The 6th International Conference of the Indonesian Chemical Society

Preface

The 6th International Conferences of the Indonesian Chemical Society (ICIC) 2017 was organized by the Department of Chemistry, Faculty of Mathematics and Natural Sciences, University of Sriwijaya in collaboration with the Indonesian Chemical Society. The theme for this conference was "*Stimulating of Advanced Perspective and Current Concepts on Chemistry Field*".

The goals of the conference were to provide a vehicle for the state of the art research results and trends in the chemistry field, to offer interaction, discussion and possible collaboration among chemists and the public about chemistry, to increase awareness of policy makers and the public on chemistry's rule in national development.

Therefore, this conference is a great opportunity not only for sharing knowledge and experience in chemical research, but also for starting a long and fruitful cooperation and friendship among Academicians, Researchers and practitioners of Chemistry.

In this conference, there were four keynotes speakers from USA, Japan, the Solomon Island, and Indonesia; and eight invited speakers. The 45 articles published here were picked from 229 participants who delivered them as oral or poster presenters.

Finally, I would like to thank to all participants and their respective institutions that have made this conference possible and I wish you all a pleasant meeting. I also would like to thank and congratulate the organizing committee for their dedication and tremendous efforts in organizing the conference.

Hermansyah,Ph.D.
Chairman of Organizing Committee
The 6th International Conference of Indonesian Chemical Society

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Synthesis Alumina-Activated Carbon Composite Using Sol-Gel Method As Adsorption for Methylene Blue Dye

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Synthesis Alumina-Activated Carbon Composite Using Sol-Gel Method As Adsorption for Methylene Blue Dye

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Abstract. The research about synthesis alumina-activated carbon composite using the sol-gel method as adsorption for methylene blue has been done. Activated carbon is made from durian shell. The alumina-activated carbon composite was characterized by FTIR (Fourier Transform Infrared), SEM-EDS (Scanning Electron Microscopy-Energy Dispersive X-Ray Spectroscopy. The FTIR characterization for activated carbon indicated that the functional groups O-H, C=C, C-H, and C-O while the alumina-activated carbon composite has increased of the functional group of Al-O. SEM analysis of the surface of alumina-activated carbon composite showed that alumina sticks to the activated carbon surface. EDS results showed a decrease of the element C from 64.60% to 20.87% and the increase of Al from 0.86% to 23.02%. The optimum condition adsorption of methylene blue using activated carbon obtained at an initial concentration of 25 mg/L, contact time of 75 minutes and a temperature of 55°C, while the composite alumina-activated carbon obtained at an initial concentration of 30 mg/L, contact time of 90 minutes and the temperature of 75°C. The ability of activated carbon and alumina-activated carbon composite for adsorption methylene blue were 10.7205 mg/g and 14.3662 mg/g, respectively.

Keywords : alumina-activated carbon composite, Adsorbtion, Methylen blue

1. Introduction

Nowadays research on composites is growing up. Composites are a macroscopic combination of two or more different components. The purpose of composite synthesis was to obtain new materials that have better properties than their constituent components. These properties include the ability to absorb a compound. Composite synthesis has been widely done from various materials one of them using activated carbon

Activated carbon is an amorphous carbon material having a large surface area of 300 to 2000 m^2/g. This large surface area is due to having a pore structure. These pores result in activated carbon having the ability to absorb compounds [1]. Activated carbon can be synthesized from a variety of basic materials such as gelam wood [2], cassava shell [3], coconut shell [4] and durian shell [5].

Durian shell contains cellulose content of about 50%-60% (carboxymethylcellulose) and 5% lignin [6]. Durian shell is very potentially as a base material to make activated carbon because it has a

high enough carbon content of 60.31% [5]. Durian shell activated carbon has micropore and mesoporous structures. The presence of micropores and mesopores in activated carbon increases the absorbency, especially for large adsorbate molecules such as dyes. The synthesis of activated carbon from durian shell has been successfully done to absorb the methylene blue dye [7].

The ability of the activated carbon depends on the surface area, the internal structure of the pore, the surface characteristics and the functional group on its porous surface [7]. Addition alumina for activated carbon to composite is expected to increase the ability [8]. Alumina (Al_2O_3) is a porous material with macropore and mesoporous structures and has an active site in surface and thermostable [9] so that the adsorption process can be done at high temperature. The alumina surface active site is expected to interact with the methylene blue dye. The alumina-activated carbon composite can increase absorption because the adsorption process occurs physically and chemically. Activated carbon has pores that can absorb while alumina has functional groups that can interact with adsorbate [8].

