acid, sodium nitrite, sodium sulphite, sodium hypochlorite, ethanol, n-hexane, zinc chloride, potassium iodide, iodine, potassium bromide, and distilled water.

2.2 Apparatus

Oven, grinder, hotplate, water bath, drying cabinets, analytical balance, desiccators, cutter, transmission infrared (FTIR) (Shimadzu), other laboratory equipment glassware.

2.3 Preparation of material

Palm midrib cleared of sticks and leaves, dried, split and cut into small pieces, and then mashed into a smaller size (powder). Palm midrib powder refluxed with a mixture of hexane and ethanol (2 : 1) for 6 hours and dried.

2.4 Penghilangan lignin

2.4.1. Method 1

This procedure follows the method of Panyasiri, et.al [16] and slightly modified. A total of 200 g of sample is put into a beaker glass, added 2 L sodium hydroxide 4% and heated for 6 hours at 100°C and stirring occasionally. Then filtered and the residue washed with distilled water until a neutral pH. The residue was bleached with 1.3 L of sodium hypochlorite 3.5% by soaking for 24 hours at room temperature. Filtered and the residue washed with distilled water until a neutral pH.

2.4.2 Method 2

A total of 200g of sample is introduced into a beaker glass, added 1.8 L nitric acid 3.5% (containing 18 mg of sodium nitrite). Then heated at a temperature of 90°C for 2 hours, filtered and the residue washed with distilled water. Furthermore, the residue immersed in a 1,8L solution containing sodium hydroxide and sodium sulfite respectively 2% w / v at 50°C for 1 hour. Filtered again and the residue was washed with distilled water. The residue bleached with 2L sodium hypochlorite 3.5% then heated at 100°C for 10 minutes, taken the residue and washed with distilled water until a neutral pH [17].

2.5 Isolation of α -Cellulose Oil Palm Midrib

Samples has been removed its lignin isolated by 17.5% sodium hydroxide, heated at 80°C for 1 hour. Filtered and the residue washed with distilled water until a neutral pH. Furthermore, bleaching again

with 3.5% sodium hypochlorite for 5 minutes at a temperature of 100°C. Filtered and the residue washed until pH neutral and dried in an oven at a temperature of 60°C [17].

2.6 Preparation of Microcrystalline Cellulose

Weighed as much as 50 grams of alpha cellulose is inserted into a beaker glass and hydrolyzed with 1.2 L HCl 2.5 N by boiling for 15 minutes, then poured in cold water while stirring vigorously with a spatula and let stand for 24 hours. Microcrystalline cellulose produced from this process is washed with water to neutral pH, filtered and dried in an oven at a temperature of 57°C -60°C for 1 h [14, 18].

2.7 Characterization of Microcrystalline Cellulose

Characterization including organoleptic test, color reagents test, dissolution test, pH test and determination of functional groups by FTIR.

2.7.1 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 [15].

2.7.2 Colour Reagents 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 [15].

2.7.3 Dissolution 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%) [15].

2.7.4 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 [15].

2.7.5 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 [14]

2.7.6 FTIR spectroscopy

Weighed ± 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

Yield results of α -cellulose and microcrystalline cellulose of oil palm midrib (MCCOPM) with two different methods do not differ much. The α -cellulose results by method 1 is higher than method 2. However MCCOPM yield results by methods 2 higher than method 1. The yield of alpha cellulose and microcrystalline cellulose of oil palm midrib (MCCOPM) can be seen in Table 1 below.

Table 1. Rendemen α -cellulose and microcrystalline cellulose of oil palm midrib (MCCOPM)

No	Delignization Method	Yield (%)	
		Alpha Cellulose	MCCOPM
1	Metode 1	36,90 ^a	63.08 ^b
2	Metode 2	29,25 ^a	67,48 ^b

Description: ^a to the weight of oil plam midrib powder ^b to the weight of alpha cellulose

3.1 Physicochemical Properties of MCCOPM

The results of the physicochemical properties of the MCCOPM and commercial MCC (C-MCC) can be seen in Table 2. The organoleptics quality of the MCCOPM prepared by two method were in the

form fine granules, white color, odourless and tasteless. It indicated that microcrystalline cellulose had been produced from oil palm midrib was similar with commercial MCC.

