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Characterization of Spent Bleaching Earth as an Adsorbent Material for Dye Removal

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ABSTRACT

Initial research has been carried out to determine the potential of SBE as an adsorbent material through chemical and surface area characterization. Several analyses were performed, including oil content, BET, SEM-EDS, XRD, FTIR, and adsorption capacity. The oil content of the SBE samples were 0.05–0.09%, well below the standard (3%) of hazardous material classification according to the Indonesian government regulation. The chemical composition of SBE, measured by EDS, was dominated by Si and Al elements. XRD analysis revealed two 2-theta diffraction peaks indicated the presence of crystalline SiO_2 and Al_2O_3 phases. Additionally, the results of the FTIR test also showed the dominance of Si-O and Al-O-H functional groups. The SBE morphology, as observed in SEM image, exhibited irregular shape and porous surface covered by impurities. These results supported by the BET data which showed SBE surface area of 10.86 m^2g^{-1} and a mesopore volume of 2.49 $cm^3(STP)g^{-1}$. Batch adsorption study conducted using low and high range concentration of methylene blue produced a maximum adsorption capacity of 7.993 mg/g and 40.485 mg/g, respectively. The adsorption isotherm analysis showed that the adsorption mechanism was in accordance with the Langmuir isotherm model. Considering its chemical characteristic, SBE has met the criteria for adsorbent material. Nevertheless, the small surface area requires SBE to be activated prior to use.

Keywords: adsorbent, adsorption capacity, surface area, SBE.

INTRODUCTION

Bleaching earth (BE) is a natural material akin of active clay, commonly used to adsorb pigments of CPO. To produce 1 million tons of CPO, 0.5–1% BE which is equivalent to 34 thousand tons of Spent Bleaching Earth (SBE) were generated as solid waste. The increase of vegetable oil production within two years was accompanied by the increase of SBE solid waste from 184,162 tons in 2017 and reached 778,894 tons in 2019 [pslb3.menlhk.go.id]. According to the new Indonesian government regulation, the SBE wastes with less than 3% residual oil content are not classified as hazardous waste [PP No. 22 of 2021]. It opens the opportunities to manage the SBE waste at a lower cost, including reuse it for other purposes, which was not possible according to the old regulation [PP No. 101 of 2014].

The approach for SBE waste management should implement the 3R (Reduce, Reuse, Recycle) concept to minimize the rate of waste generation. Various efforts have been widely made by several parties to reuse SBE, such as for a filler in NPK fertilizer [Purba et al., 2019], substitute for cement [Sharom, 2016], substitute for briquette raw materials [Sabarina et al., 2010], and as landfill material [Utama, 2020]. Although many studies have been carried out to reuse SBE, the accumulation of wasted SBE at the final disposal site is still high in Indonesia, reaching more than 50% of the total production [pslb3.menlhk.go.id]. The wide variety of SBE reuse is expected to reduce this accumulation. Therefore, additional research is required to contribute to solving this problem.

Bleaching earth is one of the types of montmorillonite clay, which belongs to the smectite group with various chemical compositions with its volumes that can expand and shrink. Additionally, there are high negative charges on the mineral double layer structure. This type of smectite clay is able to accept and absorb metal ions and organic cations [Loh et al., 2015]. SBE is composed of aluminosilicate materials of SiO_2 and Al_2O_3 with the percentage of 60–80%, contains oil residues of about 20–40% and additional component of phosphoric acid (H_3PO_4). The aluminosilicate content enables SBE to trap particles on the surface so that it can adsorb dyes [Damayanti, 2019]. In this study, an analysis of the surface area, composition, and adsorption capacity of the SBE material was carried out to find out the characteristics of SBE as an adsorbent material in a more detailed manner, which would be useful to determine the next effective and efficient utilization step.

MATERIALS AND METHODS

Materials

This study used the SBE waste from one of the oil companies in Gresik Regency, East Java Province, Indonesia. The SBE from this company has undergone the process of oil extraction and high temperature combustion. Therefore, the material characterized in this study is classified as DSBE (De-oiled Spent Bleaching Earth).

