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Synthesis and characterization of Fe3O4-Activated Carbon and it's application to adsorb methylene blue

Synthesis and characterization of Fe_3O_4-Activated Carbon and it's application to adsorb methylene blue

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Abstract. Synthesis and characterization of Fe_3O_4-Activated Carbon (Fe_3O_4-AC) and the application to adsorb methylene blue has been conducted. The Fe_3O_4-AC was synthesized using the co-precipitation method on alkaline conditions. The Fe_3O_4 was prepared from mixing mole ratio of Fe^{3+} and Fe^{2+} = 3:2, and AC was made from an egg rack. Composite of Fe_3O_4-AC was characterized using Fourier Transform Infra-Red (FTIR), Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), and Vibrating Sample Magnetometer (VSM). FTIR characterization was indicated the appearance peak of a C-O vibration at 1033.85 cm^{-1} and a functional group of Fe-O at 540.07 cm^{-1}. XRD pattern of Fe_3O_4-AC was the presence peak of crystal structure Fe_3O_4, even though it's small intensity. SEM image of Fe_3O_4-AC was spherical with the denser pore of structure. The saturation magnetization of Fe_3O_4-AC was lower than Fe_3O_4. The result showed that adsorption of methylene blue on Fe_3O_4-AC and AC was optimum at pH 7. The sorption of methylene blue on Fe_3O_4-AC and AC was 126.043 and 102.82 mg/g, respectively. The performance of Fe_3O_4-AC was greater than that of AC.

1. Introduction

Egg rack is one of the waste paper products that can be found in traditional markets. This waste is no longer used, will be burned or stored in a pile of garbage [1]. Economically, if used as a source of activated carbon (AC), egg racks will provide an advantage because the raw material is easy to find, cheap, and found everywhere [2].

Methylene blue is one of the aquatic waste environments that are generally found in textile industries. Several methods are used to reduce methylene blue in aquatic, including ion chromatography [3], liquid-liquid extraction [4], and adsorption [2,5,6]. AC can remove methylene blue adsorption because it has a high surface area with a pore microstructure [2]. The surface interaction of AC on methylene blue predominantly occurs through a cation-exchange process and electrostatically. The modification on the surface of AC can be sufficient to increase the adsorption capacity of AC [7]. The magnetic technology application is carried out by combining Fe_3O_4 on AC to obtain new composite materials of Fe_3O_4-AC. One of the advantages of the composite can be more easily accessed using an external magnetic field [6].

Based on the above background, this research was conducted for the synthesis of Fe_3O_4-AC using AC from egg rack and was characterized by FTIR, XRD, SEM, and VSM. This application was completed to compare to adsorb methylene blue on Fe_3O_4-AC and AC.

2. Methodology

2.1. Instrumentation and materials

The equipment used was a magnetic stirrer, pH meter, FTIR-8201 PC, SEM JEOL SSM-6510 LA, XRD Shimadzu XRD-6000, UV-Vis Spectrophotometer Evolution 201 type, VSM Oxford Type 1.2H. The materials used were Egg Rack, HCl, $FeCl_3.6H_2O$, $FeSO_4.7H_2O$, NH_4OH 25 %, methylene blue.

2.2. Preparation of AC and synthesis of Fe_3O_4-AC

The egg rack sample was heated in a furnace at 250ºC for 25 minutes. The carbon solids are dried in an oven at 100ºC to constant weight, then crushed until smooth and sieved using a 140 mesh. The solids were immersed in 2 M HCl for 24 hours to form activated carbon (AC). The residue is washed to a neutral pH, dried in an oven at 100ºC to constant weight, and cooled. Ten g of AC material was added to 200 mL of aqua dest and was stirred using a stirrer while heated at a temperature of 70ºC. Add a mixture of $FeCl_3.6H_2O$ and $FeSO_4.7H_2O$ solution and 100 mL NH_4OH 25% dropwise and kept at 70ºC for 3 hours. Separated precipitate and filtrate. The residue is washed to a neutral pH, dried in an oven at 100ºC to constant weight.

2.3. Characterization of material

Materials Fe_3O_4, AC, and Fe_3O_4-AC that have been made were characterized by XRD, FTIR, SEM, and VSM.

