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Preparation and Characterization of Carboxymethyl Cellulose Production from Oil Palm Male Inflorescence as Food Stabilizer

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Abstract

Male inflorescence residue of the oil palm rich in fibre and contains cellulose content and has the potential to synthesize Carboxymethyl Cellulose (CMC). Male inflorescence is considered necessary removed in the early stages of the oil palm cultivation process to develop stem size, string and robust root systems. This activity generates plentiful of oil palm male inflorescence residues in the plantation. The aim of this research is to synthesis and characterize CMC fabricated from oil palm male inflorescence (OPMI-CMC) waste for use as an ice cream stabilizer, using a variety of treatments, which are commercial CMC 0.2%, OPMI-CMC 0.1%, 0.2%, and 0.3%. The results showed that CMC can be produced from organic waste of male flowers of oil palm plants. The water content, pH, purity, and degree of substitution meet the standard. The optimal concentration of OPMI-CMC for ice cream stabilizer is 0.3% based on melting time and overrun value. The FTIR analysis shows that the main functional group of the OPMI-CMC is highly comparable. Therefore, to possibly be employed as an alternative to organic CMC for food stabilizers, other standards must be fulfilled, and further research is required.

Keywords: CMC, lignocellulose, male flower, oil palm, residues

INTRODUCTION

The area of Indonesian oil palm plantations has continued to grow over the past five years. In 2019, Indonesia had a total land area of oil palm plantation of 16.3 million ha (Ariesca, et al., 2023). The increase in the area of oil palm plantations is followed by a rise in the quantity of solid organic residue produced. One type of oil palm solid residue that is not optimally utilized is Male Inflorescence (OPMI). The OPMI is removed from the oil palm tree and continuously deposition in soil during the oil palm vegetative growth period, therefore the amount of the residue produced approximately 432-797.5 inflorescence per hectare (Ginting & Panjaitan, 2018). The abundant availability of OPMI residue has the potential to be utilized to synthesis carboxymethyl cellulose (CMC) since it contains approximately 35.33% of cellulose (Rahhutami, Handini, & Lestari, 2020).

Carboxymethyl cellulose is considered to be one of the most promising cellulose derivatives which has a linear, long chain, water soluble and anionic polysaccharide chemical structure (Tasaso, 2015), and it is currently widely used in a variety of applications such as food, paper, pharmaceutical, wastewater treatment, bioengineering, energy storage, and so on (Rahman, et al., 2021). CMC is used as a food additive in a variety of food products to improve texture. (Sebayang & Sembiring, 2017) stated that in ice cream, CMC acts as a food stabilizer, improving the physical form and volume of the product while maintaining the level of homogeneity that makes the ice cream soft.

CMC from OPMI potentially be used as a stabilizer to manufacture ice cream. Premium ice cream on the market is usually made from commercial CMC, which relatively contains high amount of dairy fat and low amount of air (Parid, et al., 2021). (Ferdiansyah, Marseno, & Pranoto, 2016) stated that food stabilizers such as gelatin are food additives prone to source from animal ingredients. Economic ice cream, on the contrary, is made from less expensive ingredients, such as vegetable fats, and contains more air. It is critical to convert OPMI cellulose into CMC in order to improve the functional attributes and performance of oil palm biomass residue cellulose. Using organic CMC from OPMI as an ingredient for ice cream has the advantage of ensuring its safety and improving the quality of the ice cream. The usage of organic CMC is in line with (Hidayatullah, 2015), which states that adding organic CMC from palm oil as an ice cream stabilizer affects smooth texture, taste, aroma, and colour.

This study aims to obtain plant-based CMC from OPMI waste as an alternative of safer food stabilizer, to determine the characteristics of OPMI CMC as an effort to diversify food additives, and to determine the best concentration of CMC-OPMI based on the melting time and the overrun value in ice cream.

METHODOLOGY

Materials and Instrumentals

The materials used in this research are oil palm male inflorescence, which was collected from Politeknik KSCWE plantation vocational estate, NaOH (Merck), NaOCl, CH_3COOH glacial (Merck), methanol, deionized water, Isopropanol (Merck), CMC commercial, PP indicator (Merck), HCl 37% (Merck), sugar, dark chocolate, cocoa powder, vanilla essence, whipped cream, full cream liquid milk. The instruments utilized in this study were blender, filter, erlenmeyer, beaker glass, volumetric pipette, hot plate, stirrer, analytical balance, freezer, oven, desiccator, and FTIR analyzer (Prestige 21, Shimadzu).

