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Analysis of the Desorption Ability of Pb^{2+} and Cu^{2+} Metals of Silica and Silica-Chitosane Adsorbents

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Abstract

Studies have been conducted on the analysis of the desorption ability of Pb (II) and Cu (II) metals from silica and silica-chitosan adsorbents. This study aims to determine the desorption ability of Pb^{2+} and Cu^{2+} metals with variations in HCl concentrations using silica adsorbents and to determine the desorption ability of Pb^{2+} and Cu^{2+} metals with variations in HCl concentrations using silica-chitosan adsorbents. Improving desorption efficiency is crucial for the sustainable recycling of adsorbents in wastewater treatment processes. as the comparison of silica and silica-chitosan adsorbents for Pb^{2+} and Cu^{2+} desorption. The parameters optimized in this study were concentration variations, namely 0.005 M, 0.01 M, and 0.02 M. The results of the study showed that silica adsorbents, Pb (II) metal desorption was best obtained at an HCl concentration of 0.02 M, which was 10.11%. Meanwhile, in silica adsorbents for Cu (II) metals, the best desorption was also obtained at an HCl concentration of 0.02 M, which is 0.0753%. In the silica-chitosan adsorbent, the best Pb (II) metal desorption was obtained at an HCl concentration of 0.01 M, which is 0.0298%, while the best desorption of Cu (II) metal was obtained at an HCl concentration of 0.01 M, which was 12.0684%.

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INTRODUCTION

The development of the industry in Indonesia is currently experiencing very rapid progress with the advancement of the technological era. Industry can be interpreted as an economic activity that manages raw materials or semi-finished products into more valuable goods (TH et al., 2023). Metal pollution usually does not stand alone but can be carried by environmental components namely water, soil, and air. Environmental components that are polluted by inorganic compounds, therefore they may contain various heavy metals such as Cr, Zn, Pb, Cd, Fe and so on (Wardalia, 2016). The metals in question include Pb and Cu metals.

Pb (Lead) is a heavy metal that has the highest affinity for sulfur and attacks its bonds in enzymes. As a heavy metal, Pb is classified as a harmful pollutant. Pb is in water in the form of $Pb(OH)_2$. Copper metal (Cu) is a heavy metal that is toxic and can poison the human body and damage the environment (Naat et al., 2021). One way to treat heavy metal waste is by adsorption. Adsorption was chosen because it is a relatively simple method and can use adsorbents to adsorb metal ions. The selection of adsorbents in the adsorption process must be in accordance with the character and state of the substance being adsorbed (Ali et al., 2020). One of the industrial wastes that can be used as adsorbents is nickel slag.

Nickel slag is a solid waste produced by the metal smelting industry that is in the form of lumps resembling low-quality metal due to mixing with other materials that are difficult to separate. The amount of nickel slag is piling up day by day, because each refining process of one ton of nickel products produces fifty tons of solid waste. If allowed to accumulate, it will have an impact on the environment (Tanjung et al., 2022). One of the uses of nickel slag is as a silica adsorbent. Nickel slag can be used as an adsorbent due to the high levels of silica contained in nickel slag. Moreover, nickel slag has a fairly good composition to utilize. Approximately 70% of the chemical composition of nickel slag consists of 41.47% Silica, 30.44% Ferric Oxide and 2.58% Alumina (Juvelyn, et al., 2012 in Sartifa et al., 2022).

Silica is a porous solid, this porous structure is related to the surface area, the smaller the size of the silica pores resulting in a larger surface area so that the adsorption ability increases. In addition, silica has unique properties that other inorganic compounds do not have, such as inert properties, good adsorption and ion exchange properties, easily modified with certain chemical compounds (Hardyanti et al., 2017). Silica has several weaknesses, including the type of active site only in the form of silanol (Si-OH) and siloxane (Si-O-Si) groups. This group has low acidity properties and also has oxygen as a weak donor atom. However, the presence of silanol (Si-OH) and siloxane (Si-O-Si) groups is also beneficial because it allows modifications to be made in order to improve properties and for the expansion of utilization such as structural, porosity, and surface area regulation (Putra et al., 2022).

