Volume 16 Number 1 JUNE 2024

Journal of The Indonesian Society of Integrated Chemistry

P-ISSN 2085-1715 & E-ISSN 2621-5543

Published by Jambi University, Chemistry Education Study Program

JISIC

Preparation and Characterization of Adsorbents from Kluwak Shell (Pangium edule) Modified by Surfactant of HDTMA-Br

Zhafira Dwika Ananda[1*], Ratna Sari Dewi[1]

1 Department of Chemistry, Faculty of Mathematics and Natural Sciences, Medan State University, North Sumatra, Indonesia.

ABSTRACT

This research aims to determine the characteristics of adsorbents made from kluwak shell activated carbon modified by HDTMA-Br surfactant. Providing adsorbents is carried out using the contacting method. The FTIR characterization results show the presence of the CH_3 from N-$(CH_3)_3$ group possessed by the HDTMA-Br surfactant at wave number 1500 cm^{-1}. SEM characterization shows that the morphology of HDTMA-Br surfactant modified active carbon appears to have a surface with some of the pores closed due to electrostatic interactions compared to unmodified active carbon which has open pores. The results showed that the functional groups and morphology of activated carbon changed after contact with HDTMA-Br surfactant which was related to the absorption capacity of activated carbon.

Keyword: Adsorbent, characteristics, kluwak shell, modification, surfactant.

Received: June 2, 2024;
Revised: June 21, 2024
Accepted: June 22, 2024

* coresponding author: Zhafira Dwika Ananda
zhafiradwika02@gmail.com

DOI: https://doi.org/10.22437/jisic.v16i1.33946

INTRODUCTION

Currently, researchers are focusing on the development of activated carbon derived from biomass due to its renewable and cost-effective properties. Biomass waste such as nutshells, solid waste from grains, and fruit peels can be used to produce activated carbon (Yuliani et al., 2024). Kepayang, also known as kluwak (Pangium edule), is a wild plant that grows in Melanesia and Southeast Asia, including Indonesia. This plant spreads from lowlands to hilly areas, reaching a height of up to 25 meters, with very large, round leaves with pointed tips (Oktaviandra et al., 2020). Each kilogram of kluwak contains 74.9% shell, making the potential of kluwak shell estimated at 30,327 tons/year (Sirajuddin et al., 2020). Kluwak shell is a biomass with potential as an adsorbent, containing cellulose, hemicellulose, lignin, and fixed carbon at 92.15% content (Yuliani et al., 2024). Kluwak shell contains up to 70.52% cellulose (Sirajuddin et al., 2020).

Surfactant-modified activated carbon is activated carbon modified using surfactants to enhance its adsorption capacity. Several researchers have studied the enhancement of activated carbon's adsorption capacity for specific ions (Arnelli et al., 2021). Previous research by Aprilia et al. (2020) modified activated carbon using the cationic surfactant hexadecyltrimethylammonium bromide (HDTMA-Br) to adsorb nitrate ions.

Characterization results of HDTMA-Br surfactant-modified activated carbon with SEM analysis showed pore morphology filled with material presumed to be HDTMA-Br. The pore volume was slightly smaller than that of unmodified activated carbon because some HDTMA-Br surfactant molecules covered its surface. The SEM results corresponded with FTIR data, showing peaks presumably originating from HDTMA-Br.

In this study, activated carbon was modified using the positively charged surfactant hexadecyltrimethylammonium bromide (HDTMA-Br). This treatment is expected to make the activated carbon surface more polar.

The use of kluwak shell activated carbon has also been widely reported for use as an adsorbent for dyes and metals with varying efficiencies. However, in this study, kluwak shell activated carbon modified with HDTMA-Br surfactant is expected to have better surface and adsorption potential compared to unmodified activated carbon. This constitutes the novelty of this research. Based on the aforementioned points, research was conducted on the preparation and characterization of adsorbent from kluwak shell modified with HDTMA-Br surfactant.

METHODS

Equipment and Materials

The equipment used in this study includes a grinder, 120 mesh sieve, furnace, porcelain crucible, magnetic hot plate stirrer, thermometer, oven, vacuum, glassware, desiccator, pH meter, and instrumentation tools such as UV-Vis (Ultraviolet-Visible) spectrophotometer, FTIR (Fourier Transform Infrared), SEM (Scanning Electron Microscope), and TOC (Total Organic Carbon). The materials used in this study include KOH, distilled water, filter paper, Whatman No. 1, and the surfactant hexadecyltrimethylammonium bromide (HDTMA-Br).

Preparation and Carbonization

Sample preparation involved washing the kluwak shells and then drying them under sunlight for 2×24 hours. The specimens were then reduced in size to facilitate the carbonization process. Carbonization was performed at a temperature of 600°C for 2 hours. The obtained carbon was then ground using a grinder and sieved with a 120 mesh sieve (Adhar et al., 2022).

