

FUNCTIONALIZATION MAGNETITE ACTIVATED CARBON COATING WITH SILANE AGENT AS ADSORBENT FOR CRYSTAL VIOLET AND METHYL ORANGE DYES

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ABSTRAK

Penelitian ini bertujuan untuk mengoptimalkan pemanfaatan limbah tersebut dengan mengubahnya menjadi karbon aktif yang memiliki potensi tinggi sebagai adsorben. Proses pembuatan karbon aktif melibatkan karbonisasi dan aktivasi cangkang kelapa sawit, diikuti dengan modifikasi permukaan menggunakan partikel magnetik Fe_3O_4 dan senyawa organosilan triethoxyphenylsilane (TEPS). Penambahan partikel Fe_3O_4 dapat meningkatkan kemampuan adsorpsi serta memudahkan pemisahan adsorben dari larutan setelah proses adsorpsi. Sementara, penambahan senyawa TEPS meningkatkan jumlah gugus fungsional pada permukaan karbon aktif sehingga dapat berinteraksi lebih kuat dengan molekul zat warna. Karakterisasi menggunakan berbagai teknik seperti UV-Vis, FTIR, XRD, SEM-EDX, dan BET dilakukan untuk menganalisis perubahan struktur dan sifat permukaan karbon aktif setelah modifikasi. Hasil penelitian dapat memberikan kontribusi dalam pengembangan teknologi pengolahan limbah dan produksi material adsorben bernilai tambah.

Kata kunci: Adsorpsi, karbon aktif, partikel Fe_3O_4 , agen silan, zat pewarna

ABSTRACT

This study aims to optimize utilization of this waste by converting it into activated carbon which has high potential adsorbent. Process making activated carbon involves carbonization and activation of palm kernel shells, followed by surface modification using magnetic particles and organosilane triethoxyphenylsilane (TEPS). Addition of Fe_3O_4 particles can increase adsorption capacity and facilitate the separation of the adsorbent from the solution after adsorption process. Meanwhile, addition of TEPS compounds increases the number of functional groups on the surface of activated carbon so can interact more strongly with dye molecules. Characterization using various techniques such as UV-Vis, FTIR, XRD, SEM-EDX, and BET was carried out to analyze changes structure and surface properties of activated carbon after modification. The results of the study can contribute to development of waste processing technology and production of value-added adsorbent materials.

Keywords: Adsorption, activated carbon, Fe_3O_4 particles, silane agent, dye

INTRODUCTION

Indonesia is one of the countries with the largest palm oil plantations in the world. Most of the plantation results produce primary processed products in the form of Crude Palm Oil (CPO) and its derivatives (Zulaicha et al., 2022). Palm shells are one of the wastes that contribute 60% of palm kernel oil production (BPS, 2020), the amount of palm kernel shells (PKS) produced from palm oil processing is very large, as can be seen in Figure 1. There has been a significant increase in the amount of palm shell waste reaching 10.4 million tons (Deng and Brillas, 2023). PKS are produced as much as 10 million per year, where PKS are one of the materials whose added value can be increased (Zulaicha et al., 2021).

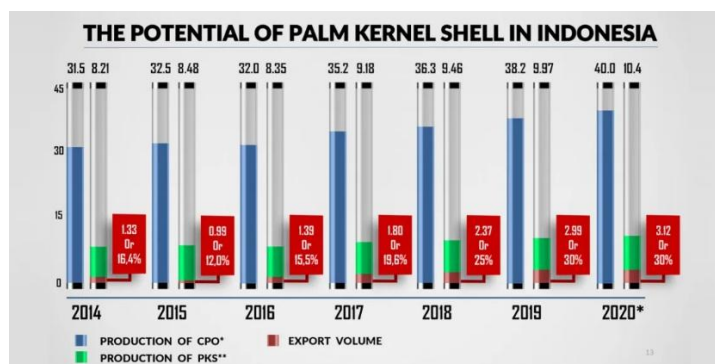


Figure 1. The potential of palm kernel shell in Indonesia per year in million tons

Activated carbon is often called the "material of the future" (Zulaicha, et al., 2021). It has excellent capabilities in a number of applications for various industries, both home industries and manufacturing industries (Gong, et al., 2016). In relation to the large use of carbon, the market demand for carbon is increasing (Dao & Le, 2020; Marrakchi, et al., 2020; Marsin et al., 2020; Buhani, et al., 2018). The use of carbon as an adsorbent is carried out by considering the policy of implementing the zero waste concept, namely utilizing agro-industrial waste to reduce other industrial waste such as toxic chemicals (Ozbay and Yargic, 2015; Saygili et al., 2015). However, along with the increase in the amount and type of waste found in the environment, it is also necessary to improve the quality of carbon (Ighalo, et al., 2022). Activated carbon has advantages as an adsorbent in many aspects, but has limitations in the separation of remaining carbon by-products and also limitations in regeneration for repeated use (Alsulaili, et al., 2023).

