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Methyl Orange Dye Removal through Adsorption using TiO_2 modified Montmorillonite Granule

Dewi Yuliana* & Erfan Priyambodo *
* Department of Chemisty, Faculty of Mathematics and Natural Sciences, Universitas Negeri Yogyakarta

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Corresponding Author:

Dewi Yuliana,

Department of Chemisty,
Universitas Negeri Yogyakarta

Email:
dewiyuliana.2018@student.uny.ac.id

ABSTRACT

This research was conducted to determine the characteristics of TiO_2 modified montmorillonite granule and to determine the effectiveness of its filtration against methyl orange dye. Montmorillonite granule particles were made through a pressing process and continued with sintering at 900°C. The granules obtained were coated with TiO_2 photocatalyst by the deep coating method. The montmorillonite granules were characterized using SEM-EDX and the methyl orange dye adsorption process was carried out using the column method. The SEM analysis showed that the granules is porous materials and the EDX confirm the increasing of TiO_2 content in montmorillonite before and after being coated with TiO_2 from 0.50% to 22.66%. The best adsorption effectiveness of methyl orange was 96.6% at the flow rate of 0.0083 L/hour, which occurred in the use of TiO_2 modified montmorillonite granule.

Keyword: Adsorption; methyl orange; granules; montmorillonite; TiO_2

1. INTRODUCTION

Textile dye waste that is disposed of directly into the environment without prior processing can result in environmental pollution (Chen & Burns, 2006). This is because textile dye waste contains hazardous substances, such as synthetic dyes, suspended solids and dissolved organic substances (Benkli, et al, 2005). The use of synthetic dyes is an alternative strategy for an industry because it has more stable properties compared to natural dyes, is easy to obtain, and has an economical price (Beldean-Galea, Copaciu & Coman, 2018). Methyl orange is a synthetic dye belonging to an azo compound which is widely used in various industrial fields such as the textile, paper, and leather industries (Mehra & Sharma, 2012)

Adsorption is a process of absorbing molecules (adsorbate) on the surface of another substance (adsorbent) caused by the attraction between the two substances (Ngapa, 2017). The adsorption process can be carried out in two ways, namely static (batch) and dynamic (column). The adsorption process with the column system is more profitable than the batch system because in general the column system has a larger capacity making it suitable for large-scale applications (Meneghetti, Baroni & Vieira, 2010).

In general, the adsorption process is carried out using various types of adsorbents, including activated carbon, coconut shell charcoal, rice husk activated carbon, and activated sand. Another adsorbent that can be used in the textile dye adsorption process is montmorillonite because it has a high surface area and cation exchange capacity (Machiril, Jumaeri & Kusumastuti, 2017). Montmorillonite is a powder that is easy to absorb water and expands easily so that it is easy to change the particle size by modifying heating so that it becomes more stable when used as an adsorbent (Tan, 1991).

The montmorillonite material preparation process can be done by changing the particle size by combining the montmorillonite powder into a larger size (granule) through the sintering process. Granules are a collection of small particles with an uneven shape and combine with each other to form larger particles. The average particle size of the granules is 1 - 3 mm with a sieve size of approximately 18 - 6 mesh (Sinala, 2016).

Montmorillonite has properties that easily absorb water (Rong, et al, 2021), so modifications are needed so that the montmorillonite grains can work optimally. Montmorillonite granules can be modified by coating photocatalyst compounds so as to increase the performance of the adsorbent. One of the most widely used semiconductor materials as photocatalysts is TiO_2 (Aliah & Yuni, 2015; Al Jitan, Palmisano & Garlisi, 2020), which has several advantages, including good optical properties, non-toxicity, economy, and good photocatalytic activity (Pang, Kho & Chin, 2012), a semiconductor with a wide energy band gap, and its abundant availability (Hema, et al, 2013).

This study examines the adsorption of methyl orange dye with a column system using TiO_2 modified montmorillonite granule which aims to determine the characteristics of TiO_2 modified montmorillonite granule and determine the effectiveness of TiO_2 modified montmorillonite granule adsorption against methyl orange dye.

