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Modification of Montmorillonite with Chitosan as Mercury Metal Adsorbent

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Abstract. Modification of montmorillonite with chitosan as an adsorbent of mercury (Hg) metal has been carried out. This study aims to determine the characteristics of the adsorbent, the optimum adsorption capacity of Hg metal, and its adsorption isotherm equation. The optimum conditions for Hg metal adsorption are determined by varying the contact time and pH of the solution. Variations in adsorbate concentration are carried out to determine the adsorption isotherm. Characterization of the adsorbent using FTIR shows the presence of -OH, Si-O, Al-O, C-C, and N-H groups. The optimum conditions for mercury metal adsorption by montmorillonite modified with chitosan occur at a contact time of 80 minutes, and a pH of 4. The adsorption process of mercury metal ions using montmorillonite adsorbent modified with chitosan satisfies both isotherm models, namely the Langmuir and Freundlich isotherms with R^2 values of 0.971 and 0.967 respectively, and a maximum adsorption capacity of 13.513 mg/g. The regeneration test showed that the adsorbent can be reused for the absorption of mercury metal ions.

Keywords: montmorillonite, chitosan, adsorption, Hg metal, adsorption isotherm

INTRODUCTION

Mercury is one of the metals widely used in industrial and laboratory activities. This is because mercury metal (Hg) has liquid properties at room temperature with high specific gravity and electrical conductivity. Mercury contained in waste in public waters is converted by the activity of microorganisms into methyl mercury components (CH_3-Hg) which have toxic properties and strong binding power in addition to their high solubility, especially in the bodies of aquatic animals. The hazards of mercury metal pollution can also inhibit enzyme activity, nerve damage, loss of walking coordination, slurred speech, blindness, and hearing loss [1].

These researchers have found various ways to overcome heavy metal pollution, especially aquatic environmental pollution. The mitigation has been done using methods including chemical precipitation and adsorption [2], photocatalytic degradation [3], ion exchange [4], metal resistance [5], and electrochemistry [6]. Research on Hg metal adsorption is generally found in sulfide compounds or mineral complexes consisting of oxygen, silica, and sulfur.

Research on adsorption has also been widely carried out using different types of adsorbents including zeolite, activated carbon, rice husks, bentonite, plant waste, and others. Other adsorption research uses chitosan-modified zeolite adsorbents to see the absorption of color on heavy metals [7]. This study will use montmorillonite from the isolation of North Aceh bentonite and then bound with chitosan as an adsorbent of Hg metal. Chitosan is a natural polymer derived from chitin that can be used as an adsorbent because it contains hydroxyl (OH) and amino (NH_2) groups that function as metal binding sites. Montmorillonite has good adsorption properties because it is easy to expand, has exchangeable cations, and has a fairly large surface area. These properties make montmorillonite very suitable for use as an adsorbent [8]. Montmorillonite in this study came from Teupin Reusep Village, Muara Batu District, North Aceh.

Mercury is widely used in various fields such as medicine, agriculture, and industry, this causes a lot of waste to be produced. Mercury metal in this waste is known to be very dangerous for humans and other living things, so the waste needs to be treated before being discharged into the environment. One way is to adsorb mercury using chitosan-modified montmorillonite as an adsorbent.

Research on the application of montmorillonite as an adsorbent for heavy metals such as Cu, Pb, Zn, and several other metals has been conducted, and the results are less than optimal. This study will utilize montmorillonite modified with chitosan for the adsorption of Hg metal, with the expectation that montmorillonite can absorb Hg metal with a higher capacity. This study was conducted by varying the pH of the solution, temperature, the effect of contact time, and adsorbate concentration. Characterization of bentonite and montmorillonite was carried out using XRF, SEM, FT-IR, and XRD, while to determine the adsorption capacity of montmorillonite adsorbent against Hg metal AAS was used.

