

Effect of Isopropanol and Ethanol on Synthesis Carboxymethyl Cellulose (CMC) from Coconut Husk

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Abstract:

Background: ACoconut husk is known as waste from coconut processing that is not optimally utilized, even sometimes just discarded, thus polluting the environment. It contained alpha cellulose that is usually exploited as raw material for the synthesis of carboxymethyl cellulose (CMC) as a pharmaceutical excipient. The solvents commonly used in CMC synthesis are isopropanol or ethanol. This research aims to determine the effect of isopropanol and ethanol as solvents in the synthesis of CMC from coconut husk.

Materials and Methods: Sample preparation, chemical analysis, alpha cellulose isolation, CMC synthesis, and evaluation are all part of this study. CMC was synthesized using two different solvents, isopropanol (F1) and ethanol (F2). CMC was evaluated for yield, moisture content, pH, degree of substitution, purity, viscosity and Fourier Transform Infra-Red (FTIR) then analyzed statistically using the analysis of variance (ANOVA) method.

Results: Based on evaluation, CMC was granular, yellowish, odorless and tasteless. Only F2 meets the degree of substitution requirements with a value of 0.57. Both of the formulas do not meet the purity requirements. FTIR evaluation showed that carboxyl and methyl groups were indicated in both formulas. The result of statistical analysis showed a significant difference (sig 0.05) in the evaluation results of F1 and F2 except for moisture content (sig 0.764) and pH (sig 0.127).

Conclusion: Ethanol usage produced a better quality of CMC than isopropanol.

Key Word: Alpha cellulose, Carboxymethyl cellulose (CMC), Coconut husk, FTIR.

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I. Introduction

Coconut (*Cocos nucifera* L.) includes palm trees that are easy to find around us. Coconuts can grow in various land conditions, so that the spread of this plant is quite wide, especially in tropical climates such as Indonesia. Almost all parts of the coconut can be processed, so that the coconut is also called the tree of life. [1]. Coconuts are usually processed into food or beverage products by taking the meat or water fruit [2]. The by-product of coconut processing is coconut husk. Coconut husk community can be reused into handicrafts, but relatively has low commercial. In addition, many coconut husks are just thrown away without reprocessing. If left continuously without any return process, over time, coconut husks will increasingly accumulate. The accumulation of coconut husk directly disturbs the comfort of the surrounding environment. In the end, it becomes a contaminant material because of its accumulation, decomposition, and other effects that result from it. This contaminant material must be addressed immediately so that it not be severe or widespread.

Coconut husk contains 33.74% alpha cellulose, 49.62% -cellulose, 4.49% ash content and 6.62% concentrate [3]. Based on this data, it is known that coconut husk has a high cellulose content. Cellulose is a polysaccharide that has insoluble properties in water and easily absorbs water, so the cellulose is usually used as a raw material to produce carboxymethyl cellulose (CMC). CMC is widely used in industries for food excipients, cosmetics, and pharmaceutical products. In the pharmaceutical field, CMC is used as a coating agent, emulsifying agent, suspending agent, disintegrant capsule and tablet, tablet binder, viscosity-increasing agent, and water solubility agent [4]. The high content of cellulose in coconut husk makes it possible when coconut husk is made into CMC. Previous research about CMC has synthesized CMC from water hyacinth and corn cob [5,6].

Synthesis CMC is divided into two main processes. They are alkalization and carboxymethylation. Alkalization is the process of alkali reaction which activates hydroxyl groups on cellulose. Cellulose activation will make cellulose structures expand and facilitate the carboxymethyl groups substitution therein [7]. The reaction of carboxymethyl groups that substitution into cellulose is called carboxymethylation [8]. The solvent or reaction medium which may be used is alcohol because of the CMC properties which are insoluble in the organic solvent so that the alcohol will not react subsequently. The commonly used alcohol is isopropanol

because isopropanol is considered the most effective based on the resulting degree of substitution [9]. However, other alcohols may be used, such as ethanol.

