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16. Synthesis And Characterization Of Adsorbent From Solid Waste Of Ceramics Industry

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Synthesis And Characterization Of Adsorbent From Solid Waste Of Ceramics Industry

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Abstract. At present, the adsorbents generally use bentonite and activated carbon, but the availability of bentonite is less so that other alternatives are needed for substitution of bentonite as an adsorbent. One of the materials that have the potential as an adsorbent is the ceramic industry waste (ceramic clay). Ceramics clay containing feldspar which before being activated using sulfuric acid has a ratio of concentration between silica and alumina (silica/alumina) of 3.6, so a process is needed to increase the ratio of silica and alumina. One such process is by activating ceramic clay using sulfuric acid. The characterization and adsorption performance of adsorbent from ceramic clay were studied with various acid concentration. Ceramic clay powder was activated with sulfuric acid. The adsorbent particles were characterized by IR Spectra, X-Ray Diffraction, X-Ray Fluorescence, and BET. Various concentrations of sulfuric acid solutions for activation have a significant effect on the characteristics and performance adsorbent. This is indicated by XRD analysis where the characteristics of the adsorbent have feldspar ($Ca-KAl_2SiO_3$) content with the ratio between silica and alumina increasing around 4.3-4.8 with an increase in sulfuric acid concentration from 10-30% and adsorbents in the form of mesoporous crystalline structure. The characteristics of adsorbent with IR Spectra analysis showed that the presence of silicate alumina groups, so the products have the potential as a bleaching agent. The best surface area is produced at a concentration of 15 % sulfuric acid solution with one hour activation time which is equal to 117.44 m^2/g.

1. Introduction

The main products of the ceramics industry are glassware, for examples plate or cup. Characteristic of the ceramic depends on geology state of where it takes. The material of a cup or a plate could from many kinds of ingredient such as feldspar, ball clay, quartz, and kaolin. In this case, the referenced of the ceramics industry is using feldspar as the material. In chemically, feldspar was divided into four groups of minerals, potassium feldspar ($KAlSi3O8$), sodium feldspar ($NaAlSi_3O_8$), calcium feldspar ($CaAlSi_2O_8$) and barium feldspar ($BaAl_2Si_2O_8$) [1].

The main use of adsorbents is that more widely used for bleaching agent of crude palm oil is bentonite with the main component is alkaline, silica and alumina. Clay minerals are used extensively as adsorbents and their adsorption characteristics strongly depend on textural, structural, and morphological characteristics, in addition to their chemical composition and impurities [2]. A process to improve the adsorption properties of clay by means of treatment using chemicals is called the clay activation process [3]. Treatment using chemicals, one of which is by using acid treatment. Treatment using this acid is the most common method used for treating clay minerals that aimed to improve specific surface areas. Acid treatment using various types of acids including inorganic acids has often

been used, one of which is sulfuric acid. [4] in his research stated that giving acid to clay caused the replacement of cations and the cations could be exchanged for H^+ ions. The structure of clay (Al^{3+}, Mg^{2+}, and Fe^{3+}) during the activation process using acid, the structure will be replaced by H^+. This shows the high adsorption capacity [2].

Clay can be used as an adsorbent in the vegetable oil bleaching process which aims to reduce the content of various impurities present in the vegetable oil. The process can use naturally active or activated clays. Naturally active clays have a high specific surface area so that use as an adsorbent shows a high ability to adsorb impurities in the process of vegetable oil bleaching. The bleaching process using activated clay has a much higher activity compared to the natural active clay. This is because activated clay has a much higher specific surface area. The bleaching process is an adsorption process in refining oil and is the most critical stage because in the process the colored or colorless pigments owned by the oil can be removed so that the final product of the bleached oil is increasing both in terms of appearance, flavor, taste, and stability of the oil [5]. [6] and [7] stated that various materials that have the capacity as adsorbents and have been widely used in the bleaching process of vegetable oils are activated clay, bentonite, activated carbon, activated earth, kiselguhr or diatomaceous earth. The study of comparing bentonite and modification of bentonite composites used as adsorbents for wastewater treatment. The modification of composite bentonite had a high ability in removing certain inorganic contaminants in water [8, 9, 10]. [11] investigated the manufacture of Activated Carbon-Clay (ACC) through a carbonization process followed by K_2CO_3 activation. The resulting ACC will be used to purify or purify olive oil. The results showed that ACC had the potential for adsorption of dyes in olive oil. [12] have conducted research on the characterization and data of adsorbent isothermal for Cd^{2+} adsorption from aqueous solutions of sugarcane-bentonite bagasse mixture using HCl and NaOH. Based on FTIR analysis, clay has functional groups that have potential in the adsorption process.

