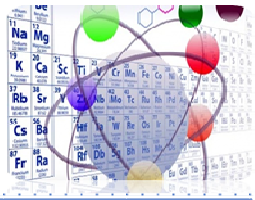
Indonesian Journal of Chemical Science and Technology (IJCST)

State University of Medan, https://jurnal.unimed.ac.id/2012/index.php/aromatika

IJCST-UNIMED 2023, Vol. 06, No. 2 Page; 136 – 142

Received : Mar 23th, 2023 Accepted : June 2nd, 2023 Web Published : July 31st, 2023



Synthesis and Characterization of Activated Carbon/Alginate/Nanocellulose-Cu Composites

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ABSTRACT

OPEFB is one source of natural fiber-based composites which have the potential to become activated carbon and nanocellulose. This study aims to synthesize and characterize the activated carbon/alginate/nanocellulose-Cu composite. The characterization used in this study is FTIR and PSA. The synthesis of activated carbon/alginate/nanocellulose-Cu composites began with a process of carbonization and activation with H_3PO_4 to produce Activated Carbon. Followed by a bleaching process with $NaClO_2$ and a delignification process with Na_2SO_3 and NaOH to produce Nanocellulose. Alginate using commercial alginate. Furthermore, the three ingredients were mixed until homogeneous and put into a 0.1M $CuSO_4$ solution to produce beads. The results of the characterization of characterization of PSA Nanocellulose obtained a particle size of 41.05 nm and the result of FTIR characterization on the activated carbon/alginate/nanocellulose-Cu composite contained the functional group OH group, triple C bond from stretching alkyne, C=C aromatic group, C-H alkane group, C-O group, the P=O stretching vibration of the P-O-C group and the alcohol OH group expressing the active carbon; there are functional groups of hydroxyl (OH), carboxyl, carbonyl, and C-O-C and –COOH bonds which represent alginate and there are OH functional groups, stretching C-H bonds, C-O stretching, stretching C-C, and β- glucosidic bonds between glucose units which indicate nanocellulose.

Keywords: *Composite, OPEFB, Actived carbon, alginate, nanocellulose, $CuSO_4$*

1. INTRODUCTION

Indonesia is a major producer of palm oil with a total of around 13.5 million hectares of oil palm plantations with oil palm plantation waste dominated by biomass, such as empty palm oil fruit bunches. Visually OPEFB has fiber that can be used for organic fertilizer, raw material for making paper, briquettes and also used as filler media such as cavity fillers. Therefore, the awareness of researchers emerged to

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examine more deeply the potential of OPEFB as a natural fiber material that can be processed into composite matrix materials.[1]

Composites are made by combining two or more microporous or macroparticles that differ in shape, chemical composition and properties with each other. The manufacture of composites requires both a fiber and a matrix, where the fibers are used as reinforcing elements to determine the mechanical properties, extend the loading of the matrix and the two materials combine to provide unique properties. Composites made from natural fibers have been developed the most compared to composites made from synthetic fibers because composites made from natural fibers have a considerable increase in physical-mechanical properties, good enough to strengthen polymers because they have relatively high stiffness and strength with lower density and better process capability.[2] One source of natural fiber-based composites that is easy to obtain is empty oil palm fruit bunches (EFB). OPEFB is included in lignocellulosic waste which consists of 40-43% cellulose, 22-25% hemicellulose and 19-21% lignin,[3] which makes OPEFB the potential to be used as activated carbon and nanocellulose. Nanocellulose is an excellent reinforcing material for composites because it has properties including high surface area, hydrophilicity, mechanical strength, biocompatibility, biodegradability, and lack of toxicity,[4] has mechanical properties with a large surface ratio, high ductility. , high tensile strength, and highly porous.[5]

The use of environmentally friendly natural polymers such as polysaccharides which are easily degraded is an alternative solution in making biocomposites as slow release fertilizers,[6] one of the natural polymers referred to is Alginate because it can form cross-links by increasing the number of cations and forming beads or granules. composite granules.[7] But the use of alginate in encapsulation has a lack of hydrophilic properties, so to increase its effectiveness it can be composited with activated carbon. Based on the description above, the use of activated carbon and alginate as a matrix or binder and the use of nanocellulose as a natural fiber material is used as a reinforcing material in composites. Therefore, this study aims to synthesize and characterize the activated carbon/alginate/nanocellulose-Cu composite.

