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Morphology Characteristic Study of Adsorbent Prepared from Sidoarjo Hot Mud

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ABSTRACT

Sidoarjo hot mud has erupted for the past 16 years and has many effects on the environment. This mud contains several porous materials, such as silica and aluminum oxide, which can be used as adsorbents. This study aimed to characterize the morphology of the adsorbent and apply it to waste cooking oil purification. The composition of virgin hot mud was 46.5% SiO_2 and 13% Al_2O_3 with an amorphous crystalline structure. The hot mud was activated with H_2SO_4 under different drying process conditions where the drying temperature differed from 150°C to 250°C, while the drying period was set from 30 to 90 minutes. After the activation, the structure of the adsorbent became more crystalline. From XRD analysis, the lowest intensity resulted from adsorbent with 225°C and 75 minutes of drying, with 191,038 m^2/gr on pore surface area. This adsorbent had a greater pore surface area and gave the best adsorption process.

Keywords: Adsorption, bleaching agent, mud, waste cooking oil

Introduction

Sidoarjo hot mud, known as Lapindo mud, was one natural disaster in 2006 at Porong, Sidoarjo, East Java, Indonesia. Some errors during the drilling process caused the geothermal mud eruption. The eruption continued and was predicted will be lasted for 31 years. The mud affected the environment, like the water bodies and livelihoods of water organisms (McMichael, 2009; Triyono et al., 2015; Wulandari et al., 2022).

The hot mud contains many chemicals, especially Al_2O_3, SiO_2, and Fe_2O_3 (Triyono et al., 2015). These materials were suitable for adsorbents that should have high porosity and a large surface area. Using acid activation would modify the adsorbent properties (Filho & do Carmo, 2004; Liu et al., 2008).

Waste cooking oil was kitchen waste that is hazardous to the environment and human health if disposed of without treatment (Foo et al., 2021). The used cooking oil color could change and generate an unpleasant odor due to reactions such as hydrolysis, polymerization, and oxidation after contact with air and high temperature (Miskah et al., 2019; Miyagi & Nakajima, 2003). Besides, the degradation of the oil could produce compounds like ketones, aldehydes, and polymers (Ningsih et al., 2021). Hence, the waste cooking oil must be treated before being thrown into the environment.

The purification process that can be applied to treat used oil is the adsorption process. Several studies have learned about the effectiveness of adsorbent from bentonite and activated carbon. Several raw materials for activated carbon could be used, like coconut shells (Khuzaimah & Eralita, 2020) and durian peel (Miskah et al., 2019).

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This research aims to process Sidoarjo hot mud becomes an adsorbent that could be used in waste cooking oil purification. The morphology of the characteristics of the adsorbent also learned to know the compositions and the structures. In addition, the performance of the adsorbent was also tested.

Material and Methods

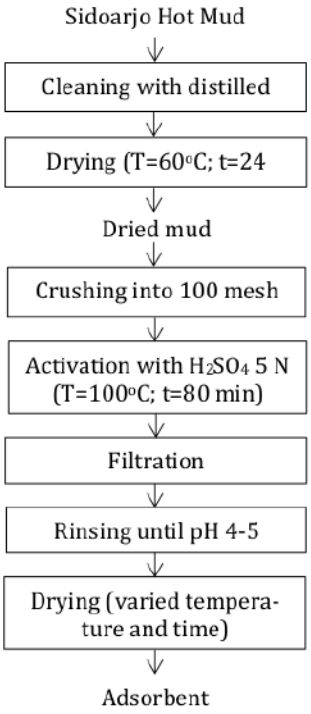

Figure 1. Process diagram for adsorbent production

Material

The adsorbent was prepared from Lapindo hot mud from Sidoarjo, East Java, Indonesia. It was taken 400 meters away from the burst center. For activation, the adsorbent was contacted with 97% sulfuric acid diluted with distilled water. The adsorbent performance was tested in waste cooking oil collected from the traditional market.