The combination of various acid concentrations is needed in the activation of a raw material to obtain adsorbents with maximum surface area and can determine the optimal conditions of the activation process in order to obtain optimal bleaching capacity [13] and [14]. Solutions to produce adsorbents with low costs are needed because of economic constraints in producing adsorbents [15, 16, 17]. Based on these reasons, ceramic clay is chosen as the raw material for making adsorbents in the hope that it can be used as a profitable adsorbent.

2. Methodology

2.1 Materials
Ceramics clay is a solid waste of ceramics industry that obtained from Tangerang, Banten, Indonesia. The chemical H_2SO_4 obtained from Bratachem Surabaya, Indonesia.

Raw materials from large-scale industrial solid waste are reduced in size by grinding the waste so that the size of the material escapes from 100 mesh. The solid waste of the ceramic industry which has been formed in powder is then dried in an oven at a temperature of 110 $^\circ$ C. The solid waste of the ceramics industry as the dried adsorbent is then analyzed by using the XRF component to find out the composition before the next process is carried out.

2.2 Synthesis Process
The clay sample in big size that has been reduced into small pieces is activated with H_2SO_4 in various concentration of 10; 15; 20; 25 and 30% with the ratio between the weight of samples and volume of H_2SO_4 solution is 1:5 and heated at 120 $^\circ$ C in 90 minutes. And then, neutralized the adsorbent product until reached pH 4-5. Dried the adsorbent product in the oven at 100 $^\circ$ C. After that, reduced the size until 100 mesh.

2.3 Characterization
The adsorbent particle from solid waste of ceramics industry were analyzed using X-Ray Diffraction, IR Spectra of FTIR, and BET [18].

3. Result and Discussion

3.1 Effect of Sulfuric Acid Solutions Concentration on the Composition of Adsorbent Products

Effect of sulfuric acid solutions concentration on the composition of adsorbent products is shown in Figure 1.
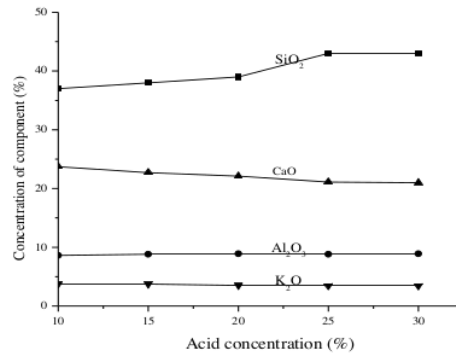

Figure 1. Effect of Sulfuric Acid Solutions Concentration On The Composition of Adsorbent Products

Figure 1 shows that the composition of the components in the adsorbent along with changes in acid concentration from 10 to 30% was as follows: SiO_2 tended to increase from 37 to 43%, Al_2O_3 quite stable in the range of 8.6 to 8.9 %, CaO slightly decreased from 23.7 to 21.0 % and K_2O stable in the range of 3.77 to 3.44%. The silica to alumina ratio is slightly increased from 4.3 to 4.8 with an increase in acid concentration from 10 to 30% for its activation.

