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Original Research Article

Synthesis and Characterization of Carboxymethyl Cellulose (CMC) from Palm Leaf Fronds (*Borassus Flabellifer)*

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ABSTRACT

Palm frond fibers are one of the wastes that are underutilized by society and have a cellulose content of 54.27% so they have the potential to be made into carboxymethyl cellulose (CMC). This research was conducted to determine the process of synthesizing CMC from palm frond fibers and the characteristics of CMC from palm frond fibers. The methods used in this research are synthesis and characterization. The CMC synthesis process consists of alkalization and carboxymethylation processes. Characteristics of palm frond fiber CMC include yield, color, pH and FITR test. The results showed that the CMC characteristics of palm leaf fiber produced a yield of 90%, pH 6.83 and a grayish white color. FTIR CMC results from palm leaf fiber show the -OH group in the absorption area of 3331.36 cm^{-1}, C-H in the absorption area of 2891.5 cm^{-1}, and the -COC- group in the absorption area of 1000-1300 cm^{-1}.

Keywords: Palm Frond Fiber, Carboxymethyl Cellulose (CMC), Cellulose, Color

INTRODUCTION

Lontar (*Borassus flabellifer*) is a plant that is very developed in Indonesia. Lontar usually grows in the eastern parts of Java, Madura, Bali, West Nusa Tenggara, East Nusa Tenggara and Sulawesi. The existence of Lontar in South Sulawesi Province is very abundant with a population of 300,000 lontar trees (Saduk & Niron, 2018). The large population of palm plants causes the large number of palm leaf fronds produced by each tree. Palm frond fiber contains 88.48% dry matter and 11.52% water. The chemical content of palm leaf fiber is 1.06% silicate, 1.12% lignin, 22.34 hemicellulose and 54.27% cellulose (Saduk & Niron, 2018). According to (Silsia et al., 2018), the cellulose content in plants has the potential to be processed into carboxymethyl cellulose (CMC).

Carboxymethyl cellulose (CMC) is a cellulose polymer that can prevent protein deposition at the isoelectric point and is biodegradable, colorless and non-toxic (Ayuningtiyas et al., 2017). CMC is usually used as a food stabilizer and emulsifier which can dissolve in water in hot and cold temperatures. According to (Ferdiansyah, 2016), the CMC formation process consists of two main processes, namely the alkalization process and carboxymethylation (esterification). These two processes greatly influence the final characteristics of CMC.

The use of different types of solutions and concentrations in the alkalization and carboxymethylation processes in CMC synthesis can apparently influence the quality of the CMC produced. According to (Silsia et al., 2018) making CMC using 30% NaOH solution in the alkalization treatment and using varying concentrations of trichloroacetic acid (10%, 20% and 30%) in the carboxymethylation treatment, resulting in different CMC pH values. The results of the research carried out showed that the highest pH value was found at a concentration of 20%. Meanwhile, the lowest pH of the three concentrations is a concentration of 30%. Based on research results (Triasswari et al., 2022), The use of trichloroacetic acid in the carboxymethylation process produces the best CMC characteristics with yield, water content, degree of substitution, viscosity and CMC purity each equal to 1,13±0,12; 22,86±0,15%, 9,57±0,53%, 170,00±10.000cPs and 99,14±0,40%. In this research, CMC synthesis and characterization aims to determine the CMC synthesis process and the characteristics of CMC from palm fronds.

MATERIALS & METHODS

Materials

Materials used in this study include aquadest (H_2O), glacial acetic acid (CH_3COOH) 90%, nitric acid (HNO_3) 3,5%, trichloroacetic acid (CCl_3COOH) 15%, hydrogen peroxide (H_2O_2) 10%, isopropanol (C_3H_8O), filter paper, methanol (CH_3OH), sodium hydroxide (NaOH) 2%, 10%, 17,5% dan 20%, sodium hypochlorite (NaOCl) 1,75% dan 6%, sodium sulfite (Na_2SO_3) 2%, alm frond fiber and waterone. FTIR (*Fourier Transform Infra Red*), desiccator, conductometer, turbidimeter, pH meter, hot plate magnetic stirrer, vacuum pump, analytical balance, shieve shaker, oven, electric stove, thermometer 110^{o}C, mortar and pestle, statives, clamps, stopwatches and glassware.