Many methods can be used to synthesize composites, one of which is sol-gel method. The sol-gel method has several advantages: lower used temperature, shorter process, and lower pollution [10]. The alumina-activated carbon composite synthesized by the sol-gel method has a high absorbency to acetone vapor [11]. The sol-gel method has been successfully used to make Nanocomposite TiO_2-Carbon Nanotubes [12] and Nanocrystalline Metal Oxide [10]. Iriani [2] also succeeded in synthesizing the alumina-activated carbon composite from the gelam wood using the sol-gel method as adsorption the methylene blue dye.

Methylene blue is a toxic dye [13]. Methylene blue can cause irritation of the gastrointestinal tract if ingested, causing cyanosis if inhaled, and irritation of the skin if touched by the skin [14]. Methylene blue is often used in the process of staining in various industries such as textile, ceramics, paper, printing, and plastics industries. In the process of coloring, the industry uses a lot of water and many produce liquid waste containing dye [7].

In this study, the activated carbon of durian shell was modified by alumina using sol-gel method. Activated carbon with alumina is applied for adsorption of methylene blue dyes. Characterization of alumina-activated carbon composite includes functional group analysis using FTIR and morphology using SEM-EDS.

2. Materials and Methods

The chemicals used are analytical grades such as KOH, HCl, NaOH, $Al_2(SO_4)_3$, $NaNO_3$ (was supplied from Merck), methylene blue dye, and aquadest. Durian shell was collected from Pasar Kuto Palembang. Prior to the process, durian shell was repeatedly washed with distilled water in order to remove dust and other inorganic Impurities. The size of these materials was reduced to ±2x2 cm and then dried at 120^{o}C for 24 h to reduce the moisture content. Subsequently, as dried durian shell was grounded until it became powder (140 mesh) and then stored in desiccators [5].

2.1 Characterization

Analysis instrument were used Spectrophotometer UV-Vis Spectronic 20. The pore structure characteristics of composite and activated carbon were determined using, SEM-EDS 6510-LA (operated using argon gas with a current of 6mA for 4 minutes and observed at a voltage of 15 Kv)

and FTIR Shimadzu 5000 (sample was measured as a mixture on KBr pelleted samples were scanned over the wavenumber range 400-4000 cm^{-1}). Characterization moisture content and ash content of activated carbon using SNI No. 06-3730-1995 method.

2.2 Synthesis of Alumina-Activated Carbon Composite

Preparation of activated carbon was conducted according to [5]. A 25 g of durian shell in the form of powder was mixed with KOH solution (50%). During the impregnation period, the mixture was stirred at 200 rpm for 5 h at room temperature. The resulting homogeneous slurry was dried at 110^{o}C for at least 24 h.The dried product carbonized at 500^{o}C for a 1 hour. These activated carbons were washed sequentially with a 0.1 M HCl solution. Consecutively, carbon powders were repeatedly washed with hot distilled water until the pH of the solution reaches 6.8 and finally washed with cold distilled water. After that, these powders were dried at 110^{o}C for 2 h and stored in a desiccator.

Synthesis of alumina-activated carbon composite was conducted according to [11]. Synthesis of alumina-activated carbon composite the sol-gel method was used by adding the activated carbon into 1 M $Al_2(SO_4)_3$ 50 mL, later 6 M NaOH by portions. Wet alumina activated-carbon derivates were granulated by extrusion, then dried at 120^{o}C for 4 h and heated at 420^{o}C for 4 h. The amount of activated carbon in the adsorbents was ~5% (wt).

2.3 pH_{PZC}

The procedure of pH_{PZC} was conducted according to [15]. The determination of pH_{PZC} using 50 mL of 0.01 M $NaNO_3$ solution was included in 10 Erlenmeyer respectively. Initial pH (2, 3, 4, 5, 6, 7, 8, 9, 10, 11, and 12) was performed by adding a 0.1 M HCl or 0.1 M NaOH solution. 0.1 g of composite is added to each further Erlenmeyer in the shake for 2 hours. The mixture was allowed to stand for 2 days and the final pH was measured.

2.4 Adsorption Experiments

Optimum condition adsorption of methylene blue dye using adsorbent o,1 g composite and activated carbon by durian shell each added into 50 mL methylene blue pH 7.25 by varying the concentration (10, 15, 20, 25, 30, and 35 mg/L), contact time (60, 75, 90, 105, and 120 min) and temperature variations (35, 45, 55, 65, 75, and 85^{o}C). The mixture was stirred at 120 rpm for 60 minutes. The solution of the methylene blue substance was separated by filtration and then measured its concentration by using UV-Vis spectrophotometer.