Modifications are made by adding organic compounds such as chitosan. Chitosan is a chitin derivative made through the chitin distillation process using strong alkaline compounds, which can bind metal ions with a fairly high capacity (Lestari et al., 2024). Silica from nickel slag modified with chitosan can produce an adsorbent that increases the adsorption power of metal ions. Chitosan is also an abundant natural polymer and is widely used as an adsorbent. Silica increases the porosity of chitosan, so that with the use of chitosan-silica as an adsorbent, it is expected to obtain better Pb2+ and Cu2+ adsorption results (Mulyasuryani et al., 2013). Silica- chitosan is used as an adsorbent in the adsorption process but its use has a disadvantage because the oxygen atoms in the silanol group, which is the main functional group of silica gel relatively speaking, are less able to bind to heavy metal ions. This weakness can be overcome through the process of desorption.

Desorption is the process of releasing both ions and molecules that are attached to or absorbed by the adsorbent. This process is used in the regeneration of an adsorbent so that the adsorbent can be reused when it is saturated. Regeneration can be carried out through desorption so that the recovery of metals that have been set aside and the reuse of adsorbents can be carried out. Desorption is carried out by contacting an adsorbent that has been used with a desorptive agent or solution. The desorption agent or solution used can be acidic, alkaline and neutral solutions (Zustriani, 2019). Heavy metal desorption needs to be done to release back heavy metals that have been absorbed by the adsorbent (e.g., soil or biomass) so that the adsorbent can be regenerated and reused. Factors that affect the adsorption and desorption processes are contact time, solvent concentration, solute concentration, temperature, acid concentration (pH) and salinity (Maslukah et al., 2020).

Based on the description above, Previous studies have focused on the adsorption of Pb^{2+} and Cu^{2+}, but less attention has been given to comparing the desorption effectiveness of these metals from different adsorbents a study was conducted to analyze the desorption ability of Pb^{2+} and Cu^{2+} metals using silica and silica-chitosan adsorbents and to evaluate the effectiveness of desorption of the two heavy metals and to compare the performance of pure silica with silica-chitosan as an adsorbent.

METHOD

Manufacture of Silica Adsorbents

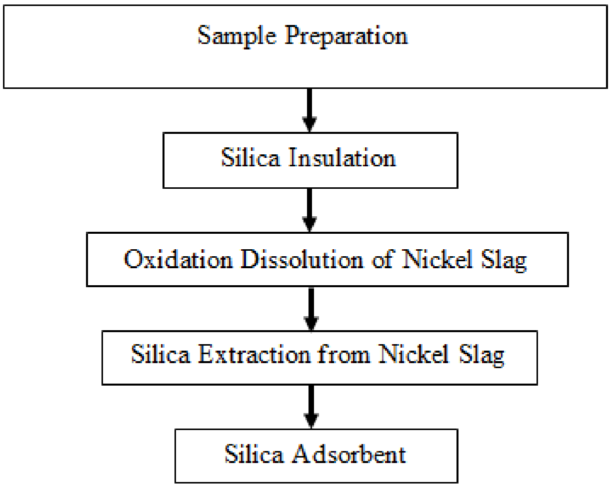

Figure 1. Manufacture of silica adsorbents

Sample Preparation

Weighing 40 grams of silica slag nickel that has been sifted with a size of 200 mesh. Put in a 500 mL beaker. Added H_2SO_4 (10%, w/v) 1:5 nickel slag weight (g) to the volume of H_2SO_4 (mL) stirred using a magnetic stirrer for 2 hours. After that, it is filtered using ordinary filter paper and dried in the oven at 105oC until completely dry.

Oxidation Solution Metal Slag Nickel Using H_2SO^4 10%

Weighing 40 grams of silica slag nickel that has been sifted with a size of 200 mesh. Put in a 500 mL beaker. Added H_2SO_4 (10%, w/v) 1:5 nickel slag weight (g) to the volume of H_2SO_4 (mL) stirred using a magnetic stirrer for 2 hours. After that, it is filtered using ordinary filter paper and dried in the oven at 105^oC until completely dry.