First and foremost, the alpha cellulose from cellulose compounds in coconut husk must be isolated so that CMC production is not hampered. Alpha cellulose is a long-chain cellulose that is insoluble in NaOH (17.5%) or a strong base solution [10]. Alpha cellulose is contained within cellulose, which is a very abundant organic compound that is present in the cell walls of plants [11]. Other than cellulose, coconut husk also contains lignin and hemicellulose attached to cellulose. Lignin is a protective layer of cellulosic structures that protects cellulose from enzymes that can break down cellulose [12]. Lignin and hemicellulose are not desirable in the process, so these substances need to be removed in order to obtain cellulose derivate compounds that can be assembled.

Processing coconut husk into pharmaceutical grade in the form of CMC has several advantages, such as increasing the commercial value of coconut husk itself, reducing the waste generated from coconut processing, reducing environmental pollution, and becoming an alternative raw material for synthesis of CMC. Chemical analysis of raw materials as a preliminary phase. Chemical content analysis is needed to characterize the material, thus providing an overview of the nature of the material and facilitating the CMC synthesis phase later. The study's goals and objectives are to determine whether coconut husk cellulose can be synthesized into CMC using two different solvents (isopropanol and ethanol), the differences in using these solvents in the CMC synthesis process, and to identify both the solvents that produce the highest quality. To determine which CMC has the highest quality, the degree of substitution (DS), Fourier Transform Infrared (FTIR), and purity are used to characterize it.

II. Material And Methods

Sample preparation: The coconut husk was put into the oven at 105 °C for 30 minutes. Dry coconut husk milled to reduce the particles to a pulp. After milling, they proceed to the sieving process using mesh 60.

Chemical analysis: The moisture content, ash content, pentosan content, extractive content, alpha cellulose level, solubility in 1% NaOH, solubility in cold water, and in hot water were determined by the TAPPI standard method. The lignin content was determined by the Rancangan Standar Nasional Indonesia 1 (RSNI 1). The holocellulose level was determined by ASTM D1104-56.

Isolation of alpha cellulose:The first process was followed by a delignification process by bleaching the coconut husk powder with a 1% NaClO₂ solution. Other than lignin, the hemicellulose has to be removed using 17.5% NaOH. At the end of the process, coconut husk cellulose was neutralized and washed using acetic acid and warm water [5]. The yield was determined by [6]:

$$\text{Yield} = \frac{\text{weight of dry cellulose (gr)}}{\text{weight of dry sample (gr)}} \times 100\%$$

In addition to yield, alpha cellulose content was also determined by the TAPPI T203 [13].

Synthesis of carboxymethyl cellulose (CMC):The synthesis of CMC was carried out by following alkalization, carboxymethylation, and neutralization. Synthesis CMC used two formulas, F1 and F2. The F1 used isopropanol as a solvent and the F2 used ethanol as a solvent.

Table no1: Formulas of synthesis of carboxymethyl cellulose (CMC)

Formula	Material			
	Sample	Solvent	Alkalization	Carboxymethylation
F1	Alpha cellulose	Isopropanol	NaOH 30%	C ₂ H ₂ ClNaO ₂
F2	Alpha cellulose	ethanol	NaOH 30%	C ₂ H ₂ ClNaO ₂

Coconut husk cellulose 10gr was introduced into a three-neck flask and added isopropanol for F1 and ethanol for F2. The cellulose and solvent were stirred by the disintegrator until all the wet ingredients were combined. The sample was added by 27.5ml NaOH 30% dropwise in stirring with a disintegrator for 1 hour at 25 °C. After the alkalization process was completed, the temperature was raised to 55 C. Furthermore, the material was added by 10 gr sodium mono-chloroacetate and the stirring process continued for 3 hours, known as the carboxymethylation process.