Characterization

SBE was tested for residual oil content prior to being used. Surface analyses were performed to determine the physical and chemical characteristics, such as morphological and elemental content analysis by SEM-EDS (JEOL-JSM-6390), crystalline phase by XRD (Philips X-Pert Pro-MPD), and surface functional groups by FTIR (Bio-Rad FTS-3500). The surface area and porosity of the material were analyzed by BET (BEL JAPAN).

Adsorption capacity test

The adsorption capacity of SBE was tested by performing the batch adsorption test using methylene blue solution. The standard methylene blue solution was made with the variation concentration of 5, 10, 15, 20, 25, and 30 mg/L, with its absorbance measurement at the maximum wavelength of 665 nm. A total of 1-gram SBE was placed into 50 mL of methylene blue solution with of low concentrate variations of 30, 60, 90, 120, 150, 180 mg/L, and high concentrations of 200, 300, 400, 500, and 600 mg/L. An Erlenmeyer glass contained the mixture of methylene blue solution and SBE was then stirred with a magnetic stirrer at the speed of 400 rpm for 120 minutes. The adsorbent was separated from the solution by filtration using Whatman filter paper no. 42. The obtained filtrate was then diluted 5 times for the low concentration and 20 times for the high concentration. The concentration of methylene blue before and after adsorption was determined by UV-vis spectrophotometry.

Adsorption isotherm model: Langmuir and Freundlich

The adsorption mechanism of SBE is analyzed using Langmuir and Freundlich isotherm models. The Langmuir isotherm model was associated with adsorption process through the single layer formation process (monolayer). This model is based on the assumption of homogeneous distribution of adsorption site, constant adsorption energy, neglectable interactions between adsorbate molecules [El-Sayed, 2011]. The Langmuir linear isotherm model can be expressed by Eq.

$$\frac{Ce}{qe} = \frac{1}{Kl\,qm} + \frac{1}{qm}Ce \tag{1}$$

The Freundlich isotherm represents multilayer adsorption on the heterogeneous surface with the considerable interaction between the adsorbed molecule with the distribution of its non-uniform absorption energy between the surfaces [Sadaf & Bhatti, 2014]. The equation model can be defined by Eq. (2).

$$\log qe = \log Kf + \frac{1}{\mathrm{n}}\log Ce \tag{2}$$

where: *Ce* represents the equilibrium concentration of adsorbate (mg/L), qe is the amount of adsorbate which was absorbed by the adsorbent (mg/g), *Kl* is the ratio of adsorption and desorption rate (L/mg), *qm* is the maximum adsorption capacity (mg/g). *Kf* [mg/g (L/mg)n] is the Freundlich constant which is related to the adsorption capacity (referring to the quantity of adsorbate in the adsorbent) and n is the Freundlich constant which indicates the intensity of adsorption. The value of n gives an indication of how well the adsorption process is.

RESULTS AND DISCUSSION

Oil content

The value of residual oil content in SBE can reach above 30% [Wafti et al., 2011] and up to 40%. The residual oil content that is too high can inhibit the performance of adsorbent. The residual oil content contained in SBE can be determined through the extraction process. This research used SBE that has been undergoing de-oiling process through petroleum benzene in Soxhlet extraction method. This treatment aims to increase the SBE surface area by removing the impurities attached to the SBE surface. The test results showed that the oil content of SBE samples were within the range of 0.05–0.09%. Having oil content in this range, this SBE is excluded from hazardous material classification according to Indonesian Government which set the standard below 3%. Due to the tendency of the residual oil to oxidize and the autocatalytic property of SBE, the direct landfill of SBE at local locations as a traditional disposal route leads to spontaneous self-ignition of SBE and other environmental problems. In this context, several viable recycling strategies to alleviate the SBE disposal problems have been reported, such as the production of biofuels, bio-lubricants and biodiesel. The regeneration of SBE as an inexpensive water / waste water adsorbent through chemical, physical and thermal processes is viewed as an interesting topic [Merikhy et al., 2020].

Surface area and porosity

The BET theory is developed from Langmuir's theory because it can be applied to any layer (multilayer) in which, physically, gas molecules can be adsorbed onto an infinite layer of solids. The BET test aims to determine the surface area of the material, the pore distribution of the material, and the pore volume of the SBE. The BET test in this study resulted in the surface area of the SBE material which can be seen in Table 1.