The amount of dye adsorbed is calculated based on the following equation:

$$q_t = \frac{(C_o - C_t)\,V}{m}$$

3. Results and Discussion

3.1 Water Content and Ash Content

Based on Table 1 it can be seen that the activated carbon of durian shell has good water content because it has fulfilled the requirement of active carbon quality according to SNI No.06-3730-1995, but the ash content has not fulfilled the requirement because it exceeds the maximum limit.

Table 1. Characteristics of Activated Carbon

Characteristics of Activated Carbon	Research Result	SNI No. 06-3730-1995 (powder)
Water Content (%)	3.6494	Max. 15
Ash Content (%)	13.5877	Max. 10

The amount of content ash is caused by the amount of mineral content in activated carbon. The process of heating when the manufacture of activated carbon is not flowed by nitrogen gas, this is caused inside the furnace there is still oxygen. This oxygen causes the formation of oxide minerals on the activated carbon produced. This metal oxide causes high ash levels. The metal is according to EDS results (Table 3) ie Mg, Si, K, Ca, and Fe. The concentration of HCl used for washing affects the amount of ash content. Washing process should use HCl with concentration more concentrated, this is like done by [5] using HCl 0.5 N in the washing process.

3.2 Characterization

The result of the synthesis of active alumina-carbon composite using sol-gel method with 5% carbon content showed smaller size and uniform [2]. Figures 1a and b show the activated carbon and alumina-activated carbon composite. The alumina-activated carbon composite is lighter than the activated carbon. This is due to the presence of alumina on the activated alumina-carbon composite.

(a) (b)

Figure 1. Image of (a) Activated Carbon and (b) Composite Alumina-Activated Carbon

FTIR. FTIR spectroscopy is used to identify functional groups. The functional group identification is used to determine the chemical interaction between the active functional groups in the alumina, the activated carbon, and the alumina-activated carbon composite. The FTIR spectra alumina, activated carbon, and alumina-activated carbon composite were shown in Figure 2. The characteristic band of alumina, activated carbon, and alumina-activated carbon composite at 3450.4, 3446.5, and 3448.5 cm^{-1} is the peak of O-H. The functional group of O-H on alumina comes from the remaining H_2O. Activated carbon and composite showed functional groups C=C at wavenumbers 1627.8 and 1639.4 cm^{-1} respectively. Alumina shows peak C=C at wave number 1639.4 cm^{-1} caused by impurity on the alumina. The activated carbon C-O functional group appears at 1022.2 cm^{-1} wavenumbers.

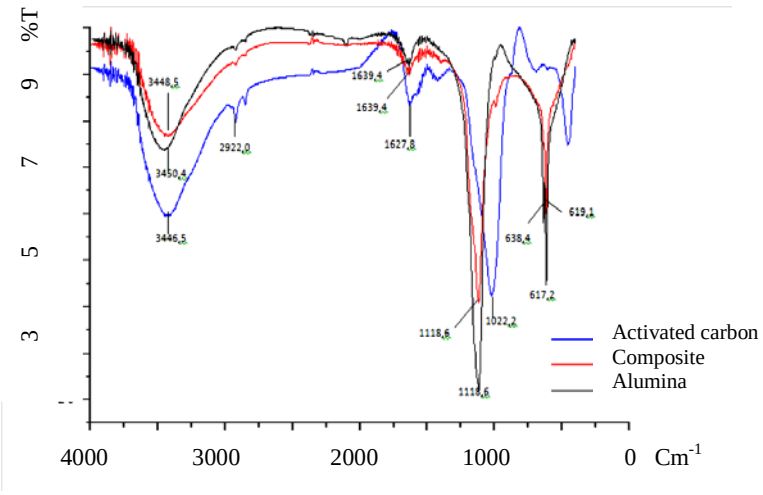


Figure 2. FTIR spectra of Alumina, Alumina-Activated Carbon Composite

Alumina and composite alumina-activated carbon have peaked at 1118,6 cm^{-1} indicating the SO_4^{2-} free functional group. this is indicated by the presence of a peak at 617.2 cm^{-1} in alumina and 619.1 cm^{-1} in the composite [16].The SO_4^{2-} functional group is derived from $Al_2(SO_4)_3$. The other broad spectra showed at 620 cm^{-1} to 850 cm^{-1} is the peak of Al-O group [11]. Alumina and alumina-activated carbon composite show Al-O functional group at wave number 638,4 cm^{-1}. The FTIR spectra of alumina, activated carbon and alumina-activated carbon composite can be seen in Figure 2.