Methods

Synthesis of CMC from Palmyra Palm Fibers

A. Preparation of Palm Leaf Fiber Samples

Sample preparation was carried out by taking samples of palm leaf fronds Wajo district, Maniangpajo district, Anabanua sub-district, South Sulawesi. Next, the drying stage is carried out. After that, flour is carried out by taking the palm leaf fiber, then cutting it into small pieces and crushing it into powder using a blender. The powder obtained was sieved using a shieve shaker with a 100 mesh sieve size and dried again for 1 hour using an oven 60^{o}C (Rusdin, 2020)

B. Cellulose Extraction

Cellulose extraction was carried out by soaking 50 grams of palm frond fiber powder using 500 mL of 10% sodium hydroxide (NaOH) solution, then stirring thoroughly until all the palm frond fiber powder was submerged. Soaking is done for 24 hours. After that, a filtering process is carried out using a filter cloth. Then, the residue from the filter results is taken and continues with the bleaching process. The bleaching process is carried out by means of the residue that has been obtained, then adding 150 mL of 6% sodium hypochlorite solution and heating for 60 minutes at temperature 60˚C. Next, it is washed using warm water and filtered to remove remaining sodium hypochlorite (NaOCl). This process is carried out 3 times until white cellulose is obtained. The resulting cellulose is then dried using an oven at 60˚C until dry, then stored in a desiccator (Rusdin, 2020).

C. Synthesis of Carboxymethyl Cellulose (CMC)

5 grams of cellulose was weighed, then put into a 250 mL beaker. Add 100 mL of isopropanol and stir for 10 minutes. Then alkalization was carried out by adding 20 mL of 17.5% NaOH. The mixture was heated with a magnetic stirrer hotplate at 30^{o}C for 1 hour. After the alkalization process is complete, it continues with the carboxymethylation process by adding 20

mL of 15% trichloroacetic acid and heating at 45˚C for 3 hours. After that, the mixture is filtered and the residue continues to the neutralization process (Rusdin, 2020)

D. Neutralization of CMC

After the carboxymethyl process is complete, the magnetic stirrer hotplate is turned off, then the mixture is filtered and the residue is transferred into a beaker and measured. The pH. Next, 90% glacial acetic acid was added to pH 7. After that, the mixture was filtered and washed with waterone. The residue obtained was then soaked using 100 mL of methanol for 24 hours. The solid (CMC) obtained from the filtration results is then dried using an oven at a temperature of 60◦C until dry. The dry solid is then crushed using a mortar and pestle to obtain CMC powder (Safitri et al., 2017)

E. CMC Characteristics of Palm Palm Fiber Cellulose

The CMC characteristics of palm leaf fiber cellulose include the degree of acidity (pH), CMC weight, yield and color.

RESULT AND DISCUSSION

Cellulose Extraction

The results of palm leaf frond fiber cellulose extraction were carried out with an initial volume of 75 grams. After extraction, the volume obtained was 8.3 grams which resulted in a yield of 11.06% with a gray color. The low yield produced can be caused by the washing and filtering process. Apart from that, there is a high lignin content in palm leaf fiber. This is because the fibers from palm fronds are hard and strong, long and brown in color. This is in accordance with research (Penelitian & Hidup, 2017), that wood that is classified as containing high lignin can be seen from its darker, harder and more durable color. Meanwhile, wood that is classified as having a low lignin content has a lighter color. The color of the cellulose produced is gray. This indicates that there are still lignin compounds remaining so that the cellulose produced is not white.

Synthesis CMC

The process of making carboxymethyl cellulose (CMC) is carried out using 2 stages, namely the alkalization and carboxymethylation processes. The alkalization process in this research uses a solution of isopropanol and 17.5% NaOH with stirring for 1 hour at a temperature of 30˚C to make α-cellulose change to alkaline cellulose with the addition of isopropanol as a catalyst and the contribution of Na+ groups from 17.5% NaOH. The carboxymethylation process aims to substitute the anhydroxyl group in each anhydroglucose unit using 15% trichloroacetic acid. The addition of trichloroacetic acid causes an esterification reaction to take place, where the O- on the C-6 atom of cellulose which is a nucleophile will be attacked by the carbonyl group of trichloroacetic acid which is an electrophile and forms cellulose ether (Rusdin, 2020).

Characteristics of Palmyra Palm Fiber CMC

Yield

The resulting yield from CMC is 28%. The yield of CMC from the results of the research conducted was lower than the results of research (Safitri et al., 2017) in the synthesis of CMC from durian skin which obtained a yield of 36.573%.

Color

Carboxymethyl cellulose (CMC) from palm fronds produces a grayish white color as shown in Figure 1. This is not in accordance with previous research, Sebayar and Sembiring, 2017, that the CMC produced is white. This is because there is still a little lignin remaining and it reacts with NaOH which causes CMC to not be white. Based on (Alizadeh Asl et al., 2017) In the synthesis of CMC from sugarcane bagasse, the color produced from CMC synthesis produces a yellowish color using sodium monochloroacetate reagent in the carboxymethylation process. By increasing the NaOH concentration to 40%, the yellowish color decreases. So it can be concluded that increasing the concentration of NaOH greatly influences the color of the CMC produced.