Table 1. BET test result of SBE material

Parameter	Value	Unit
V_m (Volume mesopore)	2.4969	[cm^3(STP) g^{-1}]
$a_{s,BET}$ (Surface area)	10.868	[m^2g^{-1}]
Total pore volume (p/p_0= 0.315)	0.0056131	[cm^3g^{-1}]
Mean pore diameter	2.066	[nm]

On the basis of Table 1, the average pore diameter of SBE is 2.066 nm. The pore diameter of SBE is related to the ability of the pores to absorb the adsorbate from a particular fluid. This diameter affects the density and porosity of the SBE material. Larger pore diameter value is related to the smaller density value. The pore diameter surely affects the adsorption capacity of SBE, because more fluid can flow in the pores. Thus, the value of the pore volume is positively correlated to the adsorption ability of SBE.

On the basis of the results of BET test, the volume of SBE mesoporous value is 2.49 cm^3 (STP)g^{-1}. The greater value of the surface area of the SBE, the more pores will be in the unit area of SBE. From the test results, the surface area value ($a_{s,BET}$) is 10.86 m^2g^{-1}. Similar results have been reported by [Majid & Mat, 2017], wherein the obtained value of surface area was 19.4 cm^2g^{-1}. This value is relatively smaller compared to other adsorbent materials which able to have a surface area above 100 m^2g^{-1}. In another study, Activated Spent Bleaching Earth (ASBE) with the acid-heating combination method had a surface area of 100.38 m^2g^{-1} [Sabour & Shahi, 2018]. Similar result was also reported by Meziti & Boukerroui [2012] who produced an ASBE surface area of 153 m^2g^{-1}. Thus, SBE must be activated to increase the surface area to its maximum value to match or exceed the other adsorbents.

Surface morphology and elemental content

The SBE characteristics based on SEM image are used to see the surface morphology structure, grain size, structural defects, and pollution composition. The results of SEM image obtained using 1000x and 2000x magnification can be seen in Figure 1. Morphology of SBE appears to have an irregular surface, relatively spherical shape and has pores. The narrow pore space may indicate the presence of impurity compounds which filled some of the pore spaces in SBE. The SBE morphology is shaped like flakes and large spheres with irregular shapes that show the morphology of montmorillonite [Mana et al., 2007; Sabour et al., 2017].

The EDS test aims to determine the composition of the elements or the constituent elements of SBE material. The EDS analysis is based on the comparison of measurement results of X-ray intensity ratio, generated by the elements in the material sample with similar element from the

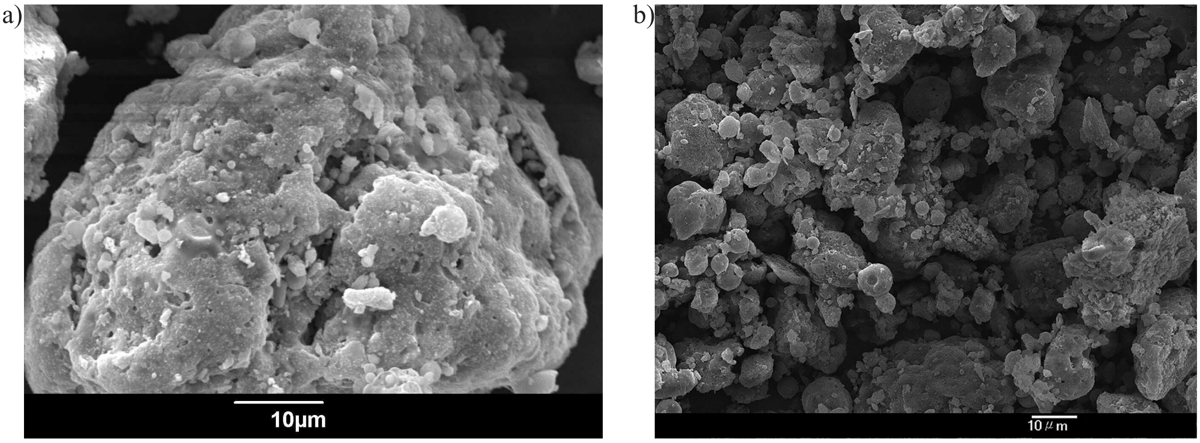

Figure 1. SEM image of SBE material under (a) 2000x, (b) 1000x magnification

standard sample. The X-rays characteristics generated from each element are proportional to the concentration of the element, the probability of X-ray production or ionization of the element, and the path length of the electrons. This characteristic is compared with the standard value to obtain the components of the SBE material. The results of the EDS spectrum are shown in Figures 2a and 2b.