Methods

OPMI Powder Preparation

The male inflorescence of the oil palm is cleaned and cut into small pieces. The slices were then washed and dried out in the oven at 105°C for 2 hours before being ground with a blender and sieved with a mesh size of 60.

OPMI Powder Cellulose Isolation

Oil palm male inflorescence of as much as 35 g of powder was mercerized with 700 ml of 15% (w/v) NaOH solution at 100℃ for 3 hours. The remaining solids were washed with 100 ml of distilled water 2–3 times, and then the PP indicator was used to determine the free solids from the remaining NaOH. Furthermore, soaking was carried out with 700 ml of distilled water mixed with 14 g of NaCl and 35 ml of 10% CH_3COOH for 1 hour. The washing process was carried out with 100 ml of distilled water 2-3 times. The remaining solid was bleached with 700 ml of 6% NaOCI solution and 700 ml of 3% (w/v) Na metabisulfite solution at 60℃ with a cooking time of 30 minutes each. Following the bleaching process, the pulp (cellulose) was washed 2-3 times with 100 ml of distilled water until it no longer smelled of hypochlorite. The obtained cellulose was then dried in an oven at 50 °C for 3 hours (Adapted from (Ferdiansyah, Marseno, & Pranoto, 2016)

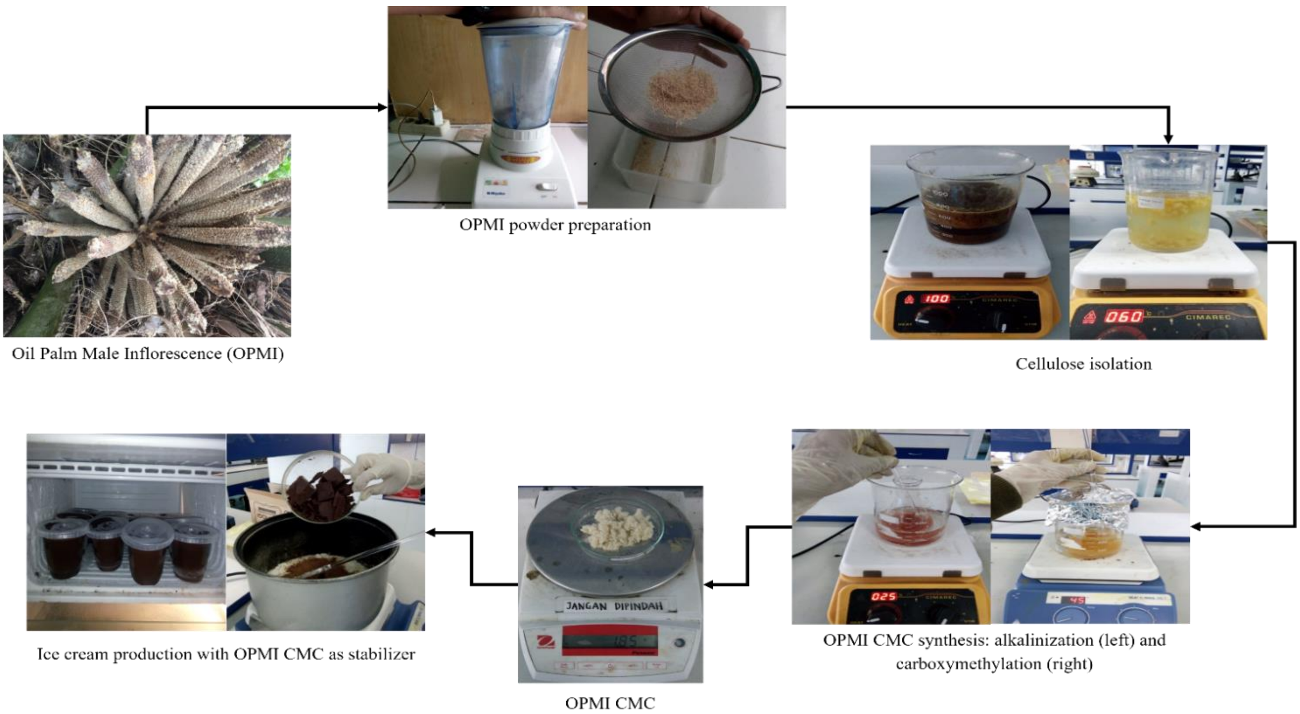

Figure 1. OPMI CMC synthesis and application as food stabilizer

Synthesis of CMC from Palm Oil Male Inflorescence Cellulose

In an Erlenmeyer flask, 5 g of cellulose flour was combined with 100 ml of isopropanol and alkalinized with 20 ml of 10% NaOH solution. The mixture was placed in an Erlenmeyer flask and stirred for 1 hour at 25°C in a beaker filled with water. Following alkalinization, the carboxymethylation process was carried out in an Erlenmeyer at 45°C for 3 hours by adding 4.57 g of NaMCA. The condition was then neutralized with 100 ml of 90% CH_3COOH and a pH indicator at pH 7. After neutralization, the solid was washed with 100 ml of 96% alcohol solution. The solid particles obtained from the filtering were oven-dried at 60°C for 3 hours. CMC of oil palm male inflorescence was acquired by grinding the dried solids (Adapted from (Ferdiansyah, Marseno, & Pranoto, 2016).

CMC-OPMI Purification

A total of 3 g of CMC from oil palm male inflorescence was dissolved in 100 ml of distilled water. The solution was heated for 10 minutes on a hot plate at 80 °C while stirring. After heating, the solution was centrifuged for 1 minute at 4000 rpm. The precipitate was separated from the solution, and CMC from the precipitation was dissolved in 100 ml of acetone. CMC was wrapped in aluminum foil and dried for 4 hours at 60 °C and placed in a desiccator.

Production of Ice Cream