3.2 Effect of Sulfuric Acid Solutions Concentration on the Characteristics of Adsorbent Products

The result of component analysis by X-Ray Fluorescence on ceramics clay showed that the solid waste of ceramics industry was contained silica (SiO_2) of 41.1 w%, alumina (Al_2O_3) of 12.1 w%, calcium oxide (CaO) of 24. 6 w%, and potassium oxide (K_2O) of 4.4 w%. Adsorbent material from solid waste of ceramicsindustry that would be tested for its adsorption ability of crude palm oil was first analyzed its structure using X-ray diffraction.
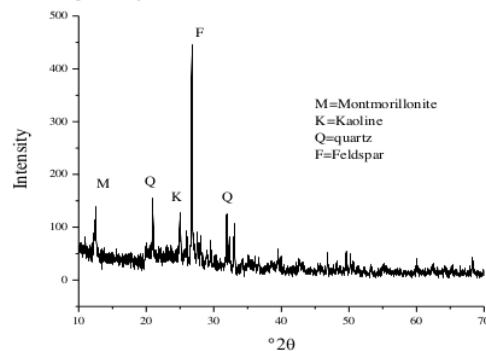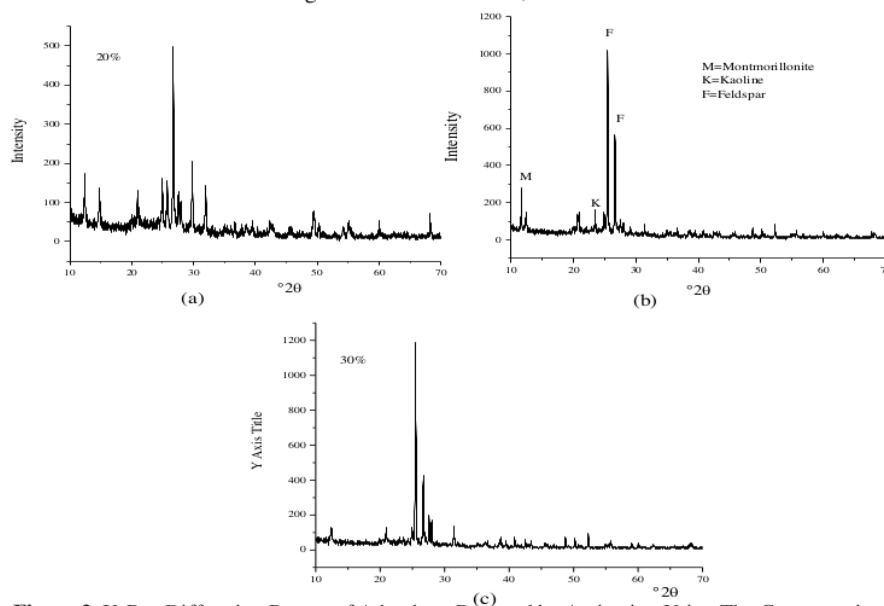

**Figure 2**. X-Ray Diffraction Pattern of Adsorbent Without Activation

Figure 2 shows the diffraction pattern of the adsorbent before being activated using sulfuric acid solutions. The result showed that at angle 15 °2Θ there was a sharp peak indicating as montmorillonite, at an angle 22, 27 and 33 °2Θ. [1] and [19] stated that indicating the existence of feldspar or quartz and around the angle 24 °2Θ indicating the presence of kaolin.

At a low angle position within 10 to 30 ° 2Θ indicates that the diffraction pattern with intense sharp peaks. The crystalline structure of the wall material that has uniform diameter pores in the mesoporous range is characterized by a single sharp peak pattern [20].

Figure 3 (a), (b), and (c) shows the diffraction pattern of adsorbent prepared by activation using sulfuric acid solutions in the range concentration of 20, 25 and 30 %.

Figure 3. X-Ray Diffraction Pattern of Adsorbent Prepared by Activation Using The Concentration of H_2SO_4 Solutions of (a) 20%, (b) 25%, and (c) 30%
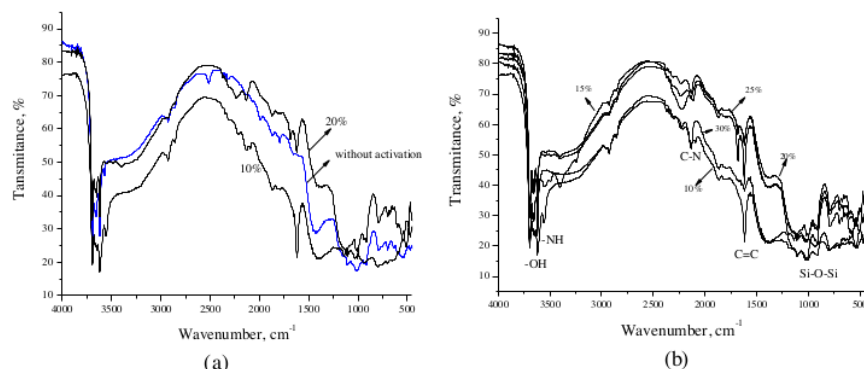
Figure 4. The IR Spectra of The Adsorbent: (a) Product by Acid Activation and Without Activation, (b) Prepared by Activation Using Sulfuric Acid Solutions Concentration of 10 – 30%