Method

The preparation of the adsorbent (Figure 1) was started by cleaning the mud with distilled water. The mud was dried using an oven at 60°C for 24 hours to remove the water content. The dried mud was then crushed and conditioned at 100 mesh on size. The composition and structure of the mud were analyzed using XRF (X-Ray Fluorescence) and XRD (X-Ray Diffraction).

The mud activation was done by adding 500 mL H_2SO_4 5 N and 100 gr dried mud in a stirred vessel. The mixing condition was set at 100°C for 80 minutes. The activated mud was filtered and rinsed with distilled water until the pH was up to 4 or 5. Then, it dried again in the oven at varied times and temperatures. The drying time was set for 30 until 90 minutes, while the temperature was varied from 150 to 250°C. The adsorbent was analyzed using XRD to characterize the material properties.

The adsorbent as a bleaching agent was added to 100°C waste cooking oil. The contact time was 80 minutes while kept stirred and heated. After the adsorption process, the adsorbent was separated by filtering.

Results and Discussion

In this study, the hot mud was analyzed whether before or after activation. Analysis was carried out using XRF to determine its composition. Based on the XRF results, the adsorbent material to be used is composed of several compounds, as listed in Table 1. The most abundant component was silica (46.5%) and aluminum oxide (13%). These two components have a porous structure that makes the hot mud can be used as a natural adsorbent (Golomeova & Zendelska, 2016).

Table 1. The XRF result of Sidoarjo's hot mud

No	Compound	Composition (wt %)
1.	Aluminum oxide (Al_2O_3)	13
2.	Silica (SiO_2)	46.5
3.	Sulfite (SO_3)	2.2
4.	Potassium oxide (K_2O)	2.75
5.	Calcium oxide (CaO)	6.195

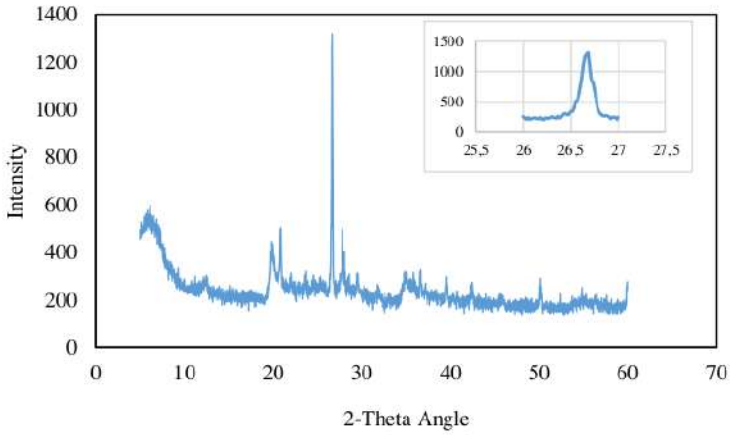

Figure 2. Diffraction pattern of non-activated hot mud

The crystal structure of the hot mud was also determined with XRD analysis. The XRD analysis result was shown in Figure 2 to Figure 7. Analysis was done at 2-theta angle 5° to 60° with 0.15406 nm X-ray wavelength from copper metal. From Figure 2, the highest intensity of non-activated hot mud was 1295 at 26.6661°. This condition indicated that the crystal structure is amorphous because it could not occupy the lattice without activation.

The adsorbent diffraction pattern of 30 and 40 minutes drying of activation procedures (Figure 3 and Figure 4) were dissimilar to the non-activated adsorbent. Several angles at 20-30° have not appeared that indicate crystal structure has become more crystalline. The heating on drying process made atoms fulfill the lattice and ordered crystalline structure.