Figure 2a shows that SBE component contains of the following substance, sequentially from the most to the least based on weight %: O (30.46%), Si (30.13%), Fe (11.94%), Al (9.03%), Zr (6.80%), Ca (4.40%), Mg (2.39%), Pt (2.23%), K (1.48%) and Ti (1.15%). Meanwhile, Figure 2b contains the following substance sequentially from the most to the least based on weight %: O

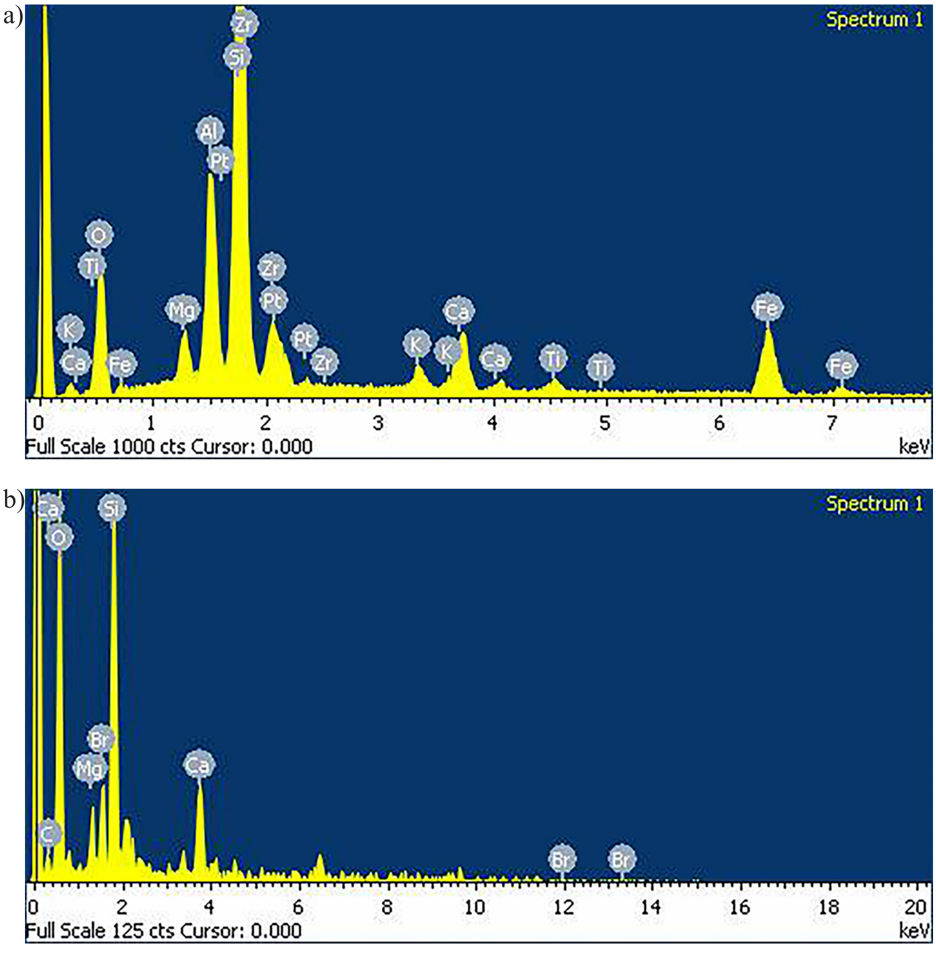

Figure 2. EDS Test Results for SBE Materials

(51.75%), Si (21.20%), Ca (9.53%), C (7.08%), Br (7.80%), and Mg (2.65%). Similar things is also found by Suryani et al., [2015] with O, Si, and Al as the dominant elements of SBE. The adsorption ability of SBE is caused by Al element on the surface of the trap particles so that it can adsorb the adsorbate and depends on the comparison of Si and Al elements [Damayanti, 2019].