2. EXPERIMENTAL

2.1. Chemicals, Equipment and Instrumentation

The tools used are glassware, grinder, 200 mesh sieve, analytical balance, vacuum pump, oven, furnace, hotplate, centrifuge. The materials used are empty palm oil bunches from PTPN II PKS Pagar Merbau, Metal $CuSO_4.5H_2O$, 0.7% $NaClO_2$, 100% CH_3COOH, 5% Na_2SO_3, 17.5% NaOH, 64% H_2SO_4, 10% H_3PO_4, alginate, dialysis membrane , distilled water, filter paper and pH meter. Characterization using PSA and Fourier Transform Infrared (FTIR) Spectrophotometer.

2.2. Research Procedure

2.2.1. Carbonization and Activation of Activated Carbon

Empty Palm Empty Oil Bunches (OPEFB) are washed in running water and dried in the sun then crushed and sifted. The biosorbent was carbonized in a furnace at 500°C for 2 minutes. Carbon was activated with 10% H_3PO_4 for 24 hours, then washed until neutral and dried in an oven at 105 °C.[8] Activated carbon is characterized by FTIR.

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2.2.2. Nanocellulose Isolation

Heat the fiber in a $NaClO_2$ solution with an acidic ratio of 1:50 for 2 hours at 70–80°C. This process was repeated 4-5 times until the fiber turned white, filtered and the residue was washed with distilled water and dried. Heat the cellulose in 5% (w/v) sodium sulfite solution at 70-80°C for 2 hours, filter and wash with distilled water and dry. After drying, soak in 17.5% (w/v) sodium hydroxide (NaOH) solution for 2 hours, filter and wash with distilled water then dry in the oven. Hydrolyze α-cellulose with 64% sulfuric acid at 45°C for 45 minutes and add cold water to stop the reaction. Centrifuge the suspension at 11000 rpm for 10 minutes and repeat until pH 5 then dialyzed for 3 days.[9] Nanocellulose is characterized by FTIR & PSA.

2.2.3. Synthesis of Activated carbon/Alginate/Nanocellulose-Cu Composite

Activated carbon, alginate and nanocellulose are mixed in 100 mL of distilled water with the composition according to Table 1 until homogeneous. The solution was dripped using a syringe into 0.1 M $CuSO_4$ solution, filtered and washed after 24 hours until the pH was neutral. then dry in the oven. Activated carbon/alginate/nanocellulose-Cu composite characterized by FTIR.

Table 1. Composite Variation

Composite	Composite Variation (g)		
	Activated Carbon	Alginate	Nanocellulose
Sample-1	1	1	1
Sample-2	3	1	1
Sample-3	1	3	1
Sample-4	1	1	3

3. RESULTS AND DISCUSSION

3.1. Carbonization and Activation of Activated Carbon

Carbonization in this study aims to release and remove volatile components in order to create and open pore structures.[10] Carbonized carbon results, cooled and put in a closed container. Carbon activation in this study is a chemical activation because it uses H_3PO_4 activator which can help expand pores.[11] After 24 hours, the activated carbon was filtered and washed using distilled water until the pH was neutral to remove any remaining phosphate residue.

3.2. Nanocellulose Isolation

Nanocellulose isolation begins with the bleaching process, namely heating OPEFB fibers with 0.7% $NaClO_2$ solution to produce white cellulose by completely removing lignin, hemicellulose and cellulose impurities.[12] Followed by a delignification process, namely heating with Na_2SO_3 and soaking NaOH to remove some of the lignin, hemicellulose, and other lignin monomers in OPEFB fiber. The use of a concentration of 17.5% in NaOH solution in order to increase the purity of alphacellulose based on the degree of polymerization and solubility in 17.5% NaOH.[12] The acid hydrolysis process of nanocellulosan with 64% sulfuric acid is a heterogeneous process with the acid diffusing into cellulose and cutting the

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glycosidic bonds in the cellulose polymer and will produce a high degree of crystallinity in nanocellulose[13] which aims to remove the amorphous part of the cellulose chain so that crystal isolation can be carried out. cellulose. The suspension was centrifuged at 11000 rpm for 10 minutes to obtain precipitate and repeated until pH 5 was then dialyzed for 3 days. Analysis of the distribution of nanocellulose particles using PSA can be seen in Figure 1, with a particle size of 0.04105 µm to 41.05 nm.