The material is then removed from the three-neck flask and transferred into a filter funnel. The neutralization process was carried out by rinsing the residue with ethanol 96%. Furthermore, CH₃COOH 90% was added until the pH reaches the range of 6.5-8.5, then the residue was dried at room temperature [14]. The crude yield from the result of the synthesis carboxymethyl cellulose process was calculated by [6]:

$$\text{Yield} = \frac{\text{weight of dry CMC (gr)}}{\text{weight of dry cellulose (gr)}} \times 100\%$$

Characterization of carboxymethyl cellulose: The Characterization of CMC was done based on ASTM D 1439-03 [15]. Infrared spectra of CMC were obtained by Fourier transform infrared to identify carboxyl and methyl groups in CMC. Pellets were made using 2 mg CMC samples and mixed with 200mg potassium bromide, then transmitted at a wavenumber range of 4000-400 cm⁻¹ [16].

Statistical analysis: The statistical analysis was used to compare between F1 and F2 whether there were differences in the results of the CMC evaluation performed. The ANOVA method was used to process data. The results were processed by SPSS 18.0.

III. Result

Chemical content of coconut husk

Chemical content analysis in order to characterize the material, thus providing an overview of the material and facilitating the CMC synthesis phase later. The chemical content analysis (Table no.2) includes moisture content, ash content, pentosan content, extractive content, lignin level, holocellulose level, alpha cellulose level, solubility in 1% NaOH, solubility in hot water and solubility in cold water.

Table no 2: Chemical content of coconut husk.

Subject of analysis	Result (%)
Moisture	9.27
Ash	2.98
Hemicellulose	23.82
Extractive	3.16
Lignin	33.56
Holocellulose	75.40
Alpha cellulose	39.21
Solubility in 1% NaOH	19.59
Solubility in hot water	5.54
Solubility in cold water	4.86

Isolation of alpha cellulose

The result of alpha cellulose isolation was an extract yield of 35,95% with an alpha cellulose content of 96.05%. The alpha cellulose content obtained has a high enough concentration to be above 90%, so it was good to be used at the synthesis CMC.

Synthesis of carboxymethyl cellulose (CMC)

Characteristic of synthesis carboxymethyl cellulose (CMC) (Table no.3) from coconut husk include yield, moisture content, pH, degree of substitution, purity, and viscosity based on ASTM D 1439-03 [15]

The spectrum infrared of carboxymethyl cellulose (Fig no.1) was analyzed by Fourier Transform Infra-Red (FTIR).

Table no3:Characteristic of synthesis carboxymethyl cellulose (CMC) from coconut husk.

Formula	Yield (%)	Moisture (%)	pH	Degree of Substitution (DS)	Purity (%)	Viscosity (cP)
F1	173.34 ±7.37	7.27±1.04	8.02±0.12	1.18±0.00	75.66±0.51	11.32±0.62
F2	132.66±3.48	6.91±0.64	7.89±0.09	0.57±0.02	98.40±0.69	86.46±1.36

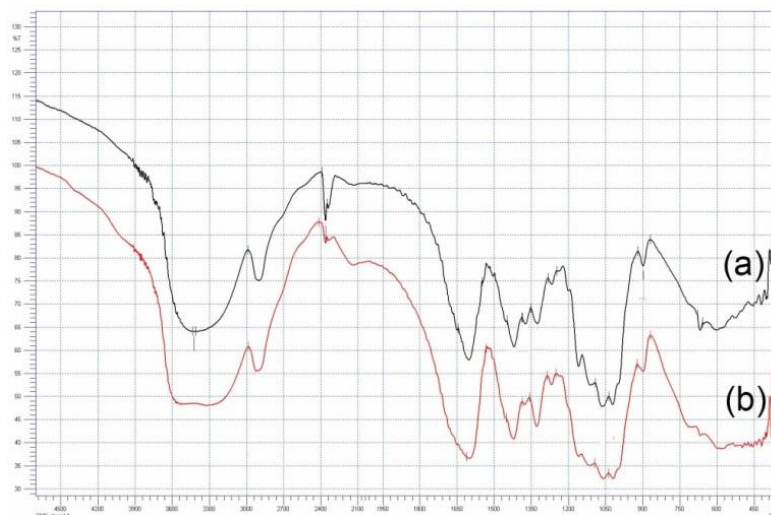


Figure no 1: FTIR spectra of the carboxymethyl cellulose (CMC) (a) F2(black) (b) F1(red).