Table 2. The physicochemical properties of the MCCOPM

Parameter	Results		
	MCCOPM Metode 1	MCCOPM Metode 2	C-MCC (Avicel)
Organoleptic (form, colour, odor and taste)	fine granules, white, odourless, tasteless	fine granules, white, odourless, tasteless	fine granules, white, odourless, tasteless
Identification with ZnCl_2 iodized solution	blue-violet	blue-violet	blue-violet
Dissolution test	0.20%	0.18%	0.18%
Starch test	None (not blue with iodine solution)	None (not blue with iodine solution)	None (not blue with iodine solution)
pH test	7.2	7.0	7.0

3.2 FTIR Spectroscopy

The recorded of spectra MCCOPM method 1, method 2 and avicel PH 102 show in Figure 1. Each MCC spectrum compared, and showed that MCCOPM prepared by method 1 and method 2 have similar peak with Avicel in the functional groups region and fingerprints region. This indicates that the MCCOPM generated, both of method 1 and method 2 are qualitatively the same.

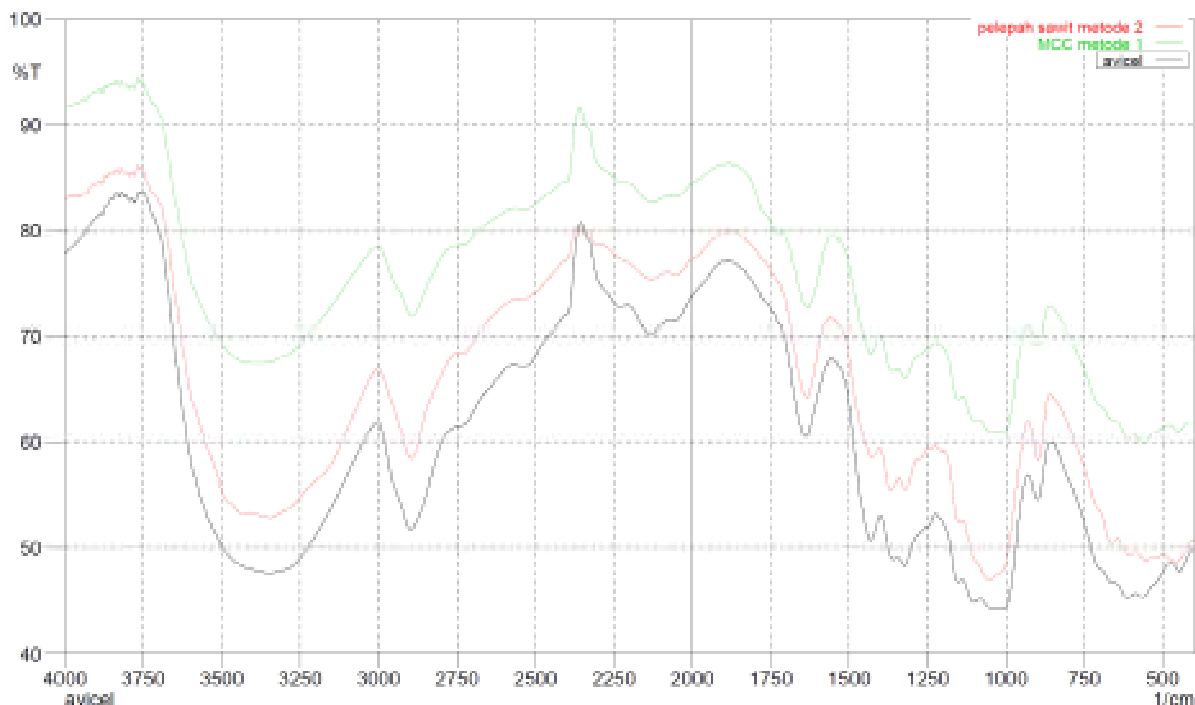


Figure 1. Infrared spectrum of MCCOPM by method 1, by method 2 and avicel Ph 102

A broad absorption band at $3500\text{--}3250\text{ cm}^{-1}$ which indicates the presence of O-H stretching vibrations from α -cellulose. The presence of peak from $2905\text{--}2901\text{ cm}^{-1}$ show that presence of more

crystalline order in MCCOPM. Absorption peak at $1644\text{--}1640\text{ cm}^{-1}$ was due to the strong interaction between cellulose and water as well as the presence of small amount of hemicelluloses [4]. Absence of absorbance peak at 1505 cm^{-1} and $1300\text{--}1200\text{ cm}^{-1}$ proved that the absence of lignin and $1730\text{--}1720\text{ cm}^{-1}$ absence of hemicelluloses [9].

4. Conclusions

The results showed that the oil palm midrib can be used as material for the manufacture of microcrystalline cellulose. Oilpalm midrib is waste that are found in plantations in Indonesia. So that the processing of waste into materials that have a selling value is an important discovery. Microcrystalline cellulose obtained by different delignization methods exhibit characteristics that meets the requirements and has similar characteristics to the microcrystalline cellulose available in the market.

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