Carbon Activation

The activation of kluwak shell carbon was carried out using 5M KOH, as higher KOH concentrations enhance the adsorption capacity of the adsorbent (Adhar et al., 2022). A 5M KOH solution was added to the carbon in a ratio of 1:4. The mixture of carbon and 5M KOH was stirred at 200 rpm and heated at 80°C for 4 hours, then left to stand for 24 hours (Adhar et al., 2022). The sample was allowed to stand for a day to separate the precipitate and filtrate. The filtrate was discarded, and the precipitate was washed repeatedly with distilled water until the pH of the filtrate approached neutral (Oktaviandra et al., 2020). The wet activated carbon was dried in an oven at 105°C (HR et al., 2021).

Activated Carbon Modification

Activated carbon was modified using 300 ppm HDTMA-Br surfactant. For modification, 1 gram of activated carbon was contacted with 25 mL of 300 ppm HDTMA-Br surfactant for 4 hours at room temperature

(Aprilia et al., 2020). The mixture was then filtered using simple filtration with filter paper to separate the residue and filtrate. The separated residue was dried to a constant weight and cooled in a desiccator for 15 minutes.

Adsorbent Characterization

Characterization was performed using FTIR to observe the functional groups present in the carbon, activated carbon, and surfactant-modified activated carbon. Additionally, SEM was used to examine the surface morphology of the carbon, activated carbon, and surfactant-modified activated carbon.

RESULTS AND DISCUSSION

SEM Characterization

SEM (Scanning Electron Microscope) is an instrument used to analyze the surface morphology of a sample. In this study, SEM testing was conducted on both the activated carbon and the surfactant-modified activated carbon (HDTMA-Br). This aims to analyze the differences in the surface morphology characteristics of the adsorbents.

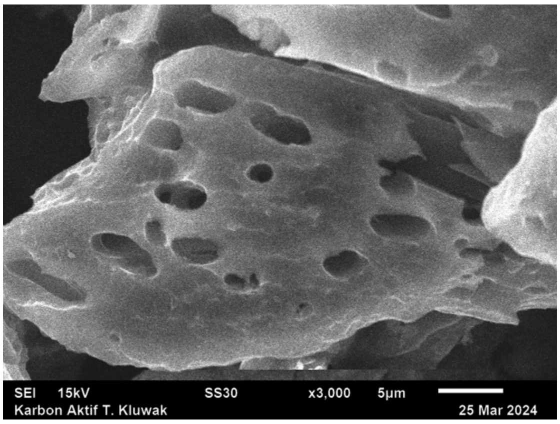

Figure 1. SEM Results of Kluwak Shell Activated Carbon

According to the research by Aprilia et al. (2020), activated carbon made from rice husks with ZnCl2 as an activator exhibited a clean pore morphology due to the activator's ability to clean the pores from impurities, such as residues from carbonization. In this study, the morphology of KOH-activated carbon, as shown in Figure 1, reveals a smooth surface with many pores on the activated carbon's surface. There are no clumps covering the pores of the activated carbon surface. The activation process with KOH causes the pores to widen due to the removal of impurities that previously covered the carbon pores. The more pores present on the carbon surface, the better the adsorption potential of the adsorbent.

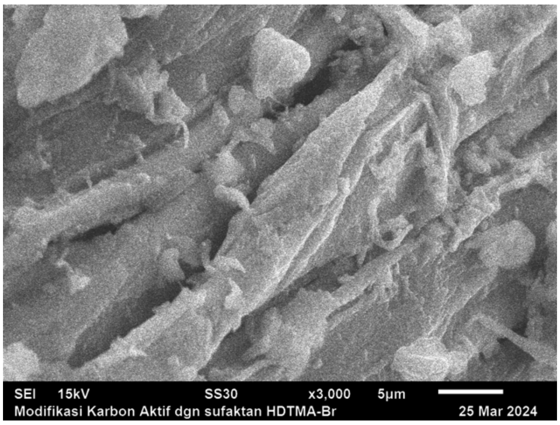

Figure 2. SEM Results of Kluwak Shell Activated Carbon Modified with HDTMA-Br Surfactant

In Figure 2, the morphological analysis of activated carbon modified with HDTMA-Br surfactant shows a slightly rough surface with some pores already covered. It is presumed that the adsorbed HDTMA-Br surfactant results in hydrophobic interactions between the surfactant and the surface of the activated carbon. This is consistent with the research by Anisyah et al. (2021), which found that modifying activated carbon with surfactant causes some of the pores to be covered because the surfactant not only adsorbs on the surface but also penetrates into the pores or between the layers of the activated carbon.