Table no 4: Assignment of main absorption bond in carboxymethyl cellulose (CMC) from coconut husk.

Wavenumber (cm ⁻¹)		Assignment
F1 (isopropanol)	F2 (ethanol)	
920.00	897.88	1.4-β glycoside
1059.90	1062.80	C-O-C group
1326.08	1325.12	-OH group
1417.70	1418.67	-CH ₂ (indicated CMC)
1608.87	1602.87	-COO (indicated CMC)
2923.17	2901.95	-CH, -CH ₂ , CH ₃
3400.00	3422.74	-OH <i>Stretching</i>

IV. Discussion

Chemical content of coconut husk

The moisture content was calculated to obtain the dry weight of the sample (coconut husk). Moisture content was determined using an oven method with the principle that the water in a material will evaporate when the material is heated at 105°C for 4 hours. Based on the analysis, the moisture content of coconut husk was 9.27%. The moisture content of the sample was below 10%, so it can be said it is feasible to proceed to the next analysis phase. If the moisture content of the sample was too high, it would accelerate the breakdown process of the sample and interfere with another analysis process.

The analysis of ash content was performed to find out about the mineral content in coconut husk. The mineral content might be metal salts, such as carbonates, silicates, oxalates, and phosphates. In general, most of the metal components were calcium, followed by potassium and magnesium. The amount of ash content in a material will be considered in determining the safety level of the material to be consumed. Based on the analysis obtained, the ash content of coconut husk was 2.98%.

The analysis of pentosan levels was performed to find out about hemicellulose in coconut husk. Based on the analysis results, coconut husk contained hemicellulose of 23.82%. Hemicellulose will be a nuisance in the alpha cellulose isolation process. Hemicellulose is a layer that wraps the cellulose, so if the hemicellulose level was high, the hemicellulose package would be thicker and alpha cellulose would be more difficult to separate.

The analysis of extractive content was performed to determine the amount of extractive content contained in coconut husk. Extractive was a substance extracted by a particular solvent carried out at the boiling point, solvent, and within a certain time. Alcohol-benzene was used in the extractive analysis because the sample has a polar and nonpolar bond while alcohol-benzene can dissolve both of them. Based on the analysis, coconut husk has an extractive content of 3.16%. The number of extractives greatly affects the synthesis. High levels of extractives can reduce yields. If the yield was low, it would multiply the alpha cellulose isolation process and increase the chemicals used.

The lignin level of coconut husk was 33.56%. The less lignin contained in the material, the better the quality of the material because lignin is a layer that envelops cellulose after hemicellulose. If the lignin levels were low, cellulose would be easier to isolate. Lignin levels can be removed by the bleaching process.

Holocellulose was a polysaccharide consisting of cellulose and hemicellulose. Based on the analysis, obtained holocellulose levels were 75.40%. The levels of holocellulose can be lowered in order to facilitate alpha cellulose isolation. Holocellulose can also be removed by the bleaching process.

The alpha cellulose was part of the cellulose that would be lost after the cellulose was soaked in 17.5% NaOH solution. Based on the analysis, the content of alpha cellulose in coco husk was 39.21%. The alpha cellulose was a raw material for synthesis of CMC. The higher level of alpha cellulose in a material will increase the yield of extract so the use of chemicals can be reduced.

The solubility performance was analyzed as an overview of raw materials quality in the solubility. Solubility analysis in 1% NaOH solution was performed to determine the number of soluble components, including organic compounds and inorganic compounds such as carbohydrates, tannins, kinin, dyes, and lignin. The results revealed that the solubility of coconut husk in NaOH was 1% by 19.59%. Solubility of coconut husk in hot water is 5.54% and 4.86% in cold water. Materials indicated as soluble in hot or cold water include organic salts, inorganic salts, sugars, xylitol, pectin, tannins, pigments, polysaccharides, and other hydrolyzed components.