METHODOLOGY

Evaluation of The Montmorillonite Percentage Contained in North Aceh Bentonite

Natural bentonite from North Aceh was heated at a temperature of 200°C for 2 hours, and then CH_3Br in alcohol (density = 2.10 g/mL) was added, shaken, and centrifuged. Montmorillonite will float and other minerals will precipitate. Montmorillonite is separated and dried until the temperature reaches 200°C for 2 hours. The percentage of montmorillonite obtained is calculated using the Meyer method using the following equation.

$$\text{Percentage of montmorillonite} = \frac{\text{Dry Weight of Montmorillonite}}{\text{Dry Weight of Bentonite}} \times 100$$

Isolation of North Aceh Bentonite into Montmorillonite (MMT)

The bentonite sample (1 kg) was sieved with a 150 mesh sieve, then dried in an oven at a temperature of 105°C until dry (4 hours), and then stored in a desiccator. Furthermore, fractionation was carried out to obtain pure montmorillonite. The bentonite fractionation method was carried out by sedimentation, namely by dissolving bentonite in distilled water and using ultrasonic waves for 15 minutes with a power of 750 Watts. The sedimentation result was montmorillonite, which was characterized using FT-IR, XRD, and SEM.

Modification of montmorillonite using chitosan

The chitosan 0.35 g was dissolved in 20 ml of 2% of acetic acid and stirred using a magnetic stirrer for 2 hours, then added with montmorillonite with variations in mass of 0.01, 0.02, 0.03, 0.04, 0.05, 0.06, and 0.07 g, respectively, and stirred for 2 hours using a magnetic. Furthermore, 3 M NaOH solution was dropped into the mixture until montmorillonite was formed, bound to chitosan, filtered and washed with distilled water until the pH was neutral, and dried in an oven for 12 hours. The montmorillonite adsorbent modified with chitosan was then used to adsorb mercury metal ions. The adsorption capacity test was carried out at variations in contact time, pH, and initial mercury concentration, and then the absorbance was measured using AAS. The adsorbent with optimum adsorption capacity will be characterized using FTIR, XRD, and SEM, and used as the adsorbent whose adsorption isotherm will be studied.

Adsorption of mercury metal ions with contact time variations

The adsorbents were weighed as much as 0.1 g each and put into an Erlenmeyer containing 10 mL of 30 ppm HgCl2 standard solution. The solution was stirred using a shaker at a constant speed of 135 rpm and varied at contact times of 50, 60, 70, 80, and 90 minutes, respectively. The adsorbents were separated using a magnetic stirrer, and 1 mL of the filtrate was taken. The filtrate was then diluted into a 200 mL measuring flask using distilled water, and the absorbance was determined by AAS [9].

Effect of pH System on Mercury adsorption

The montmorillonite modified chitosan of 0.1 g each were put into Erlenmeyer containing 10 mL of 30 ppm $HgCl_2$ solution, then the pH of the adsorbate solution was adjusted to pH 2, 3, 4, 5, 6, 7, 8, and 9 respectively using NaOH, 0.1 M HCl, 0.1 M HCl, 0.5 M HCl, and concentrated. The solution was then stirred using a Shaker at a constant speed of 135 rpm at optimum contact time. The adsorbent was separated and the filtrate was taken, then diluted with distilled water in a 200 mL measuring flask, and the absorbance was determined by AAS [9].

Adsorption of mercury metal ions with concentration variations

Adsorbent as much as 0.1 g each was put into an Erlenmeyer flask containing 10 mL of $HgCl_2$ solution and then the adsorbate concentration was varied at 40, 45, 50, and 60 ppm respectively. The solution was stirred using a Shaker at a constant speed of 135 rpm at an optimum contact time and the optimum pH. The adsorbent was separated and the filtrate was taken, then diluted into a 200 mL measuring flask using distilled water. Furthermore, the absorbance was determined using AAS [9]. These data are used to determine the adsorption isotherm equation.

Regeneration Process

The montmorillonite adsorbent used for adsorption is washed with distilled water until pH 7 and filtered. Then, the adsorbent is dried at room temperature. The dried adsorbent is then reused to adsorb Hg metal at optimum adsorption conditions. The measurement results were measured using AAS [9].

RESULTS AND DISCUSSION

Characterization of North Aceh Montmorillonite Using XRF

The XRF results showed that the composition of the North Aceh bentonite are SiO2, AlO3, Fe2O3, CaO, MgO, Na2O, and K2O, with the composition as in Table 1.