Silica Extraction from Nickel Slag Using 12% NaOH Solution

Nickel slag that has been washed with H2SO4 is put into a 600 mL beaker. A 300 mL of 12% NaOH solution is added, then heated to 85oC for 90 minutes while stirring using a magnetic stirr. After that, it is filtered using filter paper, then the filtrate is neutralized with 0.5 M sulfuric acid until there is a gel deposit and left for 18 hours. The gel deposits formed are then filtered, then washed using hot aquades as much as 1000 mL, dried in an oven with a temperature of 105oC for 5 hours and filtered and calculated the percentage of silica yield obtained (La Harimu et al., 2019).

Manufacture of Silica-Chitosan Adsorbents

The composition of silica and chitosan is regulated by a ratio of 97.5% silica used 2.5% chitosan. Chitosan is dissolved with 80 mL of 2% acetic acid. After dissolving, silica is added and then stirred overnight using a magnetic stirr. The silica-chitosan mixture is neutralized with 30 mL of NaOH 1 M and left for 1 hour. The sediment was decanted and soaked in 40 mL of glutaraldehyde 0.5 % (v/v) for 24 hours. The results obtained are filtered with Whatman paper no.1, heated to 105oC. then cooled (Mulyasuryani et al., 2013).

Adsorption of Pb^{2+} and Cu^{2+} Metals Using Silica and Silica-Chitosan Adsorbents

The adsorption process is carried out by adding silica adsorbents to each beaker and setting the pH to pH 5. Next, stirring is carried out for 15 minutes using a magnetic stirr. Then filtering with filter paper and reading of the absorption of Pb (II) and Cu (II) atoms using SSA. Then it is also repeated using silica-chitosan adsorbents. (Harimu et al., 2019)

Determination of Wavelength

The determination of the maximum wavelength is carried out by detecting adsorbtion in the standard solution Pb in the wavelength range of 249.2 – 324.7 nm.

Calibration Curve Creation

calibration curves for AAS measurements pinched as much as 0.5; 1; 1,5; 2; and 2.5 mL of Pb solution of 50 ppm and put each in a 50 mL measuring flask and added aquades to the tera limit so that a concentration of 0.5 is obtained; 1; 1,5; 2; and 2.5 ppm. A 5 mL standard solution of each concentration was sprayed into 5 test tubes and added 0.1 mL of concentrated HNO_3, after which the absorbance was measured at the maximum wavelength.

Depsorption of Pb^{2+} and Cu^{2+} Metals Using Acid Concentration (HCl)

Prepared 3 100 mL beakers are put in HCl solution into a beaker with concentrations of 0.005 M, 0.01 M, and 0.02 M as much as 50 mL, then 0.01 grams of silica-chitosan adsorbent are added and stirred using magnetic stirrer for 1 hour. Then filtering with filter paper and reading of the absorption of Pb2+ and Cu2+ atoms using SSA (La Harimu et al., 2019).

Depsorption of Pb^{2+} and Cu^{2+} Metals Using Acid Concentration (HCl)

3 100 mL beakers were prepared, HCl solution was put into a beaker with concentrations of 0.005 M, 0.01 M, and 0.02 M, then 50 mL was added to the adsorbent silica adsorbent which had been adsorbed as much as 0.01 grams and stirred using a magnetic stirrer for 1 hour. Then filtering with filter paper and reading the absorption of Pb2+ and Cu2+ atoms using SSA (Harimu et al., 2019). The depsorption ability test can be seen in the following diagram.