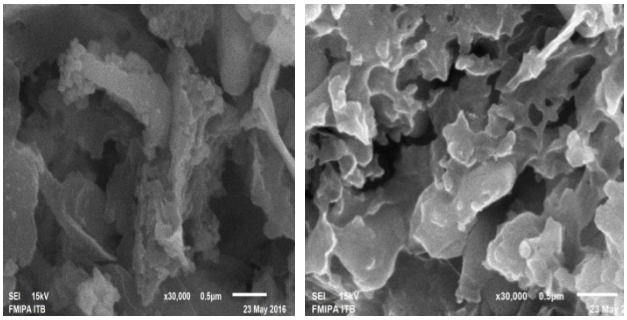
Table 2. Functional Group and Wavenumber of Functional Group

Functional Group	Wavenumber of Functional Group(cm^{-1})		
	Alumina	Activated Carbon	Alumina-activated carbon composite
O-H	3450.4	3423.4	3448.5
C-H	-	2922.0	-
C=C	-	1627.8	1639.4
C-O	-	1022,2	-
SO_4^{2-}	1118.6	-	1118.6
	617.2		619.1
Al-O	638.4	-	638.4

Shifts occur only in adjacent areas (Table 2). It indicates an interaction of the activated alumina-carbon composite, i.e. the physical interaction. The active alumina-carbon composite does not appear new peak. The resulting peak indicates a functional group derived from activated carbon and alumina.

SEM-EDS. Figures 3a and b show the morphology of activated carbon and alumina- activated carbon composites with 30,000× magnifications. The composite synthesis results show alumina attached to the surface of the activated carbon and some enter into the pores of activated carbon. The

white granules on the composite surface show alumina, Figure 3b. The entry of alumina causes some of the pore activated carbon is closed. The surface of the activated carbon initially has a larger pore but after modification, the alumina closes part of the pore so that the pore becomes smaller.



(a) (b)

Figure 3. Morphology (a) Active Carbon and (b) Alumina-Activated Carbon Composite

The adsorbent EDS spectra showed percent of active carbon element and active alumina-carbon composite (Table 3). Activated carbon and activated carbon each contained C of 64.60% and 20.87% respectively. The composition of the alumina-activated carbon composite changes the elemental composition. The presence of Al in the activated carbon shows the presence of alumina adsorption into the activated carbon [17].

Table 3. Element Analysis

Element	Activated Carbon (%)	Alumina-Activated carbon Composite (%)
C	64.60	20.87
O	13.39	39.09
Al	0.86	23.02
S	-	9.86
Na	-	4.41
Mg	0.86	0.18
Si	7.59	1.81
K	4.35	0.77
Ca	1.15	-
Fe	7,18	-

a. pH_{PZC}

pH Point Zero Charge (pH_{PZC}) is a state when the surface of the adsorbent is neutral. The pH_{PZC} value is determined by the point of intersection of the initial pH curve and the final pH. The pH_{PZC} data was

used to estimate pH conditions suitable for the adsorption process. Based on Figure 4, pH_{PZC} of activated carbon of durian shell is 7.10 (surface of the activated carbon is neutral). At pH <7.10 the surface of the activated carbon is positively charged to form $R\text{-}OH_2^+$ so it is easy to absorb the anions, whereas at pH> 7.10 the surface of the activated carbon is negatively charged to forms $R\text{-}O^-$ so it is easy to absorb the cation [2].

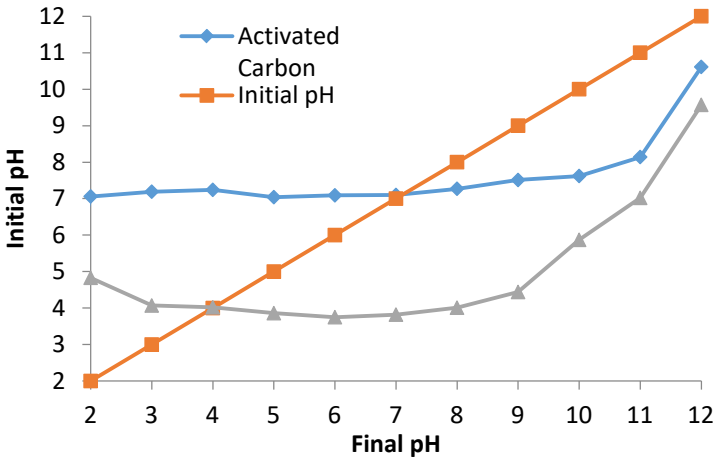


Figure 4. Curve of pH_{PZC}

Composite pH_{PZC} was obtained at pH 4.02. This pH indicates that the composite surface is neutral with the presence of a neutral Al-OH group and a zero charge. When the pH <4.02 hydroxyl group on the alumina can experienced protonation to form $Al\text{-}OH_2^+$ causes the composite surface to be positively charged so it easily absorb the anion. At pH> 4.02 hydroxyl group on the alumina can experience deprotonation to form $Al\text{-}O^-$ so the composite surface is negatively charged so it easily absorbs the cations [17].