The IR spectra of adsorbent particles before activation and after activation is shown in Figure 4 (a). Interpretation of the infrared spectra of the adsorbent before activation using sulfuric acid (H_2SO_4) solutions as follows: alcohol (OH) groups appear on several peaks, namely wave numbers 3590-3650 cm^{-1} and 3200-3600 cm^{-1} which indicate the presence of an alcohol OH monomer group (phenol), at wavenumbers 3500-3650 cm^{-1} shows OH groups of carboxylic acids and at wavenumbers 2500-2700 cm^{-1} show the presence of OH carboxylic acid and hydrogen groups.

Figure 4 (b) shows that IR spectra for sulfuric acid concentrations of 10 and 30% besides the emergence of OH groups in wavenumbers as described previously can also be shown by the emergence of peaks in wave numbers 2850-2970 cm^{-1} which indicates the presence of CH alkane groups, while the appearance of peaks in wave numbers 2210- 2280 cm^{-1} indicates the presence of CN

nitrile groups. The peak at wave number 2100-2260 cm^{-1} indicates that there is a group C = C alkyne, and C = C alkene in wave number 1610-1680 cm^{-1}

3.3 Effect of Sulfuric Acid Solutions Concentration and Activation Time on the Surface Area of Adsorbent Products

Brunauer, Emmett, and Teller Surface Area Analysis (BET) were carried out to determine the surface area, average pore radius, and total volume pores. Increased Si/Al ratio has an influence on surface area and pore volume. The effect of sulfuric acid solutions concentration and activation time on the surface area of the adsorbent product is shown in Figure 5.
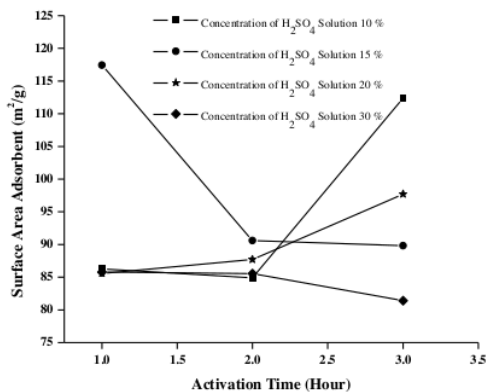
Figure 5. Effect of Sulfuric Acid Solutions Concentration and Activation Time On The Surface Area of The Adsorbent Products

Figure 5 shows that the concentration of sulfuric acid solution and activation time have an influence on the adsorbent products. The best surface area of the adsorbent was obtained at a concentration of 15 % sulfuric acid solution with one hour activation time which was 117.44 m^2/g. The second best surface area was shown at a concentration of 10 % sulfuric acid solutions with three hour activation time. This shows that at a concentration of 15 % sulfuric acid solutions with one hour activation time and a concentration of 10 % sulfuric acid solution with an activation time of three hours, the adsorbent pores are formed in increasing numbers so that the adsorbent surface area increases and the adsorption capacity increases. The results of this study are the same as those conducted by [18, 20, 21, 22] that acid activation will increase the surface area of the adsorbent.

4. Conclusion

Adsorbent from solid waste of ceramics industry (ceramic clay) has been production successfully. The XRD characterize for adsorbent showed the existence of Feldspar (Ca-KAl$_2$SiO$_3$) with the ratio of silica/alumina is about 4.3-4.8. Adsorbent products in the form of mesoporous crystalline structures. The characteristics of the adsorbent with IR Spectra analysis indicate that there is a silica alumina group, so the product has the potential as a substitute for the bentonite bleaching agent. The concentration of sulfuric acid solutions and activation time have an influence on the adsorbent product. The best surface area is produced at a concentration of 15% sulfuric acid solutions with one hour activation time which is equal to 117.44 m^2/g.

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