The coating technique with Fe_3O_4 will produce carbon that is stable in acidic conditions, separating the adsorbate quickly because the adsorbent is magnetic and the adsorption process

occurs at room temperature (Zulaicha, et al., 2022; Zulaicha et al., 2021; Buhani et al., 2017). Fe_3O_4 coating on activated carbon enables easy magnetic separation for efficient adsorbent regeneration. However, this process can reduce the number of accessible pores, limiting adsorption capacity (Buhani, et al., 2019; Wondracek et al., 2016; Negash et al., 2023). To improve the quality of carbon as an adsorbent of dyes, carbon modification will be carried out by adding functional groups as active adsorption sites by implementing a silanization process using organosilane compounds such as triethoxyphenylsilane (TEPS). So, coating of Fe_3O_4 particles with dual-function silane agents. Silane acts as a strong binding agent between Fe_3O_4 particles and activated carbon surface, improving the stability of the composite. In addition, the functional groups on silane can provide additional active sites for interaction with dye molecules, thereby increasing the adsorption capacity.

METHOD

Carbon Activation Process

A total of 100 grams of carbon with a size of 100-200 μm from palm shells is kiln-fired for one hour at a temperature of 700°C . Then 25 grams of activated carbon from physical activation is soaked in 50 mL of 10% H_3PO_4 solution for 24 hours. Next, the activated carbon is filtered and washed with distilled water until pH 6 and dried in an oven at 100°C for 1 hour to produce physical and chemical activated carbon (KAFK).

Making Magnetite Activated Carbon

Making of Magnetite Physics and Chemistry Activated Carbon (KAFKM)

Variations in making KAFKM 25% of the magnetic composition. These variations are produced by differentiating the composition of FeCl_3 and $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$. The FeCl_3 and FeSO_4 solutions are then mixed and stirred at a temperature of 60°C for 30 minutes, then 50 mL of distilled water that has been heated to a temperature of 60°C is added with 5 grams of KAFK, then mixed with a mixture of FeCl_3 and FeSO_4 solutions and stirred with a magnetic stirrer without heating for 30 minutes. After that, 10 M NH_4OH is added slowly to pH 10 and left for 24 hours. The results are filtered and rinsed until the pH is neutral, then dried in an oven at a temperature of 50°C for 5 hours (Javadian et al., 2018; Mohan et al., 2011).

Preparation of Magnetite Chemical Activated Carbon with Silane Agent (KAKM-Silan)

Activated carbon that has been modified with magnetite particles is mixed with TEPS and ethanol in a reflux flask. The mixture is heated at 80°C for 24 hours with magnetic stirring. The silanized adsorbent is washed with ethanol and dried in an oven at 100°C for 24 hours (Wong et al., 2016).

Adsorbent Characterization

Activated carbon produced, both activated carbon from physical activation and physical-chemical activation, as well as activated carbon that has been coated with magnetite and magnetite are characterized by XRD to identify the crystalline phase, SEM-EDX to determine the morphology of the adsorbent surface and the elemental composition of a sample and IR to determine the presence of functional groups on the surface of activated carbon.

RESULTS AND DISCUSSION

The adsorbent preparation process is presented in Figure 2. The charcoal is then ground into powder and heated at high temperatures (700°C) without any additional chemicals. This heating process is called physical activation. The selection of a temperature of 700°C is based on the results of previous studies showing that this temperature is the most effective for producing the best quality activated carbon (Gong et al., 2016; Marsin et al., 2020).