2. RESEARCH METHOD

Montmorillonite powder was analyzed using XRD instrument to determine the structure of montmorillonite. Montmorillonite granules are made by pressing a mixture of montmorillonite and water, followed by sintering and crushing them into granular sizes. The montmorillonite granules were then coated with TiO_2 by a deep coating technique. Granule characterization was carried out using the SEM-EDX instrument. The granules that have been obtained are then put into the adsorption column as much as 11 grams, then the methyl orange dye with a concentration of 28.7 ppm is flowed into the column in a down flow manner. The adsorption results that have been obtained were characterized using a UV-Vis spectrophotometer.

3. RESULTS AND ANALYSIS

3.1 Characterization Montmorillonite Powder

The analysis used to confirm the montmorillonite sample is with the XRD instrument. The data obtained from the analysis are used to determine the crystal structure of the material and its purity. The diffractogram of the montmorillonite material in this study is presented in Figure 1.

Figure 1. Diffractogram of montmorillonite powder

Based on Figure 1, it can be seen that there are sharp peaks, this indicates that this material has a crystalline part. The results of the characteristics of montmorillonite used in the study showed that there were peaks of 2-theta (2θ) montmorillonite, at 5.56°, 19.66°, 26.48°, 34.96°, dan 61.70°. So that it can be seen that the main material used in this study is montmorillonite. However, the main ingredient is not pure montmorillonite because there are still some impurity components contained in it, this can be seen in Figure 1. Another peak found in the XRD analysis results is quartz, where quartz is a non-clay mineral component.

3.2 TiO_2 modified Montmorillonite Granule

Montmorillonite is an alumina silicate species, where the alumina silicate can be utilized so that it is expected to obtain a modified alumina compound product with better physical and chemical properties than before (Leonard, 1995). One way that can be used to improve the physical and chemical properties is by pillarization. Pillarization of montmorillonite granules was carried out by coating the TiO_2 photocatalyst compound so as to increase the performance of the adsorbent.

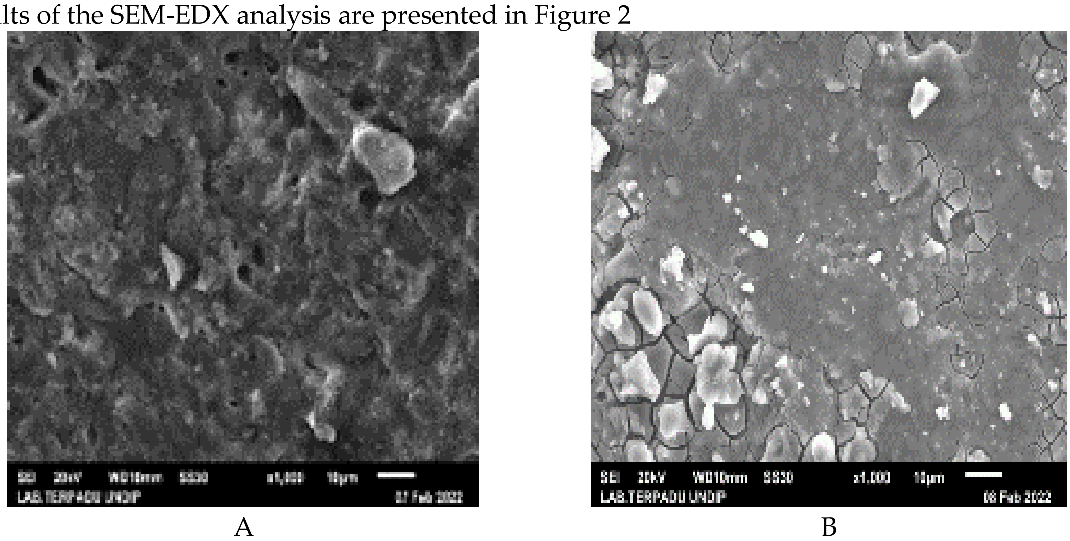
The TiO_2 coating process is carried out by dipping the montmorillonite granules into the $Ti(OH)_4$ solution obtained from mixing the $TiCl_4$ solution, ethanol solvent, and ethylene glycol. The result of TiO_2 coating process is shown at Figure 2.