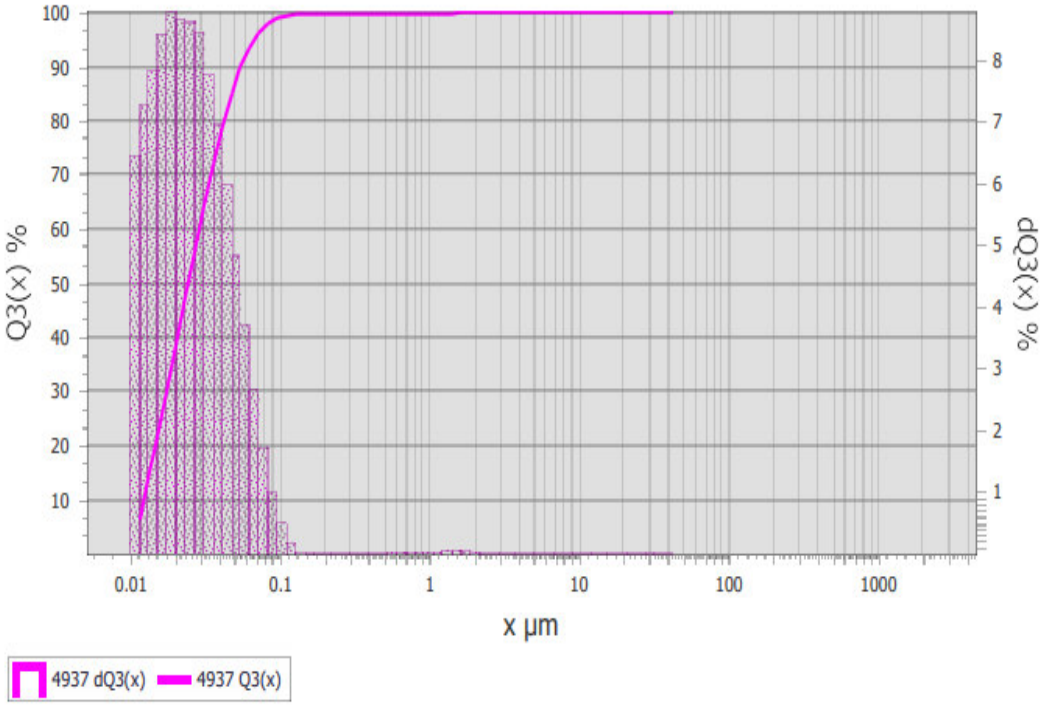

Figure 1. Nanocellulose PSA

3.3. Synthesis of Activared Carbon/Alginate/Nanocellulose-Cu Composite

The variation of the KAlgNCC111 composite will form beads that are dense and sturdy according to its composition, the variation of the KAlgNCC311 composite will form beads that are darker in color because the composition of activated carbon is more but less sturdy, the variation of the KAlgNCC131 composite will form beads that are very sturdy and dense in shape because of its composition. The larger alginate and the KAlgNCC113 composite variety will form beads that are slightly denser and tougher and have a more black to grayish color due to the more nanocellulos composition.

3.4. FTIR Characterization

FTIR characterization of Activated Carbon/Alginate/Nanocellulose-cu Composites can be seen in Figure 2.

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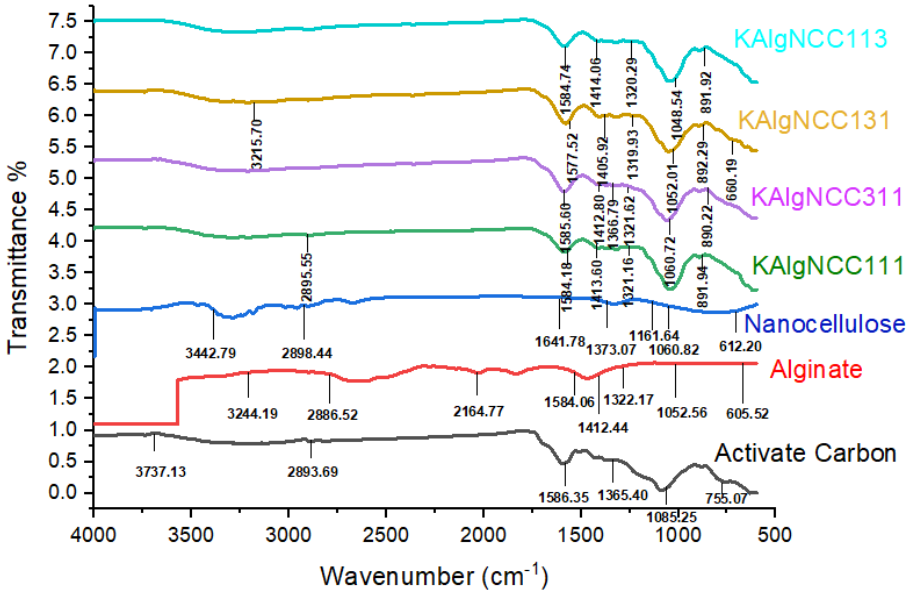