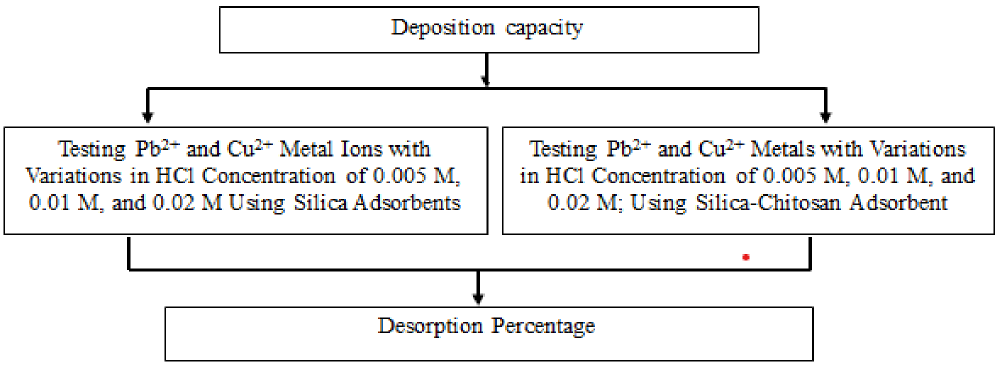

Figure 2. Desorption Ability Test

Data Analysis Techniques

Data from the measurement of the absorbance of Cu2+ and Pb2+ metal ions using AAS, was used to calculate the final concentration of Cu2+ and Pb2+ metals after adsorption (Ce) using regression equations.

$y = ax + b$

where: y = adsorbansi; x = Ce = final concentration of the metals Cu2+ and Pb2+; a = slope; b = intersep, furthermore, the price of Cc obtained is used to calculate the percentage of adsorbed metals calculated by the equation:

$$\% \text{ Adsorbed} = \frac{C0-Ce}{C0} \times 100\%$$

The percentage of depsorbed metal ions in silica and silica-chitosan adsorbents is calculated using equations;

$$\text{Depsorption Efficiency} = \frac{\text{Concetration after desorpstion}(\frac{mg}{L})}{\text{Concetration before desorpstion}(\frac{m}{g})}$$

RESULTS AND DISCUSSION

Silica Insulation From Nickel Slag

The neutralizing reaction of sulfuric acid to sodium silicate is as follows:

$$Na_2SiO_3(aq) + H_2SO_4(aq) \rightarrow H_2SiO_3(aq) + Na_2SO_4(aq)$$

An almost neutral solution of sodium silica is characterized by the appearance of turbidity and slowly forms a white gel. The deposits produced from the extraction process were obtained in the form of white silica powder which was shown at 25.92 grams of 40 grams of nickel slag with a yield percentage of 64.8%.

Manufacture of Silica-Chitosan Adsorbents

The resulting silica-chitosan is white, since its main component is naturally white. In addition, the effect of soaking time and low glutaraldehyde concentration (0.5%) is not enough to produce a strong yellow color so that the final result remains white or yellowish-white (pale). The synthesis results obtained were 7.31 grams from 10 grams of silica-chitosan with a yield percentage of 73.1%.

Pb Metal Desorption Using Acid Concentration with Silica Adsorbent

Table 1. Data on the Measurement of Pb (II) Metal Ion Concentrations in several Silica Adsorbent Mass Variations

HCl Concentration	Absorbance	C_0 Metal (ppm)	C_e Metal (ppm)	Adsorbed (mg/g)	Desorption percentage (%)
0,005 M	0,0121	50	1,7352	49,9889	3,47
0,01 M	0,0196	50	2,8382	49,9889	5,67
0,02 M	0,0347	50	5,0588	49,9889	10,11

Based on the results of measurements using AAS, desorption efficiency increased with the increase in HCl concentration. The highest desorption effectiveness was obtained at HCl concentration of 0.02 M, which was 10.11%. According to Sudiarta and Sulihingtiyas (2012), the process of desorption of metal ions by mineral acids involves the exchange reaction of H^+ ions from the acid with metal ions bound to the adsorbent functional group.

Pb Metal Desorption Using Acid Concentration with Silica-Chitosan Adsorbent

Table 2. Data on the Measurement of Pb (II) Metal Ion Concentrations at several Variations in the Silica-Chitosan Adsorbent Mass

HCl Concentration	Absorbance	C_0 Metal (ppm)	C_e Metal (ppm)	Adsorbed (mg/g)	Desorption percentage (%)
0,005 M	0,00275	50	0,0125	49,9768	0,0250
0,01 M	0,00337	50	0,0149	49,9768	0,0298
0,02 M	0,00283	50	0,0128	49,9768	0,00025

Based on the results of the study, it is shown that the effectiveness of desorption does not always increase along with the increase in HCl concentration. At a concentration of 0.005 M, the desorption effectiveness of 0.0250% shows that although the concentration of HCl is still relatively low, there has been a release of a small part of the Pb metal ions from the adsorbent surface, this is due to the interaction between the H+ ions of HCl and the Pb metal ions adsorbed on the adsorbent surface. H+ ions can swap positions with Pb ions through an ion exchange mechanism, causing some of the Pb ions to be released into the solution.