Figure 1. CMC

Degree of Acidity (pH)

The degree of acidity (pH) is a concentration of hydrogen ions in a water solvent which is used to determine the level of acidity or alkalinity of a solution. The pH value ranges from 0-14. The pH produced in the CMC synthesis is 6.83. Based on (Silsia et al., 2018) in the CMC characterization of palm fronds, the resulting pH was 8.32 and 7.48. The pH produced from CMC meets FAO standards as proven by research results (Ferdiansyah, 2016), that the FAO standard pH ranges from 6.0-8.5. Based on the results of research conducted by (Suryadi et al., 2019) in the characterization of CMC from betung bamboo which gave a pH value of 7.61 using sodium monochloroacetate and the standard pH value for CMC was 6.93. The standard value of CMC pH is similar to the results of the research carried out.

FTIR Test

Identification of functional groups using FTIR includes palm frond fibers and CMC of palm frond fibers. The FTIR absorption area of palm frond fiber cellulose and the CMC of palm frond fiber are shown in Table 1.

Table 1. FTIR Absorption Area of Palm Leaf Fibers and CMC of Palm Palm Fibers

Absorption Area (cm^{-1})		
Palmyra Fiber	**Palm Palm Fiber CMC**	**Functional groups**
3426.18	3331.36	O-H *stretching*
2928.87	2891.54	C-H *stretching*
1735.70	-	C=O
1510.38	-	C=C
1425.04	1417.47	C-H *bending*
1378.60	1365.88	C-H
1249.49	1226.15	C-O-C
-	1157.2	C-O
897.60	894.23	C-H *bending*

Based on Table 1, palm leaf fiber powder analyzed using FTIR produced a peak at wave number 1735.70 indicating the C=O group. This indicates that the palm leaf fiber samples before being treated still contained lignin compounds. This is in accordance with research (Putera, 2012), which extracts cellulose fiber from water hyacinth plants, that the 1700 cm -1 area shows the presence of acetyl and ester groups in hemicellulose or carboxylic acid groups in the ferulic and p-cumeric groups in lignin, which are indicated by C=O groups. From the results of research that has been carried out, after the CMC treatment has been carried out, the wave number of 1700 cm^{-1} has disappeared, which indicates the loss of some of the lignin compounds from palm leaf fiber. The results of functional groups in FTIR CMC of palm frond fibers show the presence of –OH groups at a wave number of 3331.36 cm^{-1}. The C-H group is at a wave number of 2891.5 cm^{-1} and the ether group (-COC-) is at a wave number of 1000-1300 cm^{-1}. Based on research results (Pily, 2017) in the synthesis of CMC from plantain using trichloroacetic acid as a reagent, the wave number 3441 cm^{-1} shows the –OH group, which indicates that an esterification reaction occurred between trichloroacetic acid and cellulose. This is in accordance with the research results (Astuti, 2017) in the synthesis of cellulose ether from Ambon banana peel with trichloroacetic acid, the wave numbers found in CMC are wave numbers 3410.15 cm^{-1}, 1627.92 cm^{-1}, 1095.57 cm^{-1}, 1033.85 cm^{-1} and 864.11 cm^{-1} . Based on (Salimi et al., 2021) that the FTIR CMC results of the NaOH concentration used showed –OH groups, carboxyl groups (COO-), –CH2 bonds and ether groups (-COC-).

CONCLUSION

In this study, the characteristics of palm frond fiber CMC include yield, color, pH and FITR test. The results showed that the CMC characteristics of palm leaf fiber produced a yield of 90%, pH 6.83 and a grayish white color. FTIR CMC results from palm leaf fiber show the -OH group in the absorption area of 3331.36 cm^{-1}, C-H in the absorption area of 2891.5 cm^{-1}, the -COC- group in the absorption area of 1000-1300 cm^{-1}.