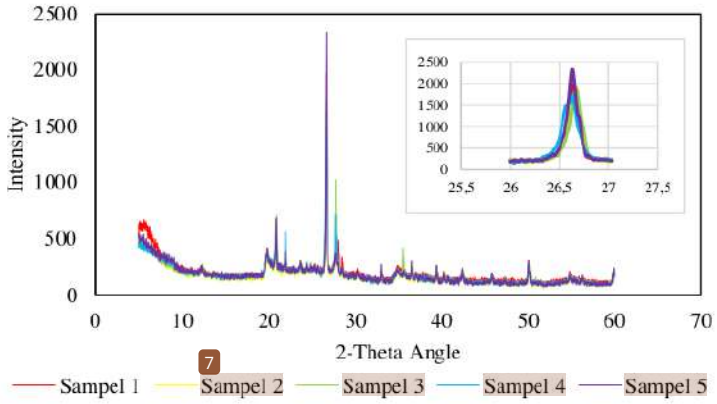

Figure 3. Diffraction pattern of hot mud adsorbent activated in 30 minutes of drying

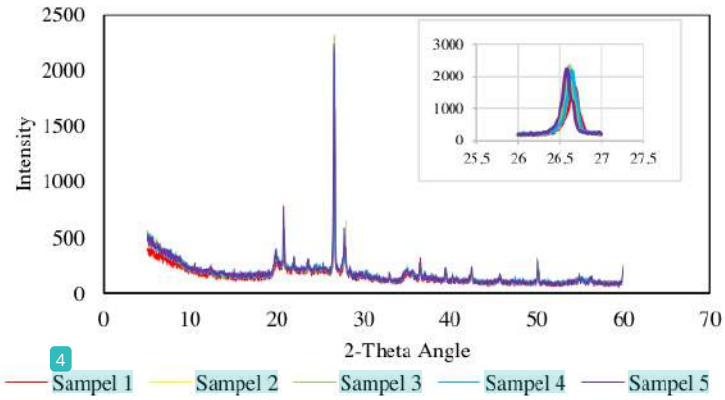

Figure 4. Diffraction pattern of hot mud adsorbent activated in 45 minutes of drying

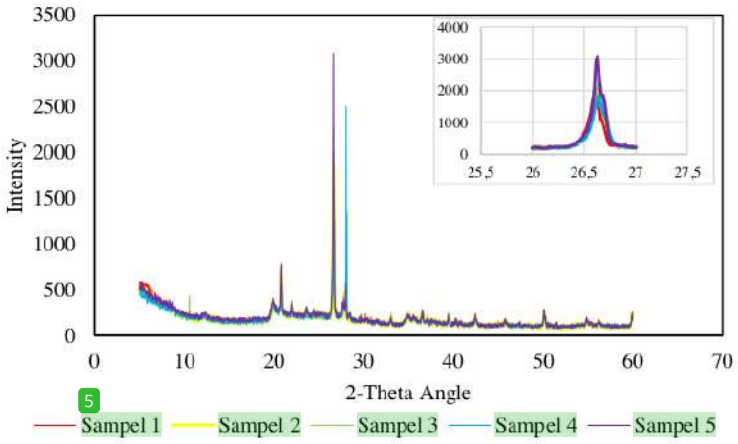

Figure 5. Diffraction pattern of hot mud adsorbent activated in 60 minutes of drying

When the drying process was set at 60 minutes (Figure 5), the intensity was increased. Higher intensity implied a greater particle size. The highest intensity was achieved on sample 5, where the drying temperature was 250°C. On other conditions, the intensity was not much different from each other.