2.4. Adsorption of methylene blue on AC and Fe_3O_4-AC

A total of 10 mg of samples AC and Fe_3O_4-AC were added to 25 mL of methylene blue with a 60 mg/L concentration with various pH adjustments 3, 4, 5, 6, 7, 8 9. The solution was stirred with a shaker for 90 minutes. Filtered and the filtrate analyzed using a UV-Vis spectrophotometer.

3. Results and discussion

Synthesis of Fe_3O_4-AC uses the co-precipitation method by dissolving $FeCl_3.6H_2O$ and $FeSO_4.7H_2O$ with a mole ratio of 3: 2 [8], then with the addition 10.0161 g of AC and NH_4OH solution. It was obtained that the number of Fe_3O_4-AC is 10.6123 g. The synthesis preparation of Fe_3O_4 is carried out in open air; Fe^{2+} will be oxidized to Fe^{3+} so that there is difficulty in maintaining the Fe^{3+}: Fe^{2+}= 2:1 ratio of mole. The way to overcome, still prepare open-air, but by reducing the Fe^{3+}: Fe^{2+}= <2:1 mole ratio, so that after the oxidation of Fe^{2+} to Fe^{3+}, the mole ratio will approach 2:1 [9].
The following are AC, Fe_3O_4, and Fe_3O_4-AC, which can be seen in figure 1.

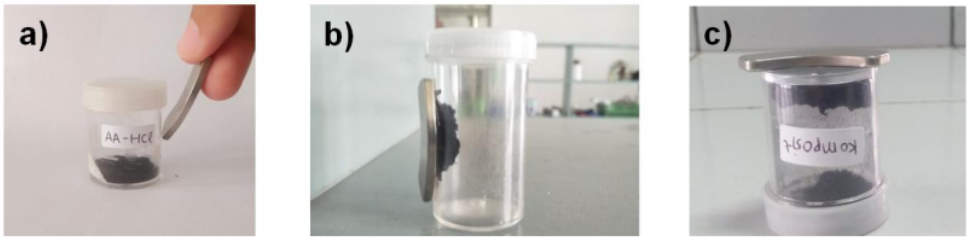

Figure 1. a) AC, b) Fe_3O_4 and c) Fe_3O_4-AC .

The material of AC is no attraction to the external magnetic, Fe_3O_4 is a strong attraction, and then Fe_3O_4-AC is a weak pull. There is a non-magnetic fraction in the composite, namely activated carbon, to reduce its magnetic power.

Characterization of FT-IR for the identification of functional groups of material. The FTIR spectra of Fe_3O_4, AC, and Fe_3O_4-AC, which can be seen in figure 2.

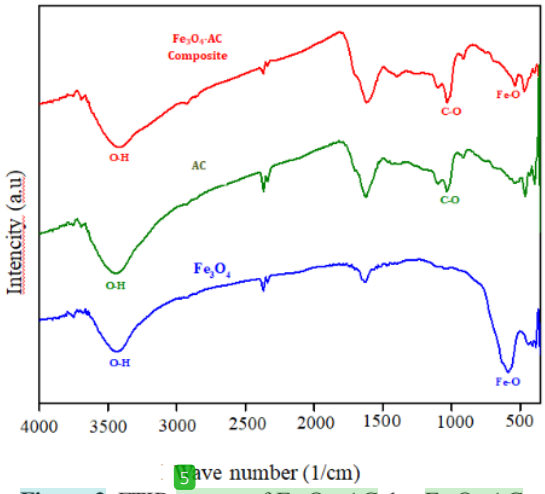

Figure 2. FTIR spectra of Fe_3O_4, AC dan Fe_3O_4-AC.

FTIR spectra of Fe3O4, the complete absorption appears at 3441.01 cm^{-1}, is stretching vibration of O-H bond, and wavenumber of 586.36 cm^{-1} is the vibration of Fe-O bond. FTIR spectra of AC, a broad absorption at a wavenumber of 3448.72 cm^{-1}, is stretching vibration of O-H bond, and at 1103.28 cm^{-1} is stretching vibration of the C-O bond. The FTIR spectra of Fe_3O_4-AC, the absorption at 3425.58 cm^{-1}, indicates an O-H bond, the C-O bond's vibration at 1103.28 cm^{-1}, and stretching vibration Fe-O bond at 540.07 cm^{-1}. It suggests that the Fe_3O_4 material has been successfully composited on the surface of AC. The following are the functional groups for the Fe_3O_4, AC, and Fe_3O_4-AC shown in table 1.