FTIR Characterization

The FTIR characterization of kluwak shell activated carbon and surfactant-modified activated carbon (HDTMA-Br) using FTIR instruments produces graphs showing the relationship between transmittance (%) and wavenumber (cm-1). Fourier Transform Infrared Spectrophotometry (FTIR) is a highly useful spectroscopic method for detecting and analyzing the molecular structure of a compound. In this process, the obtained infrared spectrum provides information about chemical bonds and molecular structures. Each functional group in a molecule has a characteristic absorption frequency, which can be used for qualitative and quantitative compound identification (Subamia et al., 2023).

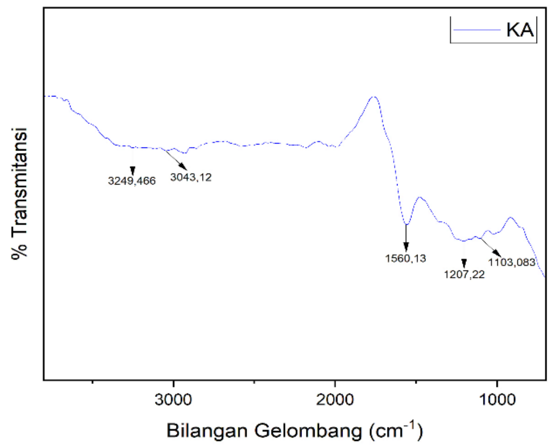

Figure 3. FTIR Results of Kluwak Shell Activated Carbon

The FTIR results of the activated carbon show a peak at 3249.466 cm-1, indicating the presence of O-H functional groups. The FTIR results of kluwak shell activated carbon also show an absorption peak at a wavenumber of 1560.13 cm-1, indicating the presence of aromatic C=C stretch groups, such as cellulose and hemicellulose (Muhajir et al., 2021). Additionally, there is a C-O-C ether vibration at a wavelength of 1207.22 cm-1. The absorption band at 1103.063 cm-1 indicates the C-O vibration of a primary C-OH. Furthermore, the activated carbon shows a new peak at 3043.12 cm-1, indicating the absorption of C-H groups. The presence of O-H and C-O functional groups suggests that the resulting activated carbon tends to be more polar. Therefore, the produced activated carbon can be used as an adsorbent for polar substances.

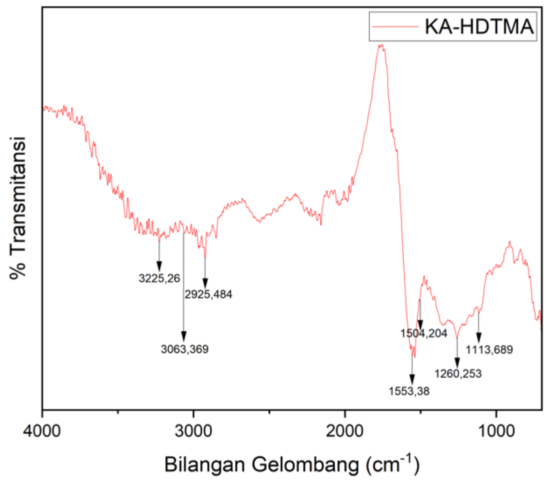

Figure 4. FTIR Results of Kluwak Shell Activated Carbon Modified with HDTMA-Br Surfactant.

According to previous research by Aprilia et al. (2020), rice husk activated carbon with added HDTMA-Br surfactant showed a peak at 1500 cm-1, which corresponds to the C-N group from N-(CH3)3 originating from the HDTMA-Br surfactant. In this study, the FTIR results of HDTMA-Br surfactant-modified activated carbon in Figure 4 show a new peak at a wavelength of 1504.204 cm-1, which represents the CH3 group from N-(CH3)3 on the HDTMA-Br surfactant. The FTIR results for other groups on HDTMA-Br surfactant-modified activated carbon do not differ significantly from carbon or activated carbon. There are peaks at wavelengths of 3225.26 cm-1 and 3063.369 cm-1, which correspond to the O-H and C-H groups, respectively. The surfactant-modified activated carbon also shows a peak at 1553.38 cm-1, indicating the presence of aromatic C=C stretch groups such as cellulose and hemicellulose (Muhajir et al., 2021). At a wavelength of 1260.253 cm-1, there is a C-O-C ether vibration, and the C-O group appears at a wavenumber of 1113.689 cm-1.

According to previous research by Aprilia et al. (2020), HDTMA-Br-modified activated carbon shows that, in SEM characterization, the surface of the modified activated carbon has partially closed pores, potentially covered by HDTMA-Br. FTIR characterization shows the appearance of a new group, the C-N group from N-(CH3)3, originating from the HDTMA-Br surfactant. HDTMA-Br-modified activated carbon has potential as an adsorbent, particularly in nitrate ion adsorption. The modified activated carbon exhibits better absorption capacity compared to unmodified activated carbon. This demonstrates that the characterization results of kluwak shell activated carbon and HDTMA-Br surfactant-modified activated carbon have potential as adsorbents in the adsorption process of metals and dyes in wastewater.