Cu Metal Desorption Using Acid Concentration with Silica Adsorbent

Table 3. Data on Cu (II) Metal Ion Concentration Measurement Results on several variations in silica adsorbent mass.

HCl Concentration	Absorbane	C_0 Metal (ppm)	C_e Metal (ppm)	Adsorbed (mg/g)	Desorption percentage (%)
0,005 M	0,0242	50	0,0048	26,524	0,0180
0,01 M	0,0457	50	0,0098	26,524	0,0369
0,02 M	0,0882	50	0,01999	26,524	0,0753

Based on the results of measurements using AAS, the desorption efficiency increased with the increase in HCl concentration of 0.02 M, which is 0.0753%. According to Zustriani (2019), the process of desorption of metal ions is caused by an acid medium, carboxyl, carbonyl or hydroxyl group in the adsorbent becoming protonated and not attracting positively charged metal ions, resulting in the release of metal ions into the solution or desorption agent.

Cu Metal Desorption Using Acid Concentration with Silica-Chitosan Adsorbent

Table 4. Data on Cu (II) Metal Ion Concentration Measurement Results at several Silica-Chitosan Adsorbent Mass Variations

HCl Concentration	Absorbance	C_0 Metal (ppm)	C_e Metal (ppm)	Adsorbed (mg/g)	Desorption percentage (%)
0,005 M	0,124220	50	5,3521	49,9939	10,7055
0,01 M	0,13607	50	6,0335	49,9939	12,0684
0,02 M	0,13346	50	5,8794	49,9939	11,7602

Based on the results of the study, it was shown that the desorption of Cu metal with silica-chitosan adsorbents did not always increase along with the increase in HCl concentration. The highest desorption effectiveness occurred at the HCl concentration of 0.01 M and decreased both at the lower concentration of 0.005 M and the higher concentration of HCl

0.02 M. At the concentration of 0.005 M, the effectiveness of desorption was 10.7055%, this shows that the number of H+ ions is not enough to replace the bound Cu2+ metal ions strong on the surface of the adsorbent (silica-chitosan), thus causing low desorption because the ion exchange process does not run optimally. This is in line with the opinion of Pohan (2016), who states that H^+ ions have a greater electrostatic attraction, so they are able to replace metal ions from bonds in functional groups through ion exchange mechanisms.

Comparison of Desorption Ability Using Silica and Silica-Chitosan Adsorbents Against Pb and Cu Metal Ions

Table 5. Data on Comparative Results of Percent Desorption of Metal Ions Pb (II) and Cu (II) Using Silica and Silica-Chitosan Adsorbents at Several Variations in HCl Concentration

Adsorben	Ion Logam	HCl 0,005 M	HCl 0,01 M	HCl 0,02 M
Silika	Pb	3,47%	5,67%	10,11%
	Cu	0,0180%	0,0369%	0,0753%
Silika-Kitosan	Pb	0,0250%	0,0298%	0,00025%
	Cu	10,7055%	12,0684%	11,7602%

Based on the data in the table above, it shows that the percentage of Pb ion desorption in silica increases significantly with an increase in the concentration of HCl from 3.47% (0.005 M) to 10.11% (0.02 M). This indicates that increased acidity (HCl concentration) facilitates the release of Pb ions from the silica surface. In contrast, the desorption of Cu ions in silica was very low despite a small increase from 0.0180% to 0.0753%. This shows that the bond of Cu with silica is stronger or the Cu adsorption mechanism is different, so desorption with HCl is less effective. In silica-chitosan adsorbents, Pb ion desorption is very low and even decreases at the highest HCl concentration from 0.0250% to 0.00025%. This shows that Pb is very strongly bound to silica-chitosan and difficult to desorpt with HCl.

On the other hand, the desorption of Cu ions in silica-chitosan is relatively high and stable at the concentration of HCl 0.005 M percent desorption of 10.7055, the concentration of 