Table 1. Functional groups of Fe_3O_4, AC, and Fe_3O_4-AC.

Functional Groups	Wave Number (cm^{-1})			
	Literature [10]	Fe_3O_4	AC	Fe_3O_4-AC
O-H Stretching	3300-3600	3441.01	3448.72	3425.58
C-H Stretching	2850-2970	2931.80-2862.36	2931.8	2924.09-2862.36
C-H Bending	1340-1470	-	-	1396.46
C=O	1540-1800	1635.64	1620.21	1620.21
O-H Bending	1500-1600	1527.62	-	-
C-O	1050-1330	1273.02	1103.28	1103.28
Fe-O	500-610	586.36	-	540.07

The Diffractogram of XRD shows to identify the crystalline phase of the material. The characterization XRD pattern of Fe_3O_4, AC, and Fe_3O_4-AC can be seen in figure 3.

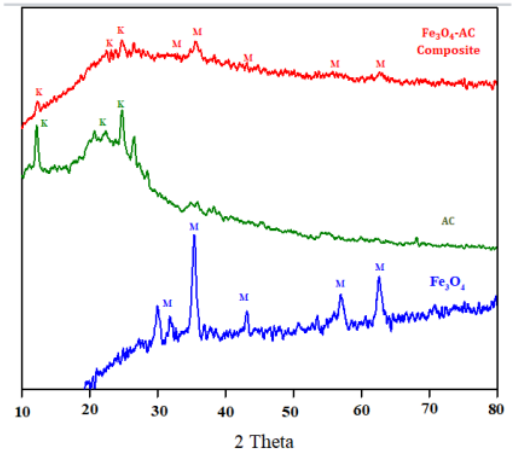

Figure 3. The XRD pattern of Fe_3O_4, AC, and Fe_3O_4-AC; diffraction peak (K= Kaolinite, M= Magnetite).

Diffraction peak of Fe_3O_4 is sharp peaks which indicate that is the crystalline structure at 2θ of 30.27°; 35.28°; 43.56°; 57.46° and 63.20° where the angles correspond to JCPDS-ICDD No.07-0322 for magnetic phase iron oxide Fe_3O_4 with a cubic structure. Meanwhile, in AC and Fe_3O_4-AC, a broad peak appears at 2θ of 20-30°. It indicates that the carbon is amorphous, although the intensity is relatively small in the Fe_3O_4-AC. Besides, other visible diffraction peaks were also seen at 2θ of 12.23°; 24.82° and 25.31°, which indicate that there is another phase besides carbon, namely the kaolinite phase found in activated carbon from the egg rack [6].

The determination of surface morphology on Fe_3O_4 and Fe_3O_4-AC was carried out using SEM. The following images of SEM analysis results can be seen in figure 4.

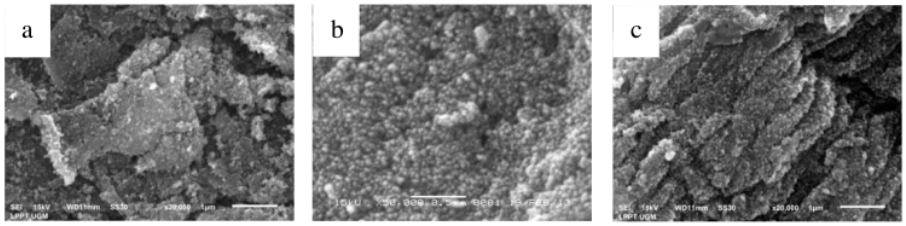

Figure 4. SEM image of Fe_3O_4 magnification of 20,000x (a) 50.000x (b) and Fe_3O_4 -AC magnification of 20,000x (c).