Figure 2. a) cleaned and dried palm shells and b) results of burning palm shells (carbon)

In order for the charcoal pores to become more and larger, the charcoal from physical activation is then soaked in a phosphoric acid solution (H_3PO_4). This acid will dissolve the remaining dirt and react with the charcoal to form new holes (pores). This process is called chemical activation (Zulaicha et al., 2021)

Adsorbent Characterization

Characterization using Scanning Electron Microscope (SEM) coupled with Energy Dispersive X-Ray (EDX)

SEM analysis is used to determine the surface morphology of the adsorbent. Then, SEM is also combined with EDX to see the composition of the elements contained in the adsorbent. The results of the analysis can be seen in Figure 4. The addition of these pores is caused by the activation process which dissolves impurities and forms new pores. KAFKM has a rough surface due to the magnetite coating that partially closes the pores. EDX analysis shows that activated carbon consists mainly of carbon (C) and oxygen (O). The magnetite coating on KAFKM adds iron (Fe) to its composition (Buhani et al., 2023).

Characterization Using X-Ray Diffractions

The emergence of new peaks at 2θ values of 39.09° and 60.746° indicates the formation of a new crystalline phase from Figure 3. These peaks are identical to those found in the magnetite diffractogram, suggesting that the magnetite coating on KAFK has been successfully achieved. The peaks coated with magnetite are observed with sharp and broad intensities as seen in the diffractogram of KAFKM-Silan (Jeswani et al., 2015; Wondracek et al., 2016; Zulaicha et al., 2022).

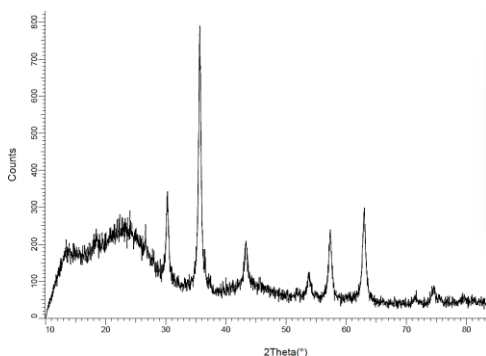
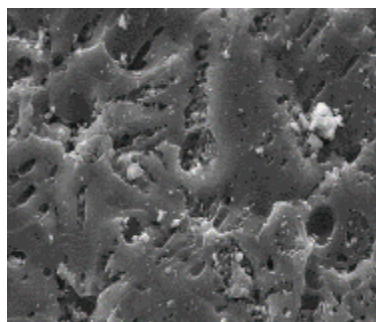


Figure 3. Diffractogram of KAFKM-Silan

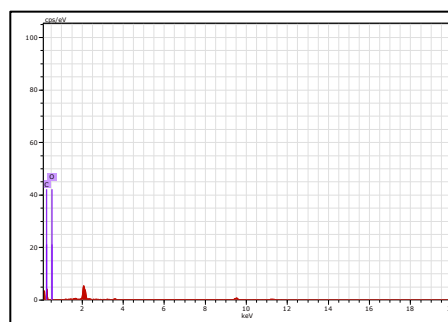
Characterization With SEM-EDX

Characterization of the synthesis of activated carbon before and after activation with variations of KAF, KAFK, and KAFKM has been conducted, as presented in Figure 4. The SEM-EDX characterization results show that images a-e represent carbon before activation, where no distinct image or spectrum was detected. In images b-f (KAF) and c-g (KAFK), differences in the pore structure are visible; KAF has larger and rougher pores, while KAFK has smaller and

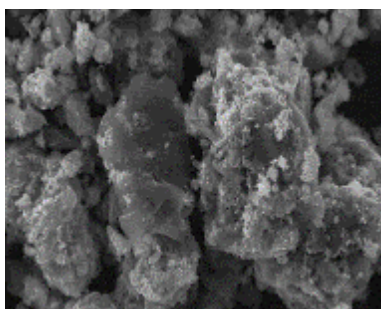
smoother pores. Additionally, the spectrum peaks indicate that the content of C and O elements in KAFK is higher than in KAF, due to the presence of chemical functional groups from carbon activation. In images d-h, which represent magnetite-activated carbon (KAFKM) successfully synthesized, the spectrum peaks for Fe and O from Fe_3O_4 are evident, confirming the presence of magnetite particles adhered to the surface of the activated carbon (Zulaicha et al., 2021).



a)



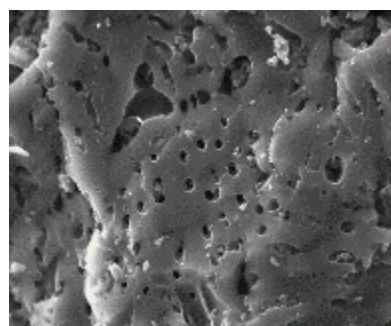
e)