CONCLUSION

The SEM results show that the surface morphology of the carbon has a rough, clumpy texture. The morphology of activated carbon has a smooth surface with more pores compared to non-activated carbon. Additionally, the morphology of HDTMA-Br surfactant-modified activated carbon shows that the pore surface is covered, potentially by the HDTMA-Br surfactant. The FTIR characterization of HDTMA-Br modified activated carbon shows a new peak, indicating the presence of the CH3 group from N-(CH3)3 in the HDTMA-Br surfactant, which appears at around 1500 cm-1. This indicates that the HDTMA-Br surfactant was successfully adsorbed onto the surface of the activated carbon. Based on the conclusions of this study, further research can be conducted on modified activated carbon with potential applications as an adsorbent for various types of pollutants, such as metals and dyes in wastewater.

ACKNOWLEDGEMENTS

We would like to thank the Chemistry Laboratory at FMIPA, State University of Medan, for providing the research equipment, which made this study possible.

REFERENCES

Adhar, Zainal, Iidris, Setiawati, & HR. (2022). Karakteristik Karbon Aktif Tempurung Kluwak (Pangium edule) Sebagai Adsorben Pada Penjerapan Methylene Blue. *Jurnal Pengendalian Pencemaran Lingkungan*, *4*(1).

Anisyah, Arnelli, & Astuti, Y. (2021). Pembuatan Karbon Aktif Termodifikasi Surfaktan Sodium Lauryl Sulphate (SMAC-SLS) dari Tempurung Kelapa Menggunakan Aktivator ZnCl2 dan Gelombang Mikro sebagai Adsorben Kation Pb(II). *Journal of Environmental Chemistry*, *1*(1), 1–6.

Aprilia, Arnelli, & Astuti. (2020). Modification of Activated Carbon from Rice Husk using Hexadecyltrimethylammonium Bromide (HDTMA-Br) Surfactant and ZnCl2 activator and Microwaves for Nitrate Ion Adsorption. *Jurnal Kimia Sains dan Aplikasi*, *23*(11).

Aprilia, Y., Arnelli, & Astuti Yayuk. (2020). Modification of Activated Carbon from Rice Husk using Hexadecyltrimethylammonium Bromide (HDTMA-Br) Surfactant and ZnCl 2 activator and Microwaves for Nitrate Ion Adsorption H. *Jurnal Kimia Sains dan Aplikasi*, *23*(11), 377–382. Diambil dari http://ejournal.undip.ac.id/index.php/ksa

Arnelli, Santoso, B., & Astuti, Y. (2021). Modification of activated carbon of rice husk using HDTMA-Br (SMAC) surfactant as nitrite ion (NO2-) adsorbent. *Journal of Physics: Conference Series*, *1943*(1). IOP Publishing Ltd.

HR, Alwina, Zainal, Idris, Setiadi, & Adhar. (2021). Adsorpsi Metilen Biru Menggunakan Karbon Tempurung Kluwak Teraktivasi KOH. *Jurnal Kimia dan Rekayasa*, *2*(1), 1–7.

Muhajir, A., Machdar, I., & Mariana. (2021). Produksi karbon aktif arang tempurung kelapa menggunakan kombinasi metode aktivasi secara kimia dan steam tekanan rendah. *Jurnal Litbang Industri*, *11*(2), 110116–116.

Oktaviandra, R. R. R., Nurlaeli, P. D., & Billah, M. (2020). Pemanfaatan Tempurung Kluwak Sebagai Adsorben Dalam Menurunkan Kadar Logam Berat Tembaga. *Journal of Chemical and Process Engineering ChemPro Journal*, *01*(01), 41–45. Diambil dari www.chempro.upnjatim.ac.id

Sirajuddin, Harjanto, & Agustin, A. D. (2020). Pengaruh Temperatur Karbonisasi Terhadap Karakteristik Arang Aktif Dari Tempurung Kluwak (Pangium edule). *Prosiding 4th Seminar Nasional Penelitian & Pengabdian Kepada Masyarakat 2020* (hlm. 60–64).

Subamia I. D. P, Widiasih, N. N., Sri W. I. G. A. N, & Pratami, K. P. . L. (2023). Optimasi Kinerja Alat Fourier Transform Infrared (FTIR) Melaui Studi Perbandingan Komposisi dan Ketebalan Sampel-KBr. *Jurnal Pengelolaan Laboratorium Pendidikan*, *5*(2), 58–69.

Yuliani, H., Dian, R., Miftahul, J., Fadhila, Z. N., & Budiman, A. (2024). Preparation and Characteristics of Kluwak Shell Carbon Adsorbent. *Jurnal Ilmiah Teknik Sipil dan Teknik Kimia*, *9*(1), 1–10.