Isolation of alpha cellulose

In the process of alpha cellulose isolation, alpha cellulose was separated from hemicellulose and lignin. However, when a substance has a high impurity content, the bleaching process should be repeated until pure alpha cellulose is obtained. The bleaching process is usually done 2 times for materials that have a relatively low hemicellulose and lignin content. The alpha cellulose isolation from coconut husk bleaching was done by 3 times. It has high hemicellulose and lignin.

When the bleaching process was only done by 2 times, the alpha cellulose produced was still yellowish. The yellowish color indicates that the material has not been completely cleaned from the impurities, so the bleaching process was needed until white or almost white. If the concentration of alpha cellulose was low, the sample would not be feasible to be used as a raw material for synthesis CMC because there are still a lot of impurities and, of course, the CMC would not meet the expected performance.

Synthesis of carboxymethyl cellulose (CMC)

The synthesis of CMC consists of 3 steps: alkalization, carboxymethylation, and neutralization. The alkalization was carried out using NaOH 30% that was added dropwise. Alkalization aims to open the cellulose structure in order to be substituted by carboxyl groups. The addition was done by dropwise to get the perfect reaction. After the structure of cellulose was opened, the carboxyl groups were substituted with those donated by sodium mono-chloroacetate. That was called the carboxymethylation process. The last was neutralization, which aims to neutralize the pH and eliminate byproducts or impurities from the reaction.

The use of NaOH and sodium mono-chloroacetate in the synthesis of the CMC process should be appropriate because there is a critical point that will determine the quality of the CMC. Therefore, the use of chemicals in a formula should be approaching a critical point. The CMC that has been synthesized was yellowish. This occurs because of alkalization treatment using NaOH 30%. The yellowish color may be because of a reaction between lignin and NaOH or a heating effect that causes discoloration (browning) during the synthesis process. However, based on the reference, it was still in accordance with the CMC color requirements [4].

Characteristic of carboxymethyl cellulose (CMC)

The percentage of yield from each formula was 173.34% for F1 and 132.66% for F2. Both formulas have a yield above 100% because of weight gain from the addition of sodium mono-chloroacetate to cellulose as much as 1: 1, so that yield is close to 2-fold. This yield was not quantitative. The percentage of yield depends on the accuracy and skill of the researcher when discharging the CMC from the three-neck flask.

The moisture of the two formulas met the requirements of 10% [4]. There was a slight difference in the moisture of both formulas because the drying process was carried out at room temperature. From the statistical analysis, there was no difference in moisture between F1 and F2 was marked with sig value of 0.764 (sig > 0.05).

pH evaluation aims to ensure that CMC does not affect the acidity of other materials mixed with it. Therefore, the pH requirements of CMC are close to neutral (6.5-8.5) [4]. The pH was determined by a pH meter with a concentration of pH 1% [17]. Both formulas meet the pH requirements for CMC. The pH from both formulas approaches the alkaline pH. This might be because sodium mono-chloroacetate was used

excessively, so that NaCl increased and shifted the acidity to the alkaline [8]. Based on statistical analysis, it is known that there was no difference in pH value between F1 and F2 where the sig value was 0.127 (sig > 0.05).

The degree of substitution (DS) was analyzed to determine the amount of carboxymethyl groups that have substituted into the alpha cellulose. DS for CMC as an excipient for drug was 0.55-1.0 [18]. Based on the analysis, the DS for each formula was 1.18 for F1 and 0.57 for F2. Based on the test, only F2 has met the standard value of DS. The difference in DS between F1 and F2 was because of the solvents that were used. A more non-polar solvent would be preferable for the alkalization process. Alkalization was the process of opening the alpha cellulose group to allow the carboxymethyl group to be distributed therein, so in this case, the synthesis of CMC with a more non-polar solvent such as isopropanol (F1) gives a higher than DS. Based on the statistical analysis, there was a significant difference in DS between F1 and F2 and the sig value was 0.00 (sig 0.05).