The result of the EDS test shows that the SBE material is classified as Ca-montmorillonite and contains several impurities, such as Fe, Ca, Mg, Br, Zr, Ti, Pt, and K, which fill the surface of SBE [Merikhy et al., 2019]. The appearance of C element in SBE indicates that there is an organic compund and residual oil which left behind during bleaching process, but the amount is only 7.08%. Extraction process of residual oil has been done toward SBE material based on the residual oil test, showing a very small amount of 0.05–0.09%. However, the impure elements has fulfill around 30% of SBE surface, which caused the results of SBE surface area to small with the value of 10.86 m^2g^{-1}.

Cristallinity

The XRD analysis aims to determine the nature of amorphous and crystalline phases formed in SBE. The basic principle of XRD testing is to diffract light that passes through the crystal slit and comes from a radius with a wavelength equivalent to the distance between atoms of about 1 Angstrom. The results of the XRD test shows that: (1) the position of diffraction peak which can provide an overview of the lattice parameters, the distance between the fields, the crystal structure and the orientation of the unit cell; (2) the relative intensity of the diffraction peaks gives an idea of the atomic position in the unit cell; (3) the shape of the diffraction peaks gives an idea of the crystallite size and lattice imperfections. The results of XRD testing of the SBE materials can be seen in Figure 3.

On the basis of Figure 3, it can be seen that the peak diffraction shows the characteristic of mineral montmorillonite, quartz, and a little bit of minor crystal [Farahiyah et al., 2020; Hindryawati et al., 2019; Sabour et al., 2017]. The XRD test analysis shows that the peak diffraction of two 2-theta between 20° to 50° shows the appearance of montmorillonite ($Al_2O_3.4SiO_2.H_2O$) and quartz (SiO_2) minerals. The peak diffraction of two 2-theta shows the SiO_2 from the SBE sample which appeared on 20.8299°, 21.7561°, 25.4454°, 26.6214°, 36.5271°, 39.4329°, 62.7940°, and 67.8940°. On the basis of the mineralogy analysis, the SBE contains SiO_2 in both crystal phase and amorphous phase. The peak diffraction of two 2-theta indicates aluminum oxide (Al_2O_3) which appeared on 35.5537°, 43.2784°, 50,0869°, and 53.8783°. There are other minor crystals contained in SBE which is calcite, shown by the peak diffraction two 2-theta of 29.3915°.

Surface functional group

The FTIR spectroscopy method is an infrared spectroscopic method equipped with Fourier transform to analyze the spectrum results. Figure 4 shows the infrared spectrum from the FTIR spectroscope test of the SBE sample. The FTIR analysis of the SBE sample shows the structure of montmorillonite and the characteristic of residual oil in the