Dry ingredients for ice cream included 87.5 grams of sugar, 125 grams of dark chocolate, 15 grams of cocoa powder, and a teaspoon of vanilla essence were mixed. After that, 300 mL of whipped cream and 150 mL of full cream liquid milk were added to a 40 °C mixing bowl. CMC-OPMI 0.1%, 0.2%, 0.3%, and commercial CMC 0.2% stabilizer variations were used. Three egg yolks were added to each concentration, and the mixture was stirred until homogeneous. The homogeneous mixture was pasteurized for 25 seconds at 80 °C while stirring before being cooled to room temperature. After bringing the mixture to room temperature, it was homogenized for 15 minutes with a mixer before being stored for 4-12 hours in a refrigerator at 4°C. The mixture was homogenized for 15 minutes after it had been aged for 12 hours. Furthermore, the freezing process is carried out for 4-12 hours at temperatures ranging from -5°C to -18°C. The ice cream is then kept in the freezer (Sebayang & Sembiring, 2017).

CMC-OPMI Characterization and Ice Cream Melting Time and Overrun Test

CMC quality analysis based on SNI and FAO standards includes water content, pH, viscosity, purity, and degree of substitution according to Ferdiansyah et al. (2017). The chemical properties of the CMC and CMC-OPMI were evaluated using Fourier Transform Infrared (FTIR) spectrum analysis (Prestige 21, Shimadzu).

For melting test, the 100g ice cream were placed in a beaker glass at room temperature. The stopwatch was turned on and then, it was turned off when the ice cream melts and yields 100 mL liquid in a beaker glass. Then the time takes for the ice cream to melt was recorded adapted from (Sebayang & Sembiring, 2017).

The overrun test was done by calculating the volume of the ice cream before and after the refrigerated. The value of the overrun was taken by comparing the volume of ice cream and the dough mixture. The value is calculated according to the formula below that volume A (ml) is the volume of the ice cream and volume B (ml) is the volume of the dough mixture before refrigerated, adapted from (Muse & Hartel, 2004); (Sebayang & Sembiring, 2017).

$$\%\ \text{Overrun} = \frac{(\text{Volume A} - \text{Volume B})}{\text{Volume B}}\ \text{X}\ 100\%$$

RESULTS AND DISCUSSION

OPMI CMC Synthesis

The OPMI cellulose isolation was carried out using NaOH and bleached with NaOCl, as indicated in Figure 1. Additionally, the extracted cellulose was used in the OPMI CMC's production, which involved etherification with NaMCA and mercerization with 10% NaOH. The loss of hemicellulose and lignin is accelerated by the use of NaOH and NaOCl, according to (Gulati, Park, Maken, & Lee, 2014). Lignin was removed during the bleaching process, which also enhanced carboxymethylation formation. In addition to increasing the contact surface area, the NaOH induces the breakdown of intramolecular hydrogen bonds, weakens the bonds, and causes them to break down. In addition, (Maulina, Adriana, & Teuku, 2019) stated that The etherification process known as carboxymethylation involves attaching carboxylate groups to the cellulose molecule. Monochloroacetate of sodium contains carboxylate groups.