The purity was analyzed to determine the purity of the synthesized CMC. The impurities referred to in this case were byproducts during the process of alkalization and carboxymethylation reactions, such as sodium glycolate and sodium chloride. The requirement for the purity of the CMC was 99.50%. That means almost no impurity in the CMC [19]. Based on purity evaluation, it is known that F1 has a lower purity of 75.66% than F2 of 98.40%. In this study, F1 has a lower purity because F1 uses isopropanol as a solvent that has a lower polarity than the ethanol that is used in F2. Isopropanol would be better to open the alpha cellulose group and cause more carboxymethyl groups to be substituted, and byproducts from this reaction such as sodium glycolate and sodium chloride will have more effect on the decrease in CMC purity. Both formulas do not meet the purity requirements. The statistical analysis showed a significant difference in purity between F1 and F2 with a value of sig 0.00.

The viscosity was to see the performance of CMC by increasing the viscosity of a substance. This viscosity test was performed using a viscometer Brookfield LVT-type. The LVT type was chosen because the viscometer was able to read the viscosity of the sample that had low viscosity, such as CMC. Based on the analysis, F2 has a higher viscosity of 86.46 cP than F1, which is only 11.32 cP. The viscosity of CMC can be affected by DS and purity.

The cellulose groups that have been substituted by carboxymethyl groups will be more reactive to water and will affect the viscosity of CMC. The longer cellulose chain still bonded with the carboxymethyl group will cause increased viscosity, becoming a thick and thermoreversible solution [7]. In this case, F1 may have a higher DS than F2 but it has a poor purity that decreases the viscosity of F1. Based on the viscosity value, F1 and F2 were included in the low viscosity CMC type [20]. The results of statistical analysis showed a significant difference in the viscosity between F1 and F2 with a value of sig 0.00.

The spectrum infrared of carboxymethyl cellulose was analyzed by Fourier Transform Infra-Red (FTIR). The analysis aims to ensure qualitatively that the carboxymethyl group has substituted into the cellulose group. The carboxyl group and the methyl group were the marker groups of the CMC. The representation of the spectrum infrared is attached in Fig. 1.

The FTIR spectrum (Fig 1 and Table no 4) show that the 1,4- glycoside groups of cellulose were found at wavenumber 897.88 for F2 and 920 for F1. In addition, there is absorption of the C-O-C ether group at wavenumber 1057.97-1062.8 from both formulas. Both formulas show low intensity absorption at wavenumbers of 1325.12 and 1327.05, which indicates the presence of an-OH bonding group. The absorption peaks of the -CH₂ group were detected at wavenumbers of 1417.7 and 1420.6 wherein this group was a marker of CMC. While the absorption peak of the carboxyl group (-COO) was detected in both of the formulas at wavenumber 1599.02 and 1602.87. Then the absorption peak of long chain carbon groups as -CH, -CH₂, and -CH₃ were found at 2901.95 and 2923.17. The absorption peaks of -OH groups were found at 3300-3500 as broad-spectrum intensity. Based on the FTIR analysis, it is known that CMC was already synthesized.

V. Conclusion

Coconut husk can be used as a material for synthesis of carboxymethyl cellulose (CMC). only formula with ethanol solvent (F2) fulfills the degree of substitution requirement of 0.57. Both formulas indicated carboxyl and methyl groups but did not meet the requirements for purity. Getting lower the polarity of the solvent can increase DS but decrease the purity of CMC. Based on statistical analysis, there was a significant difference (sig 0.05) between isopropanol and ethanol as the solvent in each evaluation except for the moisture content (sig 0.764) and pH (sig 0.127). The CMC synthesis process using ethanol as the solvent (F2) provides better evaluation results compared to isopropanol.

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