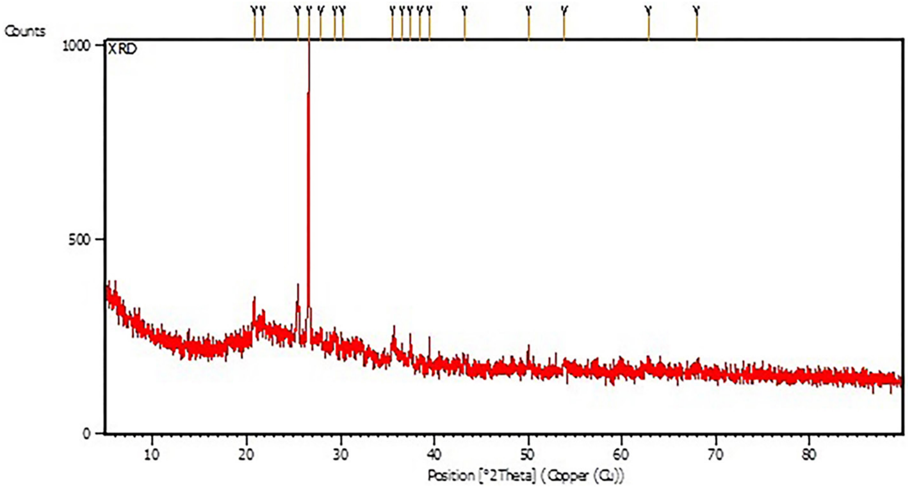

Figure 3. XRD test result of SBE material

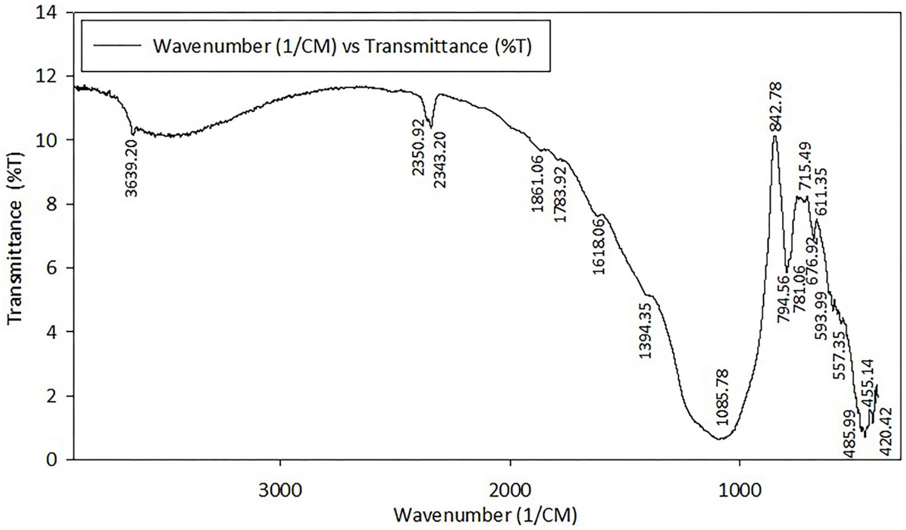

Figure 4. FTIR test result of SBE material

resulting spectrum range [Hindryawati et al., 2019; Merikhy et al., 2019; Sabour et al., 2017]. The spectrum adsorption near 3639 cm^{-1} shows the appearance of montmorillonite structure, which shows Al-O-H group strain. The group of Si-O can be seen in the spectrum adsorption of 1085 cm^{-1} (strain), spectrum of 751–794 cm^{-1} shows Si-O vibration with the quartz impurities, spectrum of 611–676 cm^{-1} shows Si-O vibration in kaolinite, spectrum of 557–593 cm^{-1} shows the changes of Si-O-Al structure, and spectrum of 420–485 cm^{-1} shows the vibration from Si-O or Si-O-Si group. Meanwhile, the characteristic of residual oil appears at the spectrum range of 1394–2350 cm^{-1}, there is a spectrum of 1394.067 cm^{-1} (CH_2 vibration) which appears in this range value, spectrum of 1618 cm^{-1} (O-H vibration), spectrum of 1783–1861 cm^{-1} (ester carbonyl strain), and spectrum of 2343–2350 cm^{-1} (it can be C-H strain or an alkene C=C strain structure). The whole spectrum range can be seen in Figure 4.

Adsorption capacity and isotherms

Figure 5a shows the methylene blue used for the experiment arranged in the order from low concentration (left) to high concentration (right), while Figure 5b shows the same solution after adsorption process. From these figures, it can be clearly observed that the lower initial concentration of methylene blue resulted in a clearer final solution. The most striking change was visually obvious at the concentration of 30 mg/L (Figure 5a and 5b on the far left), where the blue color completely disappeared after the adsorption process. Under this condition, the adsorption capacity reached 1.48 mg/g.