Water Content, pH, Viscosity

OPMI's CMC water content (Table 1) value demonstrated good flow properties and shelf life. This is due to the CMC's hygroscopic nature, which allows it to absorb water from the air quickly. The capacity of water that can be absorbed is determined by the CMC's moisture content. As a result, the shorter the shelf life, the higher the water content. Water content also affects the resistance of foodstuffs to microbial attack (Mulyadi, Susinggih, Ika, & Widelia, 2014). Thus, the CMC water content of oil palm male inflorescence shows its suitability to be applied as a food stabilizer because it has a long shelf life.

Table 1. CMC water content, degree of acidity, and Viscosity

CMC OPMI Parameter	CMC Value	FAO standard
Water content (%)	7.50	≤ 12
pH	6.98	6.0 – 8.5
Viscosity (cP)	14.80	≥ 25

The pH of male oil palm inflorescence Because of the effect of NaOH and NaMCA in the CMC synthesis process, CMC meets FAO standards as a food stabilizer. The pH of OPMI CMC synthesis process with a solution of 10% NaOH and 4.57 g of NaMCA was proven to comply with FAO standards. Furthermore, the concentration of NaOH has a significant impact on the pH produced (Maulina, Adriana, & Teuku, 2019). The higher the pH value obtained, the higher the levels of NaOH and the amount of mass of NaMCA used. Because of its stable solubility level, the pH of OPMI CMC is suitable for use as a food stabilizer (Table 1).

The CMC viscosity of oil palm male inflorescence has yet complied with FAO standards; this is presumably due to the high degree of polymerization of oil palm male inflorescence cellulose. Because of the high degree of polymerization, the molecule's remodeling process was imperfect. A high viscosity value makes it difficult for water to flow and objects to move in the fluid (Farhaniah, 2019). In order to breakdown cellulose and achieve the desired viscosity, high energy and temperature improvements are required.

Purity, Degree of Substitution

Because of the influence of by-products produced during the CMC synthesis process, the purity of oil palm male inflorescence CMC obtained did not conform to FAO standards. The addition of NaMCA reduces the purity of CMC due to an increase in by-products in the form of NaCl. The CMC purity values of OPMI did not differ significantly from the FAO standards (Table 2). This purity value demonstrates CMC's potential application in a broader range of food products, with the need for improvement in the methods of synthesis (Hutomo, 2012).

The degree of substitution of oil palm male inflorescence CMC meets FAO food stabilizer standards (Table 2). This parameter is important because the amount of NaMCA reagent used in the carboxymethylation process determines the optimization of the CMC synthesis reaction. The degree of CMC substitution increases when 5 g of NaMCA reagent is added to 7 g of CMC (Melisa & Nurhaeni, 2014). The amount of NaMCA used influences the substitution of anhydroglucose units, which influences the quality of the resulting CMC. Because of its high-water solubility, the degree of

