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Application of Kaolin Adsorbent on Fe(II) Metal Absorption in Water

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ABSTRACT

In this study, a comparison was made of the absorption of kaolin in reducing Fe(II) metal in water by modifying kaolin with anionic surfactants and also irradiating kaolin with ultrasonics. The aim of this research is to compare the optimization of metal uptake in absorbing Fe metal in water so that it can improve water quality in Ulee Pulo, Dewantara, Aceh Utara. Kaolin was ground to a size of 100 mesh, then activated using 1 N HCl, A1 adsorbent with pure kaolin, A2 adsorbent modified with anionic surfactants, A3 kaolin adsorbent using ultrasonication. The adsorption process was carried out using adsorbents A1, A2 and A3 by varying the sampling time of 30, 60 and 90 minutes. The concentration of the absorbed metal was measured using an Atomic Absorption Spectrophotometer (AAS), while the characteristics used Fourier Transform Infrared Sectroscopy (FTIR) and Scanning Electron Microscope (SEM). The most significant result was Fe absorption with removal efficiency reaching 81% within 60 minutes.

Keywords: Adsorption, kaolin, anionic surfactants, ultrasonic

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INTRODUCTION

Water is an important element needed by humans, but in a state that has been polluted water can cause problems for health. The presence of metals in water is often a problem for human health, especially heavy metals that have difficult to degrade, toxic, and accumulative properties (Alfanaar, 2017). Heavy metals are one of the sources of environmental pollution. Iron is an essential heavy metal that is a pollutant in water systems. Fe(II) in the human body is not biodegraded, so it can accumulate in the organs of the human body.

Regulation of the Minister of Health of the Republic of Indonesia 416/Menkes/Per/IX/1990 which regulates the requirements and supervision of clean water quality that shows a clean water has met health requirements. For ferrous metals has a quality standard of 1.0 mg / l. If the heavy metal content exceeds the quality standard, then the clean water does not meet the requirements and must be treated before being used for daily purposes, especially for consumption. Effective methods for removing Fe(II) metal ions in water are ion exchange, electrodialysis, and adsorption. Adsorption is one of the heavy metal removal methods that is often used because it is more effective, cheaper, and efficient (Wawan prasetyo et al, 2014).

Kaolin is one of the adsorbents that is often used in the adsorption process. Kaolin is abundant in nature, so it is cheap. (Halim, et al 2013) use pure kaolin for adsorption of lead, zinc and cadmium. From the results of the study, it turns out that the adsorption capacity of pure kaolin is still low, when compared to activated carbon, zeolite and bentonite (Salary, 2017).

Ultrasonic or sonication is an application that uses ultrasonic waves to move particles of a test material to accelerate the dissolution process of a material with the principle of intermolecular reaction breaking, which later the particles of the test material are split to nano-size (Garcia-Vaquero et al, 2017). According to Santi (2014), the capacity of adsorbents that have been activated and irradiated by ultrasonic waves increases up to two times, because ultrasonic irradiation can magnify the surface area of the adsorbent specifically.

According to research by Alfian Putra, et al (2021) In the study of kaolin-based adsorbent processing using ultasonic technology to increase its absorption to reduce metals in water by modifying kaolin with anionic surfactants and ultrasonically irradiated. The goal is to optimize the absorption of metals that are more specific in absorbing Fe and Mg metals in water using modified kaolin. Kaolin is mashed up to 100 mesh and mixed with anioinic surfactant as much as 45%, from the total weight of the adsorbent which is 200 grams. The adsorption process is carried out using surfactant modified kaolin and without surfactant modification by varying the irradiation time of 5, 10, 15, 30, 50 and 80 minutes at 55 °C. Metal concentration is measured using Atomic Absorption Spectrophotometers (AAS) and adsorption kinetics calculations are carried out. The results showed that the highest removal efficiency occurred in kaolin modified with anionic surfactant. The most significant result is that for Fe the

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removal efficiency reached 72.81% at 5 minutes irradiation time, while for Mg the removal efficiency reached 65.1% at 5 minutes irradiation time.

According to research by Alfian Putra, et al (2022) using the Response Surface Methodology (RSM) design in looking at optimizing kaolin adsorbents in absorbing Mn metal by modifying using three types of surfactants (anionic surfactants, cationic surfactants, and amfolitic surfactants) to increase the ability of kaolin to absorb Mn metal. The kaolin activation process uses two activators, namely acids and bases. The ratio between kaolin and surfactant was varied by 45%, 60% and 75% of kaolin's weight with stirring speeds of 50 rpm, 70 rpm, 90 rpm and contact times of 30 minutes, 60 minutes, 90 minutes. The initial concentration of Mn metal is 10 ppm (artificial waste). Results of optimization of Mn metal absorption process using kaolin Surfactant adsorbents showed that the optimum response of absorption efficiency was obtained at 60% anionic surfactant treatment, stirring speed of 90 rpm and contact time of 90 minutes with the efficiency of reducing Mn metal concentration reaching 79.86%. In this study, variations were carried out on the treatment of activated kaolin, modified kaolin and ultrasonic kaolin in absorbing Fe (II) metal in water with variations in surfactant and irradiation time applied in the filter housing.

1.2 Kaolin

Kaolin is a mineral found in sedimentary rocks known as clay rocks. Kaolin is a rock mass composed of high-quality clay material with a chemical composition of hydrous aluminum silicate (2H2O. Al2O3.2SiO2) and is white, gray-white, yellow-orange, gray or reddish. This kaolin contains grains that are very fine, soft and less plastic when mixed with water. Kaolin is a rock that belongs to the clay group (clay), white or yellowish in color. The chemical formula of pure kaolin is hydrated aluminum silicate (Al2O3.2SiO2.2H2O), but it is often formulated as Al2Si2O5(OH)4.

Minerals belonging to the kaolin group are kaolinate, natrite, and haloysite with the main minerals being kaolinate, often oxides such as Fe2O3, TiO2, CaO, MgO, K2O and Na2O present in kaolin as impurities. The composition of pure kaolin is SiO2 46.54%, Al2O3 39.5% and H2O 13.96% (Shaban, 2017).

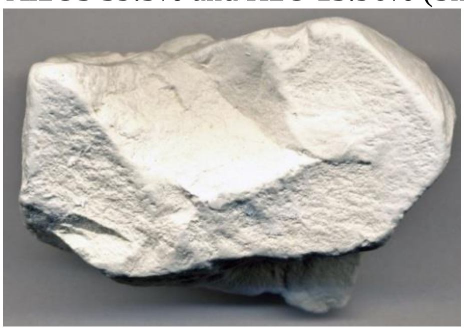

Figure 1. Kaolin stone

According to Yavuz et al, 2003, in Maftuh Hanani, 2019 states that kaolin can be used as an adsorbent. This is because the kaolin structure in the form of layers causes kaolin to absorb various materials between the layers of kaolin structures

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such as heavy metals, dyes, gases, and many more. (Utari T 1994, in Maftuh Hanafi 2019) also states that kaolin is an amorphous and porous solid and has inert, neutral, and large surface area properties so that it has large adsorption properties.

1.3 Characterizations of Kaolin

In general, kaolin is white or slightly whitish, hardness 2-2.5, plastic when mixed with water, and specific gravity between 2.60-2.63. The properties of kaolin will be greatly influenced by the mineral composition of clay soil present in kaolin.

The most important component in kaolin is kaolinite, which is because it is very well used as an adsorbent due to its high adsorption capacity, large specific surface area and good physical and chemical stability.

Table 2. 1 Chemical Compositions of Kaolin

No	Compositions	Weight (%)
1	SiO_2	71,20
2	Al_2O_3	13,36
3	Fe_2O_3	2,00
4	TiO_2	0,26
5	CaO	0,15
6	MgO	3,55
7	K_2O	0,27
8	Na_2O	0,51
9	LOI	8,70

Source: Rahayu (2015)

1.4 Adsorbtion

Adsorption is one of the most efficient and inexpensive techniques for removing harmful metal ions from liquid waste. This process is flexible in design and operation making it possible to produce high absorption quality. Furthermore, since adsorption is reversible in some cases, adsorbents can be regenerated through desorption (Burakov, 2018).

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Adsorption is generally defined as the accumulation of a certain number of molecules, ions or atoms that occurs at the boundary between two phases. Adsorption concerns the accumulation or concentration of the adsorbate substance on the adsorbent and in this case can occur on a two-phase interface. The absorbing phase is called adsorbent and the absorbed phase is called adsorbate.

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Most adsorbents are materials that have pores because they take place mainly on pore walls or certain locations within the adsorbent (Burakov, 2018).

The adsorption process takes place if a solid surface and gas and liquid molecules are in contact with these molecules, then there are cohesive forces including hydrostatic forces and hydrogen bonding forces acting between molecules of all materials. Unbalanced forces at the phase boundary cause changes in molecular concentration at the solid/fluid interface.

Porous solids that suck (adsorption) and release (desorption) a fluid are called adsorbents. Molecules that are sucked in but do not accumulate to the surface of the adsorbent are called adsorptive, while those that accumulate are called adsorbates.

METHOD

2.1 Tools and Materials
2.1.1 Tools used
This research was conducted in the laboratory of the Department of Chemical Engineering at Lhokseumawe State Polytechnic.

2.1.2 Materials used:
1. Kaolin
2. Alkil Benzene Sulfonate (ABS)
3. HCl 1 N Solution
4. Aquadest
5. Tap water

2.2 Preparation of raw materials
2.2.1 Kaolin Activation
1. Kaolin is heated using an oven with a temperature of 105 for 3 hours
2. After cooling, mixed with HCl 1.0 N and allowed to stand for 1 hour.
3. Then washed with aquadest until neutral (pH=7)
4. Next in the oven at a temperature of 105 for 3 hours.
5. Activated kaolin

2.2.2 Modification of Kaolin with Surfactants
1. Activated kaolin solids are mixed with surfactant solution (alkyl benzene sulfonate surfactant) with variations in the ratio of 25%,35% and 45% of the total weight of kaolin.
2. The mixture is stirred and allowed to stand for 2 hours.
3. After that the precipitate is filtered and washed with aquades until neutral (pH = 7).
4. Dried at room temperature
5. Modified kaolin ready for use

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2.2.3 Ultrasonic Kaolin

1. Activated kaolin is inserted into the erlenmeyer and covered with aluminum foil Masukkan Erlenmeyer kedalam Ultrasonic device, set irradiation time 20, 30 minutes with temperature 550C.
2. Kaolin is ready for use.

2.2.4 Adsorption Process

1. Prepare a set of Housing Filter tools and assemble them until they are connected between the pipe, filter housing and sample water container.
2. Weigh each adsorbent 800 gr.
3. Insert the adsorbent into each filter housing.
4. Prepare a 3-liter water sample into a bucket.
5. Flow the sample water until it passes through the adsorbent inside the filter housing.
6. Take water samples in every minute to 30,60 and 90 minutes.
7. Testing of water that has gone through the adsorption process

RESULTS AND DISCUSSION

The presence of metals in water is often a problem for human health. Especially heavy metals that have difficult-to-degrade, toxic, and accumulative properties. Therefore the use of kaolin modified adsorbents with anionic surfactants and ultrasonic irradiation is used to improve the absorption efficiency of Fe (II) metals contained in water.

Adsorbent Characteristics

- **<u>Fourier Transform Infrared (FTIR) test results</u>**

FTIR analysis aims to see the characteristics of adsobens. As seen in the image below.

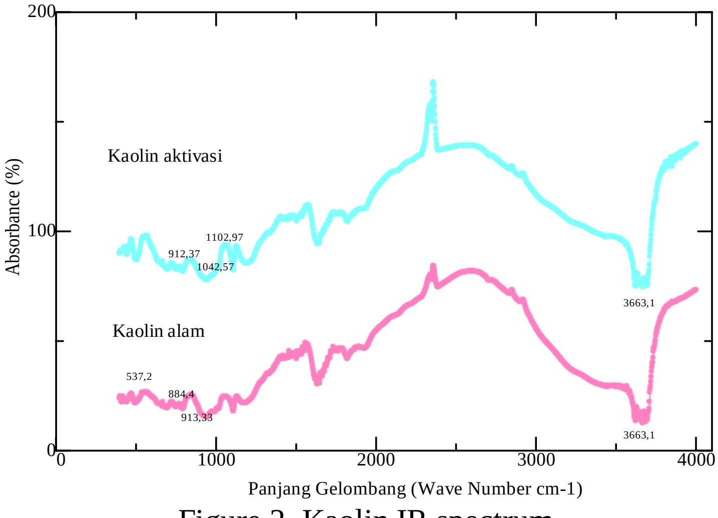

Figure 2. Kaolin IR spectrum

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Changes in the structure of Si-O-Al ulur kaolin alam were shown in the loss of peaks in the O-H vibrational region and Si-O deformation, namely at wavenumbers 537.2 cm-1 and 884.4 cm-1 and increasing the area of the peak spectrum at wavenumbers 913.33 cm-1. In kaolin activation there is a loss of peak at wavenumber 1042.57 cm-1 which is a Si-O strain vibrational region and an increase in the area of spectrum peaks at wavenumber 1102.97 cm-1. This is supported by the appearance of O-H deformation vibrations with the number 912.37 cm-1. The difference in vibration in the two spectrums, because kaolin is activated with HCl, the pore has opened so that the vibration changes. But the last peak of both spectra shows the same vibration, which is octahedral O-H with a wavelength of 3663.1 cm-1.

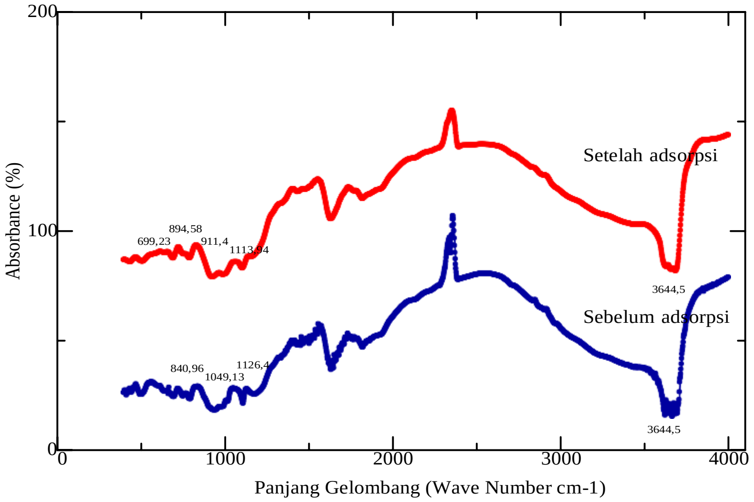

Figure 3. Modified kaolin IR Spectrum (A2 Adsorbent)

From figure 3 The results of IR kaolin spectra of anionic surfactant modification before adsorption showed that there was a change in vibrational O-H deformation (840.96 cm-1) to Si-O strain at wavelengths of 1049.13 cm-1 and 1126.4 cm-1. In the results of anionic surfactant spectra after Fe metal adsorption, the Si-O deformation vibration (699.23 cm-1) changed to the O-H deformation vibration with wavelengths of 894.58 cm-1 and 911.4 cm-1. Increasing spectral area at wavenumber 1113.94 cm-1 indicates the appearance of Si-O strain vibrations. This change in spectrum occurs due to the absorption process of Fe metal in kaolin modified anionic surfactant. The last peak in both spectrums, shows the presence of octahedral O-H vibrations with a value of 3644.5 cm-1.

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- **Scanning Electron Miscroscope (SEM) Test Results**

SEM readings on samples 1 and 2 of the tellurite glass:

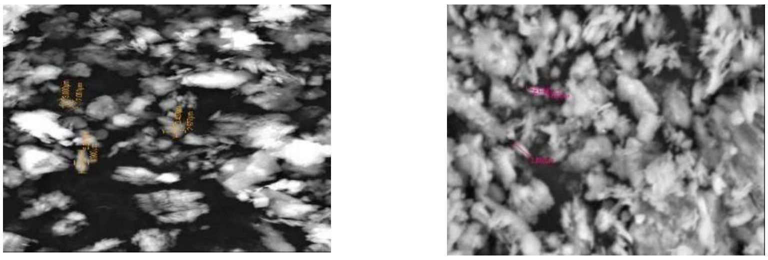

Figure 4. SEM kaolin test results of surfactant modification (A2) before (a) and after (b) absorption in Fe Metal waste (A2).

From figure 4. is SEM observation data on modified kaolin materials with surfactants before the Fe metal adsorption process shows that the surface of organooolin still has many large pores and empty cavities (figure 3.3 a). Meanwhile, in the observation of kaolin surfactant modification after the Fe metal adsorption process (figure 3.3 b) it was seen that there were cavities that had been filled and squeezed together and had smaller pores. This is due to the absorption process of Fe metal so that the cavities and pores formed shrink.

3.3 AAS (Atomic Adsorption spectrophotometers) Test Results

Types of Adsorbents	Contact Time (minutes)	Initial Concentration of Fe (ppm)	Fe concentration after adsorption (mg/L)
A1	30		41,024
	60	100	41,036
	90		40,056
A2	30		81,024
	60	100	81,056
	90		80,036
A3	30		74,816
	60	100	74,778
	90		73,708

Based on the table above, A1 adsorbent, which is an adsorbent modified with anionic surfactant, has more absorption efficiency, which is 81% within 60 minutes.

CONCLUSION

Based on the results of the study, it can be concluded that:

1. Penyisihan logam Fe dalam air dipengaruhi oleh jenis adsorben

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2. A2 adsorbent, which is a kaolin adsorbent modified with surfactant, has a good absorption ability, which is 81%.
3. Contact time affects absorption, the longer the contact time will be lower, it is likely that desorption process occurs.

SUGGESTION

Researchers suggest conducting research on the occurrence of desorption at times exceeding 80 minutes. In further Silage Making research, it is best to test the ash content and crude fiber content so that the results obtained are more optimal to determine the quality of the silage.

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