Table 1. Chemical composition of North Aceh bentonite

Compounds	Composition of Weight (%)
SiO_2	62,31 - 72,29
Al_2O_3	14,08 - 15,61
Fe_2O_3	1,94 - 10,15
K_2O	0,02 - 0,91
MgO	0,84 - 1,15
Na_2O	0,48 - 1,19
CaO	0,08 - 0,77

Based on Table 1, it is known that the main components of North Aceh bentonite are SiO_2 and Al_2O_3. In general, bentonite consists of Ca-bentonite and Na-bentonite. Na-bentonite has a relatively high Na ion content compared to Ca and Mg ions, has a greater swelling power, is more soluble when added to water, and will form a good suspension compared to Ca-bentonite [10]. Bentonite from Teupin Reusep, North Aceh has a Na content (0.48 - 1.19%), this percentage is greater than Ca (0.08 - 0.77%). These findings are consistent with earlier research[11], that bentonite from Teupin Reusep

Village, North Aceh is included in Na-bentonite. Identification of Na-bentonite can also be seen based on its color, which is silver (gray). The Na and Ca content of a bentonite occurs based on its formation process. Widhiati (2010) states that bentonite formation can occur based on weathering, hydrothermal, transformation, and sedimentation processes. Bentonite from Teupin Reusep Village is produced from the weathering process of volcanic spices. The weathering process occurs due to the reaction between H^+ ions in groundwater and silicate compounds. H^+ ions from carbonic acid are due to the weathering of organic compounds in the soil. In general, Na-bentonite is formed from the weathering process that occurs in the area, the bentonite weathering process is caused by several factors, namely the composition of rock minerals, the chemical composition of water, and the flow properties of rocks. Other factors include climate conditions, various types of rocks, relief, and plants on rocks [12].

Characterization using FT-IR

Figure 1.a shows FTIR spectra with a distinct adsorption band linked with montmorillonite. The absorption bands that occur in the wave number regions of 3624.25 cm^{-1} and 3624.25 cm^{-1} are respectively characteristic of the OH group located in the octahedral layer and the vibration of the OH group in the octahedral layer bound to the Al^{3+} cation. Absorption in the regions of 3418.97 cm^{-1} and 1627.03 cm^{-1}, these absorptions are respectively absorptions of the stretching and bending vibrations of the OH of the H_2O molecule. According to Imoto (2015), the stretching and bending vibrations of the OH group of the H_2O molecule appear in the wave number regions of 3448 cm-1 and 1635 cm^{-1}. The absorption at 1019.42 cm^{-1} indicates the Si-O stretching vibration and the absorption at 916.23 cm^{-1} is the OH bending vibration of Al_2OH [13]. The absorption peaks at 1033 cm^{-1}, 552.63 cm^{-1}, and 552.63 cm^{-1} indicate the absorption of Si-O stretching vibration, Si-O-Al bending vibration, and Si-O-Si stretching vibration, respectively. Montmorillonite has three characteristic sharp absorptions at around 455 cm^{-1}, 520 cm^{-1}, and 1045 cm^{-1} which are the absorption of Si-O bending vibration, Al-O stretching vibration, and Si-O stretching vibration, respectively [14].

Based on Figure 1. b. it is known that the FT-IR spectrum of montmorillonite modified with chitosan does not show significant differences when compared to the FT-IR spectrum of montmorillonite. The difference only occurs in the shift in wave numbers at several peaks, namely at wave numbers 1627.03 cm^{-1}, 1632.81 cm^{-1}, and 3418.97 cm^{-1}, which respectively shift to 3428.62 cm^{-1}, 3624.25 cm^{-1}, and 3626.33 cm^{-1}. Another difference is the presence of a new peak in the FT-IR spectrum of montmorillonite modified with chitosan, namely the peak at wave number 917.23 cm^{-1} indicating the presence of stretching vibrations of C-C. Another peak appears at wave number 3626.33 cm^{-1} indicating the presence of stretching vibrations of N-H. These two functional groups are functional groups of the chitosan structure, which qualitatively shows that montmorillonite has been modified with chitosan.