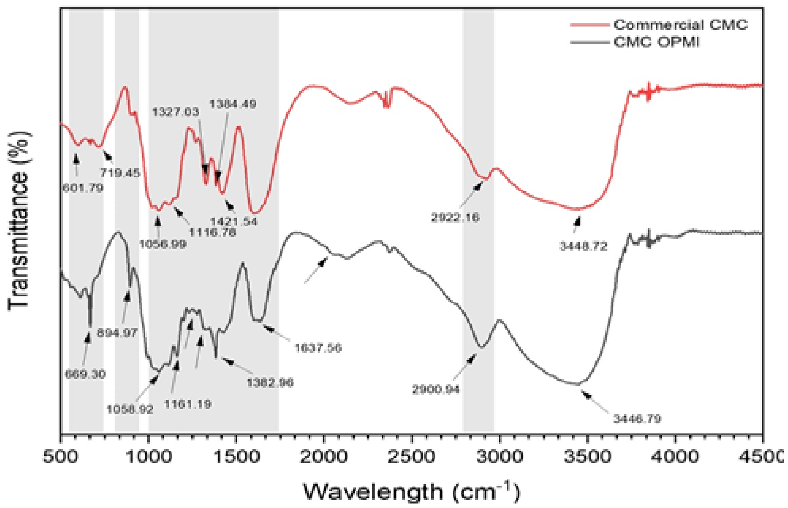

Figure 2. Spectral analysis of the OPMI-CMC and commercial CMC

substitution of CMC in oil palm male inflorescence is suitable for use as a food stabilizer.

Table 2. Purity and Degree of Substitution

CMC OPMI Parameter	CMC Value	FAO standard
Purity	97.67	$\geq$ 99.5
Degree of Substitution	1.47	0.2 – 1.5

FTIR Specta Analysis

The resulting spectra were qualitatively examined for interoperate functional groupings in each sample (Souhoka & Latuperirissa, 2018). The functional group analysis of the OPMI CMC and commercial CMC spectrums revealed the same wave pattern. The vibration of the formed bonding groups is caused by the fact that they are both derived from cellulose. Wave numbers 3446.79 cm^{-1} and 3448.72 cm^{-1} correspond to -OH (hydroxyl) groups found in cellulose (Fadillah, 2018) (Sunardi, Faramitha, & Santoso, 2021). The -C-H (hydrocarbon) group is indicated by vibrations at wave numbers 2900.94 cm^{-1} and 2992.16 cm^{-1} (Erningsih, Yulina, & Mutia, 2011). Wavelengths 1637.56 cm^{-1} and 1600.92 cm^{-1} indicate the presence of -COO (carboxyl) groups, while wavelengths 1382.96 cm^{-1} and 1421.54 cm^{-1} indicate the presence of $-CH_2$ bonds (Lestari, Titi, Siti, & Djagal, 2013). It was shown that there is a difference, namely at the wavelength 1421 cm^{-1} which is only visible in commercial CMCs and not visible in OPMI-CMC. In addition, the wave numbers 1058.92 cm^{-1} and 1056.99 cm^{-1} indicate the formation of a -O- (ether) group (Erningsih, Yulina, & Mutia, 2011). In CMC, the 1153 cm^{-1} band was related with ether bonds (Abidin, Nuriman, Pradipto, & Putra, 2022). Based on those identities, it was demonstrated that the CMC produced by OPMI had functional groups similar to commercial CMCs. The important modification done by alkaline treatment is the disruption of OH bonding in the fiber network structure by ionizing the hydroxyl groups of the various materials in the fibers (Pinto, et al., 2022). Overall, the FTIR spectra of oil palm male inflorescence CMC was found to be comparable to the commercial CMC. It was shown that the OPMI-CMC has the same pattern as commercial CMC and has constituent groups. The wavenumber pattern indicates that OPMI's CMC is safe to use as a food stabilizer. Figure 2 and Table 3 show the FTIR spectra of oil palm male inflorescence CMC and commercial CMC.

Table 3. Spectral analysis of OPMI-CMC and Commercial CMC Analysis

CMC OPMI	Commercial CMC	Functional
3446.79	3448.72	-OH (hydroxyl)
2900.94	2992.16	-C-H (hydrocarbon)
1637.56	1600.92	-COO (Carboxyl)
1382.96	1421.54	$-CH_2$
1058.92	1056.99	-O- (eter)

Ice Cream Quality Analysis

Melting time

The melting time of ice cream is influenced by the CMC application of male oil palm flowers. The 0.3% CMC treatment of male oil palm flowers yielded the fastest ice cream melting time, which was inversely proportional to the 0.2% commercial CMC control treatment. Figure 3 depicts the melting time of ice cream after the addition of male oil palm flower CMC and commercial CMC. The melting time of ice cream with 0.2% and 0.3% male oil palm flower CMC was longer than the control treatment (0.2% commercial CMC). The melting time difference between these treatments was 3.09 minutes and 8.22 minutes, respectively. The melting time difference is due to the fibrous texture of the CMC obtained from the synthesis of male oil palm flowers. The fibrous texture of CMC is thought to strengthen the bonding of the ingredients in the dough, resulting in condensed ice cream. According to (Nuralizah, 2016), the more