Based on images of SEM analysis results in figure 4, the surface morphology of Fe_3O_4 is a solid which is still heterogeneous but is dominated by small particles that tend to be spherical [11]. Whereas in the surface morphology of Fe_3O_4-AC is a layer covered by spherical particles with the denser pore structure, which is thought to have covered the surface of the AC, the composite's surface area is getting smaller. It will affect the results of the adsorption test when compared to AC.

The characterization of VSM to determine the magnetic properties of materials. The magnetic properties of Fe_3O_4 and Fe_3O_4-AC, as shown in figure 5.

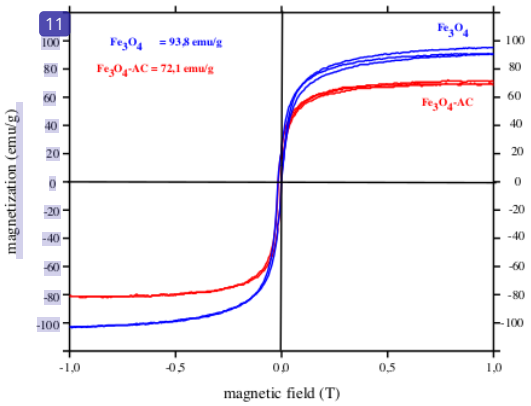

Figure 5. Magnetization curve of Fe_3O_4 dan Fe_3O_4-AC.

Based on the VSM hysteresis curve of figure 5, the Fe_3O_4 has a magnetic saturation value of 93.8 emu/g. Meanwhile, the Fe_3O_4-AC has a saturation value of 72.1 emu/g. The decrease in the magnetic saturation value is due to the non-magnetic fraction, namely AC in the Fe_3O_4-AC.

The effect of pH variations on the amount of adsorbed methylene blue (Qa) ions in AC and Fe_3O_4-AC can be seen in figure 6.

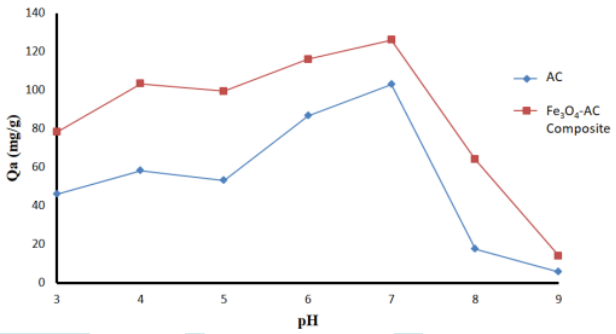

Figure 6. Effect of pH variations on the amount of adsorbed methylene blue (Qa) ions in AC and Fe_3O_4-AC.

Adsorption of methylene blue on AC and Fe_3O_4-AC at pH 3 was 45.9306 mg/g and 78.2745 mg/g, respectively. The tendency is to increase at pH 4, 5, 6, and the optimum at pH 7. The methylene blue uptake process using AC and Fe_3O_4-AC occurred at pH 7, with each Qa of 102.82 mg/g and 126,043 mg/g. There is no competition, either H^+ or OH^- ions at that pH. It indicates that thought to prevent methylene blue from being adsorbed on the adsorbent. Condition of acidic and alkaline are more H^+ and OH^- ions, and these ions will be adsorbed first on the adsorbent compared to methylene blue. So that H^+ and OH^- ions will seize the playful side of the adsorbent when adsorption takes place, and methylene blue will be difficult to bond to the adsorbent. That is why at acidic and alkaline pH, the amount of methylene blue ion adsorbed will be less than at neutral pH [12]. The adsorption methylene blue of Fe_3O_4-AC to be higher than AC.

4. Conclusion

The synthesis of Fe_3O_4-AC has been successfully carried out using the co-precipitation method by adding AC on alkaline conditions. Presence of a functional group of C-O bond and Fe-O bond at

Fe_3O_4-AC. XRD pattern of Fe_3O_4-AC was the presence peak of crystal structure Fe_3O_4, even though it's small intensity. SEM image of Fe_3O_4-AC was spherical with the denser pore of structure. The saturation magnetization of Fe_3O_4-AC was lower than Fe_3O_4. The optimum adsorption of methylene blue on the AC and Fe_3O_4-AC at pH 7. The performance of Fe_3O_4-AC was greater than that of AC.

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