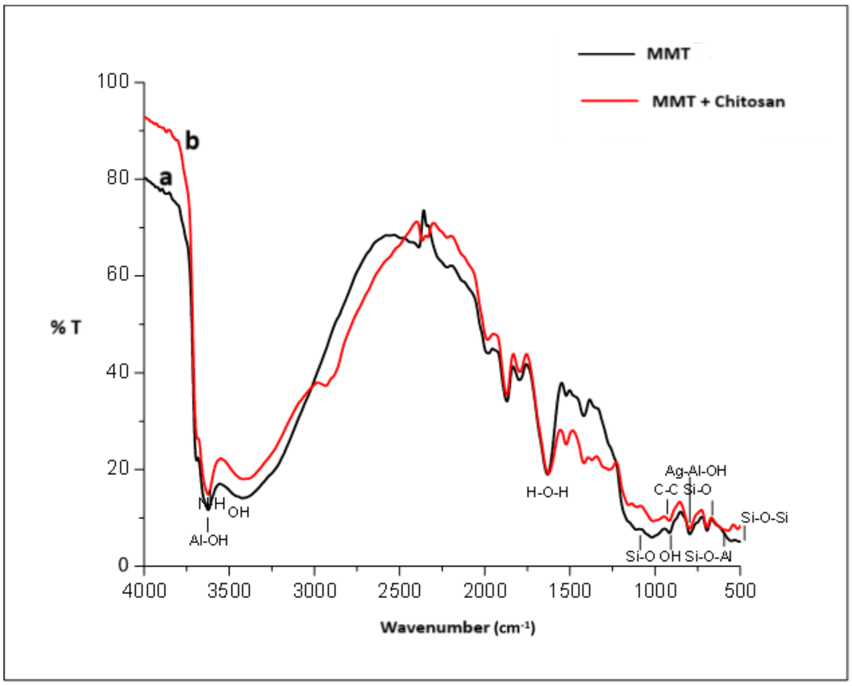

Figure 1. FT-IR spectrum of a) montmorillonite, b) modification of montmorillonite with chitosan.

Characterization using XRD

The results of XRD characterisation on a montmorillonite sample revealed the characteristic peaks of montmorillonite minerals. These peaks appear at 2θ angles, namely 5.6^o, 18.87^o, 23.60^o, and 35.20^o. The XRD spectrum of montmorillonite modified with chitosan has 2θ peaks at 20.80^o, 23.96^o, 26.59^o, and 36.25^o. The 2θ angle in montmorillonite modified with chitosan has shifted, this can be explained by whether the interaction of montmorillonite with chitosan occurs in the interlayer or out layer of montmorillonite the distance between the planes in montmorillonite is different from the distance between the planes in the modification of montmorillonite with chitosan, then there is a possibility of montmorillonite in the chitosan interlayer [15].

Characterization using SEM

The SEM characterization results of montmorillonite (Figure 2. a) show that the surface structure of montmorillonite is layered and porous on the surface of montmorillonite [16]. Meanwhile, the SEM characterization results of montmorillonite modified with chitosan (Figure 2. b) show that the surface is no longer rough and no longer shows pores, this is suspected that montmorillonite has been modified with chitosan [17].