Model of isotherm adsorption is able to give the information about adsorption maximum capacity to evaluate adsorbent performance [Wang & Guo, 2020]. Figure 6 shows the Langmuir and Freundlich isotherm models as the calculation result of methylene blue adsorption. The mathematic equation model in Figure 6 shows that adsorption at low concentration was better fitted to the Langmuir model at Figure 6a Eq. (3) compared to the Freundlich model at Figure 6c Eq. (5). The same thing happened to the mathematical equation

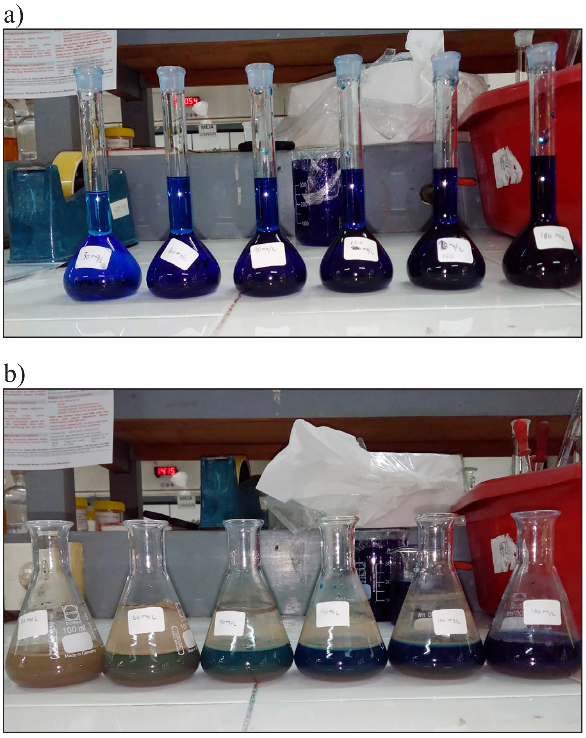

Figure 5. Adsorption test on methylene blue; a) before adsorption, b) after adsorption

model at high concentration was better fitted to the Langmuir model at Figure 6b Eq. (4) compared to the Freundlich model at Figure 6d Eq. (6).

$$\frac{1}{qe} = 0.2187\left(\frac{1}{Ce}\right) + 0.1251 \tag{3}$$

$$\frac{1}{qe} = 2.7635\left(\frac{1}{Ce}\right) + 0.0247 \tag{4}$$

$$\log qe \;=\; 0.4443\,\log qe + 0.4163 \tag{5}$$

$$\log qe \;=\; 0.621\,\log qe + 0.0311 \tag{6}$$

The coefficient correlation value of the Langmuir isotherm model (Eq. (3) and Eq. (4)) in the adsorption mechanism at low and high concentration of methylene blue are 0.9971 and 0.9938. Meanwhile, the coefficient correlation values of the Freundlich isotherm model (from Eq. (5) and Eq. (6)) are 0.9648 and 0.9863. The appropriate value of a good correlation (≥ 0.9) shows that the result test of the Langmuir and Freundlich isotherm models have given an excellent result. Therefore, the Langmuir and Freundlich isotherm models can be used in the adsorption mechanism of methylene blue with SBE adsorbent.

Although the difference is not significant, the mechanism of methylene blue adsorption by SBE is more in line with the Langmuir isotherm model compared to the Freundlich isotherm model, both in low and high concentration. The Freundlich isotherm model shows the presence of heterogeneous adsorption of more than one surface layer (multilayer) on the surface of the SBE pore space. However, the difference in binding energy for each layer in the adsorption process follows the Langmuir isotherm model. Therefore, the determination of the maximum adsorption power of SBE in the methylene blue absorption process was carried out using the Langmuir isotherm adsorption equation, which is the adsorption of a single layer (monolayer) of methylene blue on each surface of SBE with units of mg of adsorbed methylene blue/gram SBE. The Langmuir isotherm model shows the distribution of homogeneous adsorption site and constant adsorption energy. Thus, based on the calculation, the maximum capacity of SBE to adsorb methylene blue in low and high concentration is 7.993 mg/g and 40.485 mg/g, respectively.