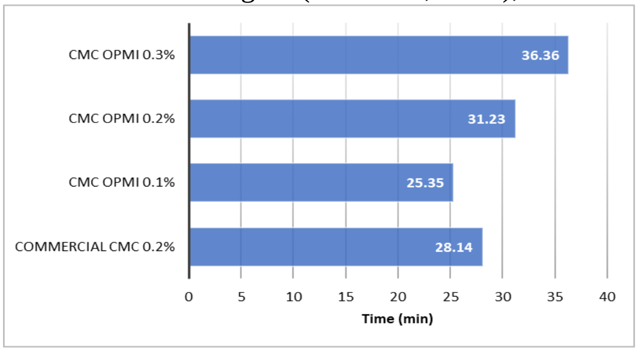

Figure 3. Melting time

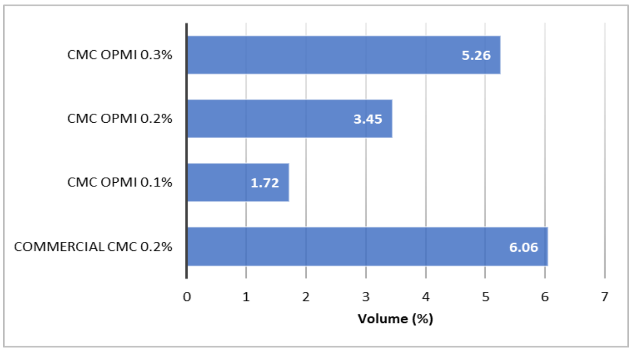

Figure 4. Overrun value.

solids in the ice cream, the slower the melting speed.

The ice cream melting time was reduced to 0.1% CMC of male oil palm flowers, which was not significantly different from the 0.2% commercial CMC treatment. The melting time difference between the two treatments was 3.19 minutes. This difference indicates that the added CMC concentration has an effect on the melting time of ice cream. The solids are added in small amounts to make the ice cream less dense. This result is greater than the melting time of Velva with CMC, which ranges from 2.29 minutes to 4.33 minutes (Maria & Zubaidah, 2014). Overall, ice cream melting time with CMC treatment of male oil palm flowers met the Indonesian National Standard (SNI), which ranges from 15 to 25 minutes.

Overrun Value

The addition of CMC to the ice cream manufacturing process has no effect on the volume expansion (overrun) produced. The ice cream overrun value in the CMC treatment of male oil palm flowers ranged from 1% to 5% and was not notably different from the commercial CMC treatment of 0.2%. Figure 4 depicts the overrun value of ice cream with its incorporation of male oil palm flower CMC and commercial CMC.

The overrun value of ice cream produced by CMC treatment of male oil palm flowers is lower than the Indonesian National Standard (SNI) for home industries, which ranges from 35% to 50%. Because of the fibrous texture of CMC, the ice cream mixture becomes denser, trapping a small amount of air in the dough. Because of the small amount of air trapped in the dough, the ice cream volume does not expand. According to (Dewi, 2010), if too much air is trapped in the dough while freezing, it will result in a higher product overrun.

Other factors, such as the freezing process, could also contribute to the low ice cream overrun value. The ice cream mixture is frozen for the first time in the refrigerator at 4 °C for 4 to 12 hours. This process failed because the freezing time exceeded the specified time, causing the ice cream mixture to freeze too quickly. As a result, the ice cream's volume does not increase during the homogenization process. (Widarona, Suprihartini, Ulilalbab, & Enggar, 2017) discovered that the slow freezing process affects the low level of overrun ice cream. As a result, the freezing process must be improved so that the resulting overrun ice cream meets standards.

CONCLUSION

By utilizing NaOH to alkalinize the cellulose obtained from the oil palm male inflorescence and then monochloroacetic acid to etherify it, CMC can be created from the extracted cellulose. In this study, the preparation and characterization of Carboxymethyl Cellulose from OPMI showed that OPMI potentially be used as an alternative to organic CMC for food stabilizers. CMC characteristics of oil palm male inflorescence in general have met FAO standards as food additives, as seen from the water content, pH, degree of substitution, and functional groups (FTIR spectra). In addition, the viscosity and purity parameters do not meet FAO standards. Therefore, further research into the optimization stage is highly recommended.

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