Figure 2. FTIR Characterization of Activated Carbon/Alginate/Nanocellulose-Cu Composite

Based on the graph above, FTIR Activated Carbon is shown to have absorption at wave number 3737.13cm^{-1} indicating the presence of OH groups, the peak with wave number 2893.69cm^{-1} indicates the presence of a triple C bond from alkyne stretching, at wave number 1586.35cm^{-1} indicates the presence of groups C=C aromatic, wave number 1434.08cm^{-1} indicates the presence of a C-H alkane group, wave number 1365.40cm^{-1} indicates the presence of a C-O group, wave number 1085.25cm^{-1} indicates a P=O stretching vibration of the P-O-C group and wave number 755.07cm^{-1} indicates the presence of the alcohol OH group.

The results of Wahyuningsih et al., (2016)[15] showed that the activated carbon of OPEFB has several functional groups, including hydroxyl (OH), carboxyl (–COOH), and carbonyl (–CO), as well as the aromatic structure of lignin and seluluce on the surface. Alginate FTIR was shown to have absorption in the 3244.19cm^{-1} region indicating hydroxyl groups, absorption in the 1584.06cm^{-1} region indicated carbonyl, absorption in the 1412.44cm^{-1} region indicated C-O-H bonds, absorption in the 1052.56cm^{-1} region indicated the presence of C-O-C and –COOH bonds. Mushollaeni and Rusdiana's research., (2011)[14] results of FTIR Alginate analysis showed the presence of hydroxyl (OH), carboxyl, carbonyl, and C-O-C and –COOH functional groups. FTIR Nanocellulose is indicated by the presence of absorption at wave number 3442.79cm^{-1} indicating the OH group, the peak with wave number 2898.44cm^{-1} indicates stretching of the C-H bonds, at wave number 1641.78cm^{-1} indicates the absorption of water by cellulose molecules, wave number 1161.64cm^{-1} shows C-O stretching absorption, wave number 1060.82cm^{-1} shows C-C stretching, and wave number 895.14cm^{-1} shows β-glucosidic bonds between glucose units. The FTIR KAlgNCC composite of the four variations showed peaks with functional groups such as hydroxyl, carbonyl, carboxyl, C-H, C-O stretching, C-C and others which were also present in the FTIR peaks of each row material.

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4. CONCLUSION

The synthesis of the activated carbon/alginate/nanocellulose-Cu composite comes from Empty Palm Oil Bunches which begins with a process of carbonization and activation with H_3PO_4 to produce Activated Carbon. Followed by a bleaching process with $NaClO_2$ and a delignification process with Na_2SO_3 and NaOH to produce nanocellulose. Alginate in this study used commercial alginate. After that, the activated carbon, alginate and nanocellulose were mixed until homogeneous and put into a 0.1M $CuSO_4$ solution using a syringe (injection) and left for 24 hours to produce composite granules or beads. The obtained nanocellulose has a particle size of 41.05 nm based on data from PSA characterization. FTIR characterization of the activated carbon/alginate/nanocellulose-Cu composite contained OH group functional groups, triple C bonds from alkyne stretching, C=C aromatic groups, C-H alkane groups, C-O groups, P=O stretching vibrations of P-O-C groups and OH groups alcohol declaring activated carbon; there are hydroxyl functional groups (OH), carboxyl, carbonyl, and C-O-C and –COOH bonds which represent alginate and there are OH functional groups, stretching C-H bonds, C-O stretching, stretching C-C, and β-glucosidic bonds between glucose units which represent nanocellulose.