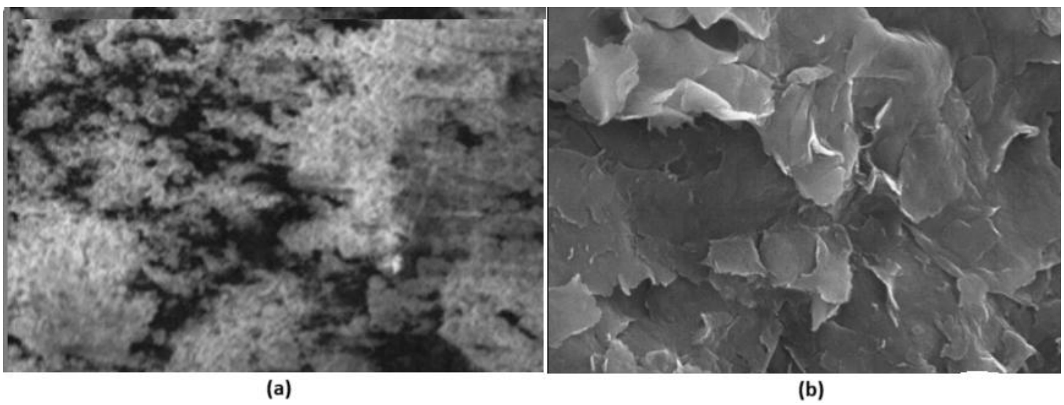

Figure 2. SEM Images of a) montmorillonite and b) montmorillonite modified with chitosan

Adsorption Test

Adsorption test of montmorillonite modified with chitosan

The adsorption test was carried out to determine the optimum absorption conditions and capacity of the montmorillonite adsorbent modified with chitosan for Hg metal. The test variables include varying the contact time (50, 60, 70, 80, and 90 minutes), and varying pH (2, 3, 4, 5, 6, 7, 8, and 9) [18].

Determination of optimum contact time

The concentration of mercury metal ions used was 30 ppm and pH 5. The test was based on research conducted by Dubey et al (2016) where the adsorption test on mercury metal ions using chitosan produced an optimum pH of 5. The results of the absorption capacity can be seen in Figure 3 below.

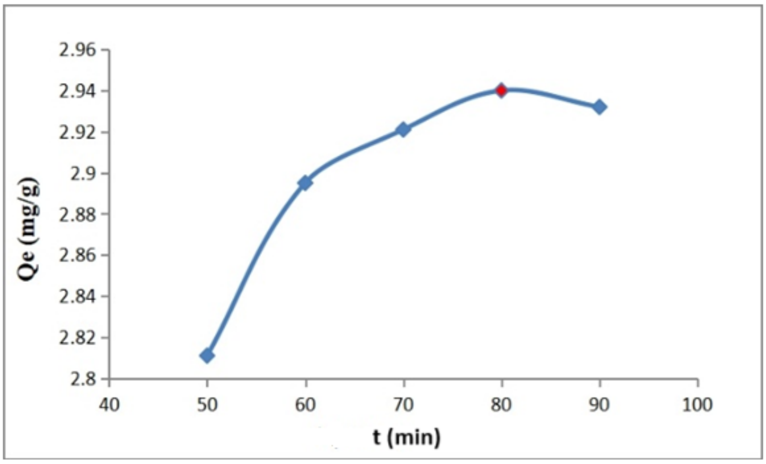

Figure 3. Relationship between time and adsorption capacity of mercury metal ions.

Figure 3 shows that there has been an increase in the absorption of mercury metal ions with increasing contact time of up to 80 minutes. Absorption began to decrease at the 90th minute. The increase in the absorption of mercury metal ions is caused by the large number of empty active sites on the surface of the montmorillonite adsorbent modified with chitosan. Increasing the contact time between mercury metal ions and the modification of montmorillonite with chitosan causes Hg ions to enter the pores of montmorillonite and also interact with chitosan decreasing adsorption capacity of the montmorillonite modified chitosan

The absorption of Hg metal ions at the 80^{th} minute was 2.940 mg/g, while at the 90^{th} minute, the absorption of Hg metal ions decreased to 2.932 mg/g, this is thought to be due to the desorption and equilibrium processes. The desorption process causes metal ions to be released from the surface of the adsorbent so that the adsorption capacity for mercury metal ions decreases [19]. The optimum contact time obtained in this test was 80 minutes.

Determination of optimum pH

The adsorption capacity of an adsorbent in the adsorption process can be affected by pH (Monier et al., 2014). The absorption of mercury metal ions using montmorillonite modified with chitosan was tested at pH variations. The composition of the pH variations tested was at pH 2, 3, 4, 5, 6, 7, 8, and 9 and was carried out at an optimum contact time of 80 minutes with an adsorbate concentration of 30 ppm. This pH adjustment was carried out using 0.1 M NaOH solution, 0.1 M HCl, and 0.5 M HCl.