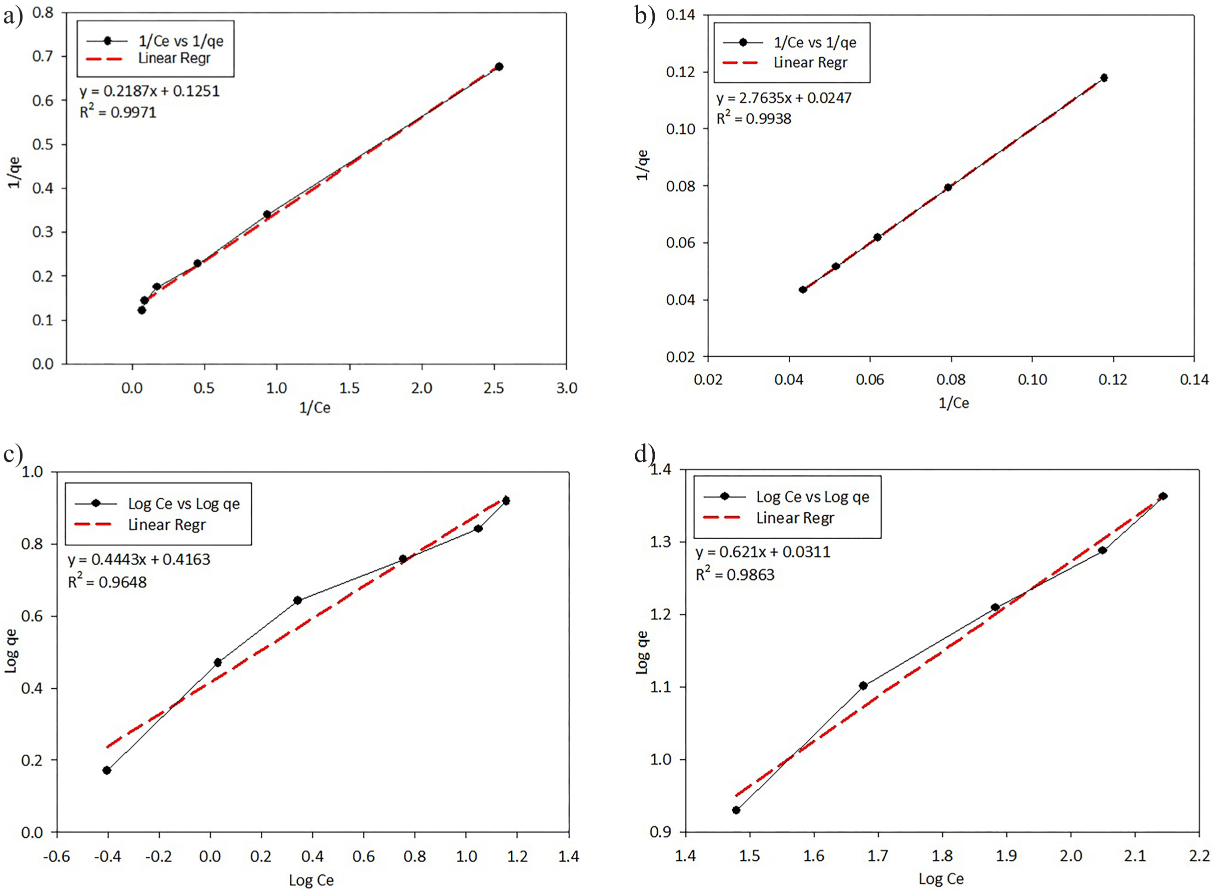

Figure 6. Methylene blue adsorption by SBE modeled by the Langmuir and Freundlich isotherm

CONCLUSIONS

On the basis of the characteristic test of SBE regarding both of surface morphology of the pore space and the composition of the chemical components, the results obtained indicate that the SBE has the potential to become an adsorbent material. The Si and Al elements dominated in the EDS analysis, and there is a dominance of SiO_2 and Al_2O_3 crystalline phase in a few peak diffractions as well as the domination of aluminosilicate functional group. However, SBE is classified as the material with a small surface area, which is 10.868 m^2/g based on the surface characteristic. The batch study using methylene blue shows the maximum adsorption capacity of 7.993 mg/g and 40.485 mg/g at low and high concentration with the adsorption mechanism following the Langmuir isotherm model.

This adsorption capacity can still be increased again through the SBE activation process with the chemical, physical, or combination chemical/physical method. In this method, the adsorption test will be investigated by considering the pH effect, reaction time effect, stirring speed effect, adsorbent mass effect and the effect of adsorbate concentration. This study aimed to improve the operational performance of SBE as an adsorbent. In the future, it is expected that SBE can be used as a raw material for adsorbent material as part of the efforts to utilize the SBE waste and simultaneously to overcome the pollution of the water environment by hazardous material adsorbate.

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