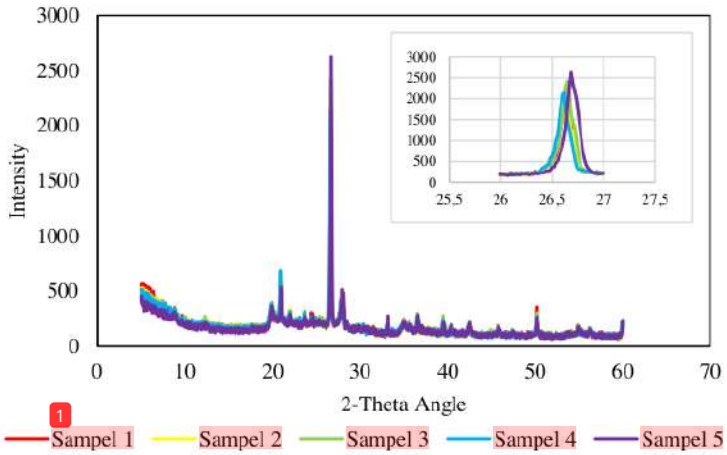

Figure 6. Diffraction pattern of hot mud adsorbent activated in 75 minutes of drying

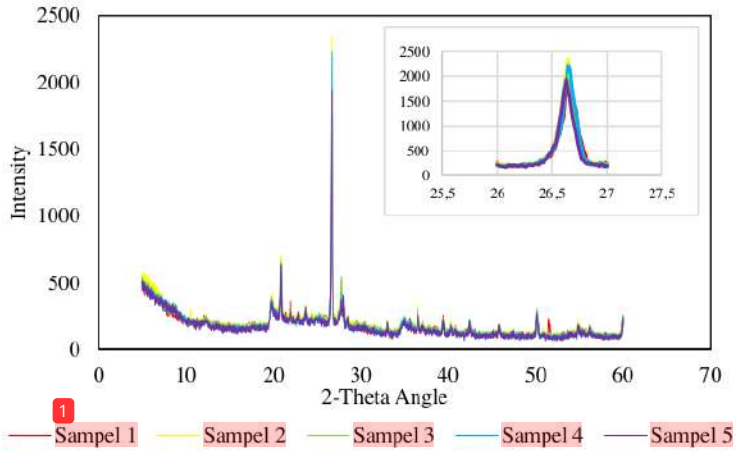

Figure 7. Diffraction pattern of hot mud adsorbent activated in 90 minutes of drying

The intensity values from XRD analysis for the adsorbent (Table 2) fluctuated at every drying time and temperature. The lowest intensity was achieved when the drying process took place at 225°C and 75 minutes. In contrast, the highest intensity was obtained at 250°C and 60 minutes drying process. If those results were compared with BET analysis, the pore surface area of the adsorbent was 191,038 m^2/gr for the sample with 225°C and 75 minutes of drying. And for the adsorbent with 250°C and 60 minutes of drying, the pore surface area was 184,402 m^2/gr. The intensity value is related to the pore surface area of the adsorbent. With higher intensity, the size of the crystal particle was getting bigger, which decreased the pore surface area.

Table 2. The intensity value of hot mud adsorbent at each drying condition

Drying period (minute)	Intensity				
	Drying temperature				
	150°C	175°C	200°C	225°C	250°C
30	1973	1665	1514	1740	2339
45	1289	1728	2315	1893	1539
60	1591	1744	2079	1828	3078
75	1404	1216	1627	1060	2625
90	1747	2341	1930	2224	1729

Figure 8. Hot mud adsorbent testing on waste cooking oil

The purification process was done using hot mud adsorbent with the best characteristic (Figure 8). The waste cooking oil had darker color due to oxidation and other reactions. The oil's color became lighter after the adsorption process took place. It indicated the adsorbent was worked by adsorbing the coloring components (Silva et al., 2014).

Conclusion

Sidoarjo hot mud contains silica (46.5%) and aluminum oxide (13%) that was promised to be processed into adsorbent. The hot mud goes through several processes before being applied for waste cooking oil purification. The morphology of the hot mud was defined with XRD analysis, where the non-activated hot mud had an amorphous crystal structure, and the activated hot mud was crystalline. The lowest intensity was achieved with a drying condition at 225°C for 75 minutes. With this condition, the pore surface area was 191,038 m^2/gr, giving the best purification result of waste cooking oil.

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