A B

Figure 2. A. Montmorillonite granules; B. TiO_2 modified montmorillonite granule

3.3 Scanning Electron Microscopy–Electron Dispersive X-ray (SEM-EDX) Analysis

The montmorillonite granules was characterized using the SEM-EDX instrument. The results of the SEM-EDX analysis are presented in Figure 2



A B

Figure 2. SEM analysis of: A (montmorillonite granules); B (TiO_2 modified montmorillonite granule)

Based on Figure 2 (a) it can be seen that the montmorillonite granules still have many gaps, while in Figure 2 (b) the montmorillonite granules that have been coated with TiO_2 have started to be covered and there are small particles that stick to the surface of the material. These results indicate that the gaps on the montmorillonite surface have been filled with TiO_2.

The EDX analysis obtained data on elemental components in the granules, which are presented in Table 1.

Table 1. EDX analysis of the granule

Test Parameters	The ratio (%)	
	Montmorillonite granules	TiO_2 modified montmorillonite granule
C	8,49	11,74
MgO	4,91	3,17
Al_2O_3	16,93	11,34
SiO_2	62,14	41,10
K_2O	0,44	0,52
CaO	1,86	1,42
TiO_2	0,50	22,66

Table 1 shows an increase in the percentage of TiO_2, where the elemental component of TiO_2 in montmorillonite granules is 0.50%, while in montmorillonite granules that have been coated with TiO_2 it is 22.66%. Based on the SEM-EDX data, it can be seen that TiO_2 successfully adhered to the montmorillonite granules.

3.4 Methyl Orange Dye Removal through The Adsorption

The maximum wavelength of methyl orange was determined by measuring the absorption using a UV-Vis spectrophotometer in the visible range of 300-700 nm so that a maximum wavelength of 462 nm was obtained. The maximum wavelength is then used to determine the absorbance of the standard solution of methyl orange. The regression line equation obtained was used to determine the concentration of methyl orange waste. The results of methyl orange dye removal through the adsorption using granules are presented in Table 2.

Table 2. The results of methyl orange dye removal through the adsorption using granules

Flow rates (L/hour)	Influent Concentration (ppm)	Montmorillonite granules		TiO_2 modified montmorillonite granule	
		Effluent Concentration (ppm)	Effectiveness (%)	Effluent Concentration (ppm)	Effectiveness (%)
0,0083	28,7	10,007	65,1	0,963	96,6
0,017	28,7	11,589	59,6	1,205	95,8
0,033	28,7	12,352	56,9	1,577	94,5

Based on the data obtained, it is known that the slower the flow rate, the lower the effluent concentration obtained and the higher the adsorption effectiveness. The longer the contact time, the more dye will be adsorbed, because the particles have more time to come into contact with the dye. This causes a lot of dyes to be bound in the adsorbent (Gultom & Turmuzi, 2014). The effectiveness of adsorption using TiO_2 modified montmorillonite granule was higher than that of adsorption using montmorillonite granules. This shows that the coating of TiO_2 photocatalyst compounds on montmorillonite granules can improve the performance of the adsorbent.

4. CONCLUSION

The results of SEM characterization showed that the montmorillonite granules had many gaps on the surface while the montmorillonite granules that had been coated with TiO_2 compound had the gaps covered and at the surfaces there were small particles attached to the montmorillonite particles. The EDX results showed that there was an increase of TiO_2 before and after being coated, from 0.50% to 22.66%. The best adsorption effectiveness occurred in the use of TiO_2 modified montmorillonite granule with a flow rate of 0.0083 L/hour with 96.6% of.the adsorption effectiveness.

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