REFERENCES

1. Rihayat, T., Salim, S., Audina, N., Khan, N S P., Zaimahwati., Sami, M., Yunus, M., Salisaj, Z., Alam P N., Saifuddin., & Yusuf, I. (**2018**). Composite material making from empty fruit bunches of palm oil (EFB) and Ijuk (*Arengapinnata*) using plastic bottle waste as adhesives. *IOP Conf. Ser: Material Science and Engineering*. 334 012083, 1-7.
2. Supu, I & Jaya, I. (**2018**). Synthesis and Compression Strength Properties of Composite Based on Sago Pulp Fiber Waste. *IOP Conf. Ser: Earth and Environmental Science*. 187 012005, 1-6.
3. Ramli, R., Junadi, N., Beg, M. D. H., & Yunus, R. M. (**2015**). *Microcystalline Cellulose (MCC) From Oil Palm Empty Fruit Bunch (EFB) Fiber via Simultaneous Ultrasonic and Alkali Treatment*. 9(1), 8-11.
4. Maghfirah, A., Fahma, F., Lisdayana, N., Yunus, M., Kusumaatmaja, A., & Marth Kadja, G T. (**2022**). On the Mechanical and Thermal Properties of Poly(Vinyl Alcohol) – Alginate Composite Yarn Reinforced with Nanocellulose from Oil Palm Empty Fruit Bunches. *Indones. J. Chem*, 22 (1), 114 - 125.
5. Trache, D., Tarchoun, A. F., Derradji, M., Hamidon, T. S., Masruchin, N., Brosse, N., & Hussin, M. H. (**2020**). "Nanocellulose: From Fundamentals to Advanced Applications". *In Frontiers in Chemistry*, 8, issue may.
6. Mayori, E., Faramitha, G N., & Sunardi. (**2018**). KARAKTERISASI BIOKOMPOSIT ALGINAT-PATI-KAOLIN SEBAGAI KANDIDAT SLOW-RELEASE PUPUK UREA. *Prosiding Seminar Nasional Lingkungan Lahan Basah*, 3(1), 191-195.
7. Bijang, C.M., Tehubijuluw, H., and Kaihatu, T.G. (**2018**). Biosorption of Cadmium (Cd2+) Metal Ion in Brown Seaweed Biosorbent (padina australis) from Liti Beach, Kisar Island. *Indo. J. Chem. Res*, 6, 51-58.
8. Zubir, M., Muchtar, Z., Syahputra, R. A., Sudarma, T.F., Nasution, H. I., Lubis, R.A.F., Fadillah, L., & Sandi, K. (**2021**). Characterization of Modified Fe-Cu Nanopartikel Actived Carbon Derived of Oil Palm Empty Bunches. *Journal of Physics*, 1-6.
9. Lani, N.S., Ngadi, N., Johari, A., & Jusoh, M. (**2014**). Isolation, Characterization, and Application of Nanocellulose from Oil Palm Empty Fruit Bunch Fiber as Nanocomposites. *Journal of Nanomaterials*, 1-9.
10. Hendrawan, Y., Sajidah, N., Umam, C., Fauzy, M. R., Wibisono, Y., & Hawa, L. C. (**2019**). Effect of Carbonization Temperature Variations and Activator Agent Types on Actived Carbon Characteristics of Sengon Wood Waste (Paraserianthes falcataria (L.) Nielsen). *IOP Conf. Ser: Earth and Envurinmental Science*. 239 012006, 1-7.
11. Mumpuni, I., Maslahat, M., & Susanty, D. (**2021**). ACTIVATION OF OIL PALM EMPTY BUNCHES WITH HYDROCHLORIDE ACID & PHOSPHORIC ACID AND CHARACTERIZATION BASED SNI NO. 06-3730-1995. *Jurnal Sains Natural*, 11, 16 – 23.

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12. W. Emmanuela Maria., Kusumawati, E., Regiana, A., & Suminar, D R. (**2020**). Production Nanocellulose from Raw Materials for Oil Palm Empty Bunches (TKKS) with Hydrolysis and *Freeze Drying* Methods. *IOP Conf. Ser: Materials Science and Engineering*. 742 012033, 1-7.
13. Tarigan, A. S., Wirjosentono, B., Zuhra C. F., & Zulnazri. (**2021**). Isolasi Nanoselulosa Dari Tandan Kosong Kelapa Sawit Menggunakan Hidrolisis Asam Sebagai Material Biomedis. *KLOROFIL*. 5(1), 1-3.
14. Mushollaeni, W., & Rusdiana, E. (**2011**). Karakterisasi Natrium Alginat dari Sargassum sp., Turbinaria sp. dan Padina sp. *J. Teknol dan Industri Pangan*. XXII(1), 26-32.
15. Wahyuningsih, P., Yusri, N., & Hamdani. (**2016**). Characterization of Activated Carbon Prepared from Oil Palm Empty Fruit Bunch by Chemical Activation using Sulphuric Acid (H_2SO_4). International Conference on Engineering and Science for Research and Development (ICESReD), 239-244.

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