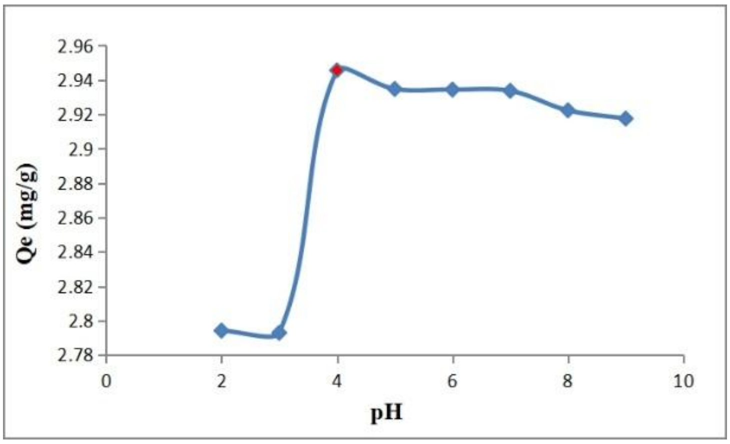

Figure 4. Relationship between pH and Qe (adsorption capacity) of mercury metal ions.

Figure 4 shows the optimum adsorption capacity for Hg metal adsorption using montmorillonite adsorbent modified with chitosan, which occurs at pH 4 (optimum pH). The adsorption capacity of Hg metal using this adsorbent will decrease at pH 1 to 3, this pH will cause protonation of H+ ions in the solution so that during the adsorption process there will be competition between mercury metal and H+ ions in the solution. Solutions with very acidic pH can also damage the adsorbent structure, so Hg metal adsorption does not occur optimally. In addition, at a very acidic pH, the functional groups in chitosan will also be protonated so that the electrostatic interaction between chitosan and mercury metal will decrease, all of which will impact the adsorption capacity.

The adsorption capacity will increase at pH 4, at this pH the adsorbent will swell which causes the active side of the adsorbent to be exposed, making it easier for Hg metal adsorption to occur. At a more alkaline pH, namely pH 5 to 9, chitosan will experience deprotonation which causes chitosan to shrink physically so that its active side is closed. In alkaline conditions, it will also produce hydroxide groups in solution which are more effective ligands than active groups in chitosan, causing Hg metal ions to be more interested in binding hydroxides and will result in a precipitation process. Both of these problems will cause the adsorption capacity to decrease slightly.

Adsorption Isotherm

The adsorption properties and interactions between the adsorbent and the Hg heavy metal ions that occur can be determined by testing them against the linear equations of the Langmuir adsorption isotherm and the Freundlich adsorption isotherm (Monier et al., 2014). The adsorption isotherm measurements were obtained by conducting an adsorption test using an adsorbent of montmorillonite modified with chitosan at various concentrations of mercury metal ions, namely 40 ppm, 45 ppm, 50 ppm, 55 ppm, and 60 ppm, and were carried out at an optimum contact time of 80 minutes and an optimum pH of 4.

The purpose of studying Langmuir adsorption isotherm is to understand monolayer adsorption on the surface and calculate the maximum adsorption capacity of the adsorbent. The Langmuir isotherm curve is made by plotting the value of 1/Qe against 1/Ce. From the Langmuir isotherm curve, a straight line equation is obtained, namely y = 0.061x + 0.074 with linear regression (R2) showing a value of 0.971. The values of x and y on the curve are obtained using the following equation 1:

$$\frac{1}{Qe} = \frac{1}{Qm} + \left(\frac{1}{bQm}\right)\left(\frac{1}{Ce}\right) \quad \text{............1)}$$

Qe is the amount of adsorbed metal ions (mg/g), Ce is the equilibrium concentration of metal ions (mg/L), Qm is the maximum amount of metal ions per unit weight of adsorbent (mg/g) and b is the adsorption equilibrium constant. The Qm value produced on the Langmuir isotherm is 13.513 mg/g. The maximum adsorption capacity (Qm) and constant (b) are obtained from the slope and intercept of the plots 1/Qe and 1/Ce. In the Langmuir isotherm, it is assumed that the adsorption that occurs is the formation of a monolayer layer and all sides and surfaces are homogeneous [20]. The Langmuir adsorption isotherm is often used to describe the absorption of solutes from liquid solutions. The Langmuir isotherm curve can be seen in Figure 5.