Declaration by Authors

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REFERENCES

1. Alizadeh Asl, S., Mousavi, M., & Labbafi, M. (2017). Synthesis and Characterization of Carboxymethyl Cellulose from Sugarcane Bagasse. *Journal of Food Processing & Technology*, *08*(08). https://doi.org/10.4172/2157-7110.1000687
2. Astuti, L. (2017). *Sintesis Eter Selulosa Melalui Reaksi Eterifikasi Selulosa Hasil Isolasi Kulit Buah Pisang Ambon (Musa Paradisiaca L) dengan Asam Trikloroasetat sebagai Adsorben Ion Logam Kadmium (Cd2+)* [Universitas Sumatera Utara]. http://repositori.usu.ac.id/handle/123456789/3290
3. Ayuningtiyas, S., Dwi, D. F., & MZ, S. (2017). Pembuatan Karboksimetil Selulosa Dari Kulit Pisang Kepok Dengan Variasi Konsentrasi Natrium Hidroksida, Natrium Monokloroasetat, Temperatur Dan Waktu Reaksi. *Jurnal Teknik Kimia USU*, *6*(3), 47–51.
4. Ferdiansyah, M. (2016). Kajian Karakteristik Karboksimetil Selulosa (Cmc) Dari Pelepah Kelapa Sawit Sebagai Upaya Diversifikasi Bahan Tambahan Pangan Yang Halal. *Jurnal Aplikasi Teknologi Pangan*, *5*(4), 136–139. https://doi.org/10.17728/jatp.198
5. Penelitian, B., & Hidup, L. (2017). *Jurnal Penelitian Kehutanan Wallacea (Basic Properties and Uses of Agathis hamii M . Dr . Wood)*. *6*, 157–167.
6. Pily, M. (2017). *Sintesis Karboksimetil Triselulosa dari Selulosa Kulit Pisang Raja (Musa X Paradisiacal AAB) Melalui Reaksi Karboksimetilasi dengan Asam Trikloroasetat sebagai Pengadsorpsi Ion Tembaga (Cu2+)* [Universitas Sumatera Utara]. http://repositori.usu.ac.id/handle/123456789/3285
7. Putera, R. (2012). *Ekstraksi Serat Selulosa Dari Tanaman Eceng Gondok (Eichornia Crassipes) Dengan Variasi Pelarut Skripsi Rizky Dirga Harya Putera 0806456801 Fakultas Teknik Program Studi Teknik Kimia Depok Juli 2012*. 45.
8. Rusdin, A. (2020). Sintesis Karboksimetil Selulosa (CMC) dari Selulosa Pelepah Lontar (Borassus flabellifer) Sebagai Flokulan. *Skripsi Fakultas Sains Dan Teknologi Universitas Islam Negeri Alauddin, Makassar*, *Cmc*.
9. Saduk, M. R. F., & Niron, F. P. (2018). Kajian Sifat Tarik Serat Pelepah Lontar dengan Singular Fiber Tensile Testing Methode. *Jurnal METTEK*, *4*(1), 8. https://doi.org/10.24843/mettek.2018.v04.i01.p02
10. Safitri, D., Abdul Rahim, E., & Sikanna, R. (2017). SINTESIS KARBOKSIMETIL SELULOSA (CMC) DARI SELULOSA KULIT DURIAN (Durio zibethinus) [Synthesis of Carboxymethyl Cellulose (CMS) of Durian Peel (Durio Zibethinus) Cellulose]. *Kovalen*, *3*(1), 58–68.
11. Salimi, Y. K., Hasan, A. S., & Botutihe, D. N. (2021). Sintesis dan Karakterisasi Carboxymethyl Cellulose Sodium (Na-CMC) dari Selulosa Eceng Gondok (Eichhornia crassipes) dengan Media Reaksi Etanol-Isobutanol. *Jambura Journal of Chemistry*, *3*(1), 1–11. https://doi.org/10.34312/jambchem.v3i1.9288

12. Silsia, D., Efendi, Z., & Timotius, F. (2018). Characterization of Carboxymethyl Cellulose (Cmc) From Palm Midrib. *Jurnal Agroindustri*, *8*(1), 53–61. https://doi.org/10.31186/j.agroind.8.1.53-61
13. Suryadi, H., Harmita, Akbar, M. H., & Lestari, P. (2019). Characterization of hydroxypropyl cellulose produced from α-cellulose betung bamboo (Dendrocalamus asper) and it's application in tablet formulation. *International Journal of Applied Pharmaceutics*, *11*(2), 123–129. https://doi.org/10.22159/ijap.2019v11i2.31078
14. Triasswari, N. P. M., Arnata, I. W., & Yoga, I. W. G. S. (2022). Karakteristik Karboksimetil Selulosa Dari Onggok Singkong Pada Variasi Konsentrasi Natrium Hidroksida Dan Asam Trikloroasetat. *Jurnal Rekayasa Dan Manajemen Agroindustri*, *10*(3), 302. https://doi.org/10.24843/jrma.2022.v10.i03.p07

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