pH_{PZC} measurements show that there is no pH adjustment during the adsorption process. The pH of methylene blue (7.25) is already above the activated carbon pH_{PZC} as well as the alumina-activated carbon composite. At the pH the adsorbents are negatively charged and easily absorb the positively charged methylene blue.

b. Methylene Blue Dye Adsorption

Figure 5 shows the optimum concentration of methylene blue dye on the activated carbon and composites 25 mg/L and 30 mg/L, respectively. During the adsorption process, the color molecules first diffuse into the surface of the adsorbent and then diffuse into the adsorbent pores [14]. The composite has an active site of alumina so that the adsorption process is not only played by pore but also played by alumina. This results in higher composite absorption than activated carbon. This alumina will bind to the methylene blue dye ions.

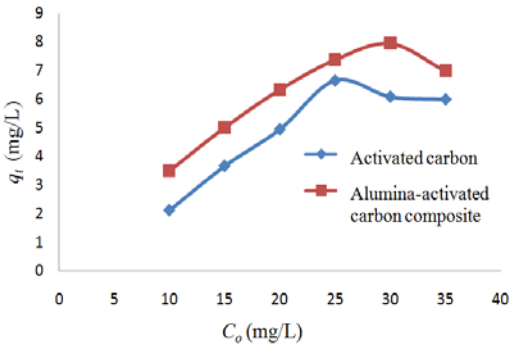


Figure 5. Effect of Concentration

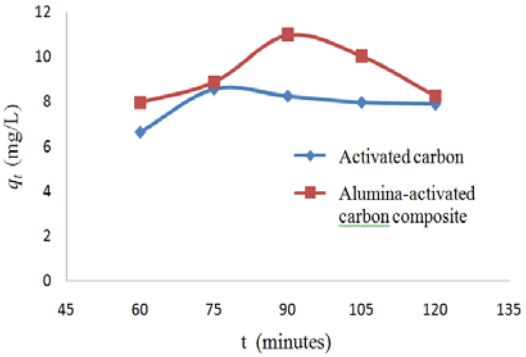


Figure 6. Effect of Contact Time

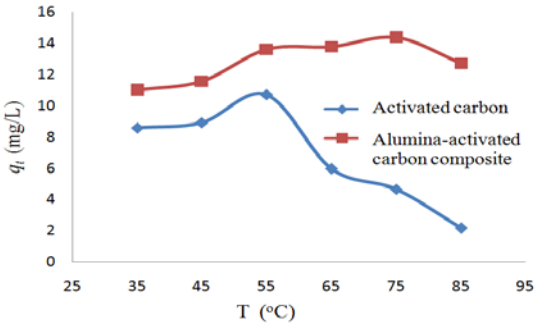


Figure 7. Effect of Temperature

Figure 6 shows the optimum contact time of activated carbon obtained at 75 minutes at 8.5823 mg/g while in the composite at 90 minutes of 10.9826 mg/g. Composites are able to absorb more dyes because the composite has an active site of alumina. Increased contact time causes more adsorbent particles to be in contact with dye ions. This causes more and more dyes ions to be absorbed by the adsorbent. This condition will continue until it reaches saturation condition or optimum contact time [18]. If the adsorbent has been saturated by the dye ions, the increased contact time does not increase adsorption, it will be desorption. This desorption is influenced by continuous stirring so that the dye ions is released again.

Figure 7 shows the optimum temperature of activated carbon and composite absorption obtained at 55°C of 10.7205 mg/g at 75°C of 14.3662 mg/g respectively. The alumina contained in the composite results in a more thermostable composite than the activated carbon. This results in higher optimum absorption temperature composite than absorption using activated carbon. Adsorption of organic components (including dyes) involves a physical bond that will decrease with increasing temperature. Increased temperatures involve greater kinetic energy so that collisions between particles are more frequent. This collision causes the dyes ion to be desorbed.

4. Conclusions

Characteristics of using FTIR showed that the active alumina-carbon composite was physically interacting. SEM analysis showed that alumina sticks to the activated carbon surface. EDS results showed a decrease of the C element and the increase of Al element. The results of the optimum condition analysis show that the alumina-activated carbon composite absorption is higher than the activated carbon.

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