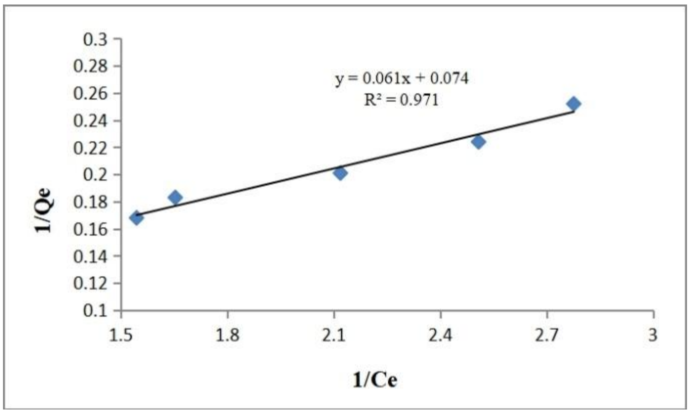

Figure 5. Langmuir isotherm for the adsorption of Hg metal ions using montmorillonite adsorbent modified with chitosan

Figure 6. shows the value of the straight line equation in the Freundlich isotherm, namely $y = 0.618x + 2.032$ and $R^2 = 0.967$. The curve below is obtained from the following equation 2:

$$\text{Ln Qe} = \text{Ln Kf} + (1/n)\ \text{Ln Ce} \quad \ldots\ldots\ldots\ldots\ldots..2)$$

The Freundlich isotherm curve in Figure 6. is made by plotting Ln Qe against Ln Ce.

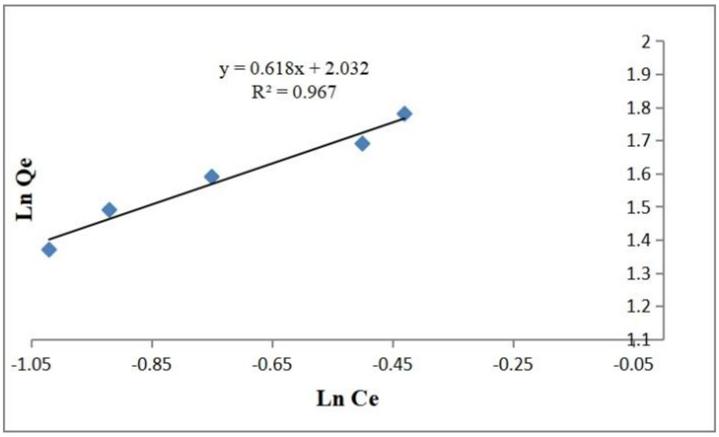

Figure 6. Freundlich isotherm for the adsorption of Hg metal ions using adsorbent of montmorillonite modified with chitosan

The adsorption of mercury metal ions by the Freundlich isotherm indicates that adsorption on the surface of the adsorbent occurs on the active sides which are heterogeneous [21]. The absorption potential can be seen from the functional groups found in the modification of montmorillonite with chitosan. The amino and hydroxyl groups play an important role in the adsorption process. Therefore, based on the Freundlich adsorption isotherm, it is assumed that these groups have different absorption potentials.

The relative ability of adsorbent montmorillonite modified with chitosan in adsorbing mercury metal ions can be determined using the Kf value. The greater the Kf value, the greater the adsorbent's ability to adsorb. The strength of the interaction between the modified montmorillonite adsorbent and chitosan in adsorbing mercury metal ions can be seen from the 1/n value which shows that the smaller the value, the stronger the interaction between the two [21]. The Kf value in this study was 0.733 mg/g and the 1/n value was 1.618. The n value determines the strength of the interaction between the adsorbent and the adsorbate. Physical adsorption can be determined if $n < 1$, while if $n > 1$ then adsorption occurs chemically [22]. In this study, the n value = 1.618 was obtained. These results indicate that the adsorption process of mercury metal ions using chitosan-modified montmorillonite adsorbent occurs chemically. Chemical adsorption occurs when chemical bonds are formed between solutes in solution and molecules in the medium. Comparison of Langmuir and Freundlich isotherm data from mercury metal ion adsorption can be seen in Table 4.2.