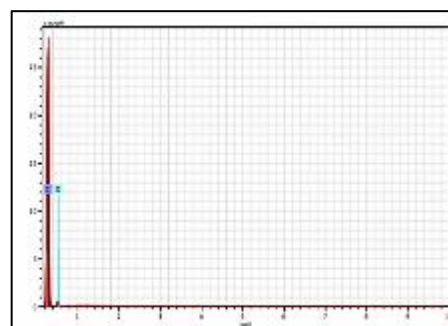
b)



f)



c)



g)

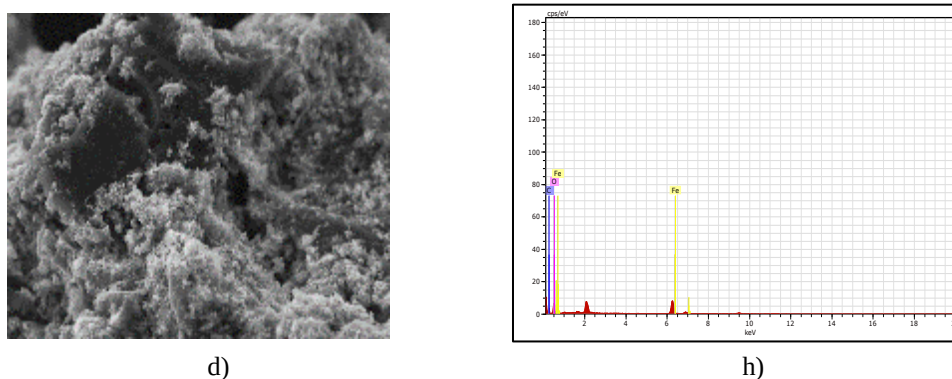


Figure 4. Characterizations Result of SEM EDX (a, e) Carbon Before Activated; (b, f) KAF; (c, g) KAFK; dan (d, h) KAFKM.

The magnetite layer reduces the surface area of activated carbon, necessitating the application of silanization with organosilane compounds such as triethoxyphenylsilane (TEPS). This study has been synthesized, but the characterization results will be presented. Activated carbon modified with magnetite particles is mixed with TEPS and ethanol in a reflux flask. Heating and washing are performed until the final products, Magnetite-Activated Carbon with Silane Agent (KAFM-Silan and KAFKM-Silan), are obtained (Lee et al., 2020; Naushad et al., 2020; Szadkowski et al., 2020).

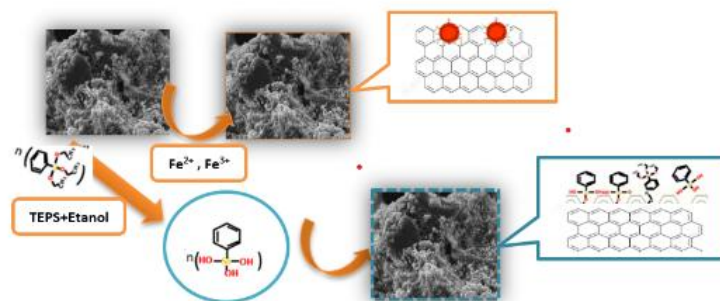


Figure 5. Synthesis Mechanism of KAFKM-Silan

The synthesis mechanism of KAFKM-Silan is shown in Figure 5. Then, silanization is performed by adding the silane reagent TEPS (triethoxyphenylsilane) in ethanol solvent, forming an active compound (Naushad et al., 2020). TEPS reacts with the magnetite surface of the activated carbon, forming a layer of silanol functional groups (Si-OH), which increases the number of active sites available for adsorption, thereby enhancing the capacity and performance of the adsorbent (Buhani et al., 2019; Sahoo et al., 2020).

Characterization Using Fourier Transform Infra Red (FT-IR)

The FT-IR spectrum provides information regarding the types and quantities of chemical bonds present in the sample (Lee et al., 2020).. Each peak in the spectrum corresponds to a specific functional group. The FT-IR analysis results for the samples KAFM, KAFKM, and KAFKM-Silan are presented in Table 1.

Table 1. Characterization Results Using Fourier Transform Infrared (FT-IR)

	Wavenumber (cm ⁻¹)						
	O - H	C - H	C - C	C=O	C - O	Fe - O	Si-O
KAFM	3465,18	-	-	1616,38	1023,25	566,15	-
KAFKM	3444,92	-	-	1615,41	1050,26	561,30	-
KAFKM_Silan	3426,88	-	-	1645,20	1044,23	550,90	475,89

The analysis results also indicate that the magnetite, as evidenced by the characteristic Fe-O absorption peaks in the KAFM and KAFKM samples in the wavenumber range of 400-700 cm⁻¹. The KAFM sample shows functional group Fe-O absorption peaks at 565.15 and 667.38 cm⁻¹, while the KAFKM sample shows peaks at wavenumbers 561.30 and 635.56 cm⁻¹ and Si-O peaks standard at 470,6 (Wondracek et al., 2016; Prarat et al., 2019).

Adsorbtion Study

Adsorbent Dosage

Figure 5(a) presents data on the variation of adsorbent dosage and its effect on the adsorption efficiency for methyl orange (MO) and crystal violet (CV) dyes. At a dosage of 0.5 g, the adsorption efficiency reaches almost 99% in all combinations (Marrakchi et al., 2020). Negatively charged adsorbents tend to be more effective in adsorbing cationic dyes such as CV (Ozbay & Yargic, 2015; Alsulaili et al., 2023).

Influence of pH

Figure 7(b) illustrates the relationship between solution pH and adsorption percentage (%A) for (MO) and (CV) dyes using KAKM and KAFKMS (KAFK magnetite silane) adsorbents. The adsorption percentage increased with increasing pH, reaching a peak at pH 10. (Zulaicha et al., 2020).

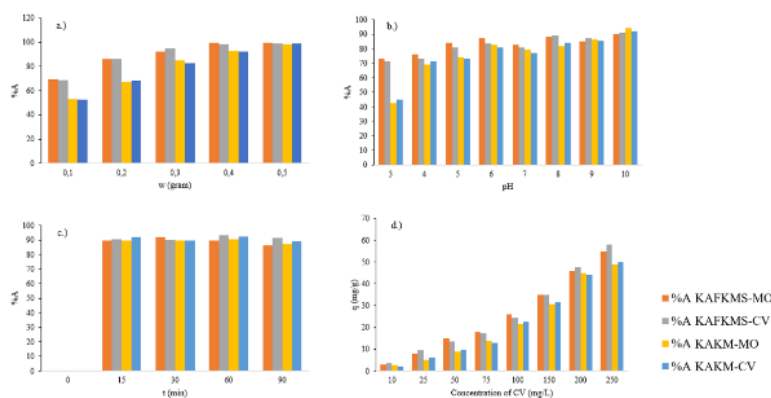


Figure 7. Effect of Variations a.) Adsorbent Dosage, b.) pH Solution, c.) Contact Time, d.) Initial Concentration

Influence of Contact Time

Figure 7(c) shows the adsorption efficiency at 15 minutes, the adsorbents KAFKMS (KAFK magnetite silane) and KAKM showed significant adsorption, with KAFKMS-MO reaching 89.75% and KAKMS-CV reaching 91.75%. The adsorption process usually occurs rapidly at the initial stage because of the many active sites on the adsorbent surface available to bind adsorbate molecules (Setiajaya et al., 2023; Zaini et al., 2023).

Influence of Initial Concentration

Figure 7(d) shows the increase in adsorption efficiency along with increasing CV (crystal violet) concentration on various adsorbents. At a low concentration, namely 10 mg/L, the adsorption efficiency is quite low, but with increasing concentration reaching 250 mg/L, the adsorption ability also increases. KAFKMS-MO and KAFKMS-CV adsorbents showed higher adsorption values compared to KAKM-MO and KAKM-CV at each concentration surface (Wong et al., 2016). Modification of magnetite silane on the adsorbent increases the adsorption efficiency by adding active sites on the surface (Szadkowski et al., 2020).

CONCLUSION

This study has successfully synthesized activated carbon adsorbent from palm shells modified with magnetite particles and silane agents. The characterization results showed that the resulting adsorbent has a large surface area and good pore distribution. This adsorbent effectively adsorbs crystal violet and methyl orange dyes with high adsorption capacity. Modification with magnetite particles facilitates the separation process of the adsorbent from the solution, while the addition of

silane agents increases the selectivity of the adsorbent. The adsorption capacity with KAFKMS-MO reached 89.75% and KAKMS-CV reached 91.75%.

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