Table 4.2. Comparison of Langmuir and Freundlich isotherm adsorption data

Adsorbent	Langmuir isotherm			Freundlich isotherm		
	Qm (mg/g)	B	R^2	Kf	n	R^2
Montmorillonite modified with chitosan.	13,513	1,213	0,971	0,733	1,618	0,967

Description: Qm (mg/g) (maximum adsorption capacity); b (Langmuir constant); R^2 (regression coefficient); Kf (Freundlich constant) and n (adsorption number)
Determination of mercury metal ion adsorption pattern by comparing the linear regression coefficient (R^2) value of the adsorption isotherm curve. Table 4.2. shows that the R^2 = 0.971 for the Langmuir adsorption isotherm and R^2 = 0.967 for the Freundlich isotherm. Based on the R^2 value, it shows that the mercury metal ion adsorption process using an adsorbent of montmorillonite modified with chitosan satisfies both isotherm models, namely the Langmuir isotherm and the Freundlich isotherm.

Adsorbent Regeneration

The regeneration process is carried out on the modification of montmorillonite with chitosan, where the adsorbent regeneration process is carried out to re-produce an adsorbent that has been used in the adsorption process. This regeneration process begins with the desorption process of mercury metal ions contained in the adsorbent. Desorption is the release of ions or molecules that have bonded with active groups on the adsorbent. Desorption is carried out by immersing the adsorbent in a 0.001 M HNO_3 acid solution. After that, the adsorbent is separated from the solution and washed until neutral using distilled water. It was then dried and reused for the adsorption process.

Figure 7. is the result of the regeneration test of montmorillonite modified with chitosan, it can be seen that the adsorption capacity of the adsorbent decreased after several regenerations. In the first regeneration, montmorillonite modified with chitosan produced high adsorption capacity. However, in the next regeneration, the adsorption capacity of the adsorbent decreased. It can be concluded that montmorillonite adsorbent modified with chitosan can be reused as an adsorbent repeatedly has strong mechanical properties and is resistant to acids.

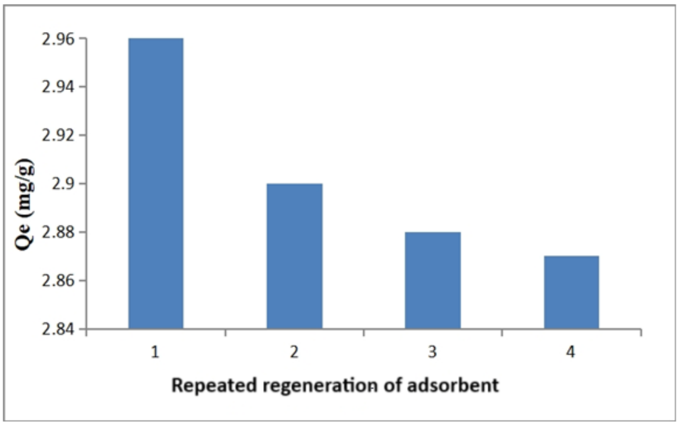

Figure 7. Adsorption capacity of the regeneration process of montmorillonite modified chitosan.

CONCLUSION

According to the study's findings, 75% (wt/wt) of the bentonite from North Aceh was isolated as montmorillonite. Based on FTIR spectra, the montmorillonite was successfully modified with chitosan and used to remove mercury from aquatic systems; however, the ideal pH of 4 and contact time of 80 minutes were found to be the most effective conditions for mercury metal adsorption. With a maximum adsorption capacity of 13.513 mg/g and R2 values of 0.971 and 0.967, respectively, the Montmorillonite adsorbent modified with chitosan adsorbent meets both isotherm models in the adsorption process of mercury metal ions. The regeneration test demonstrated that the adsorbent may be utilized again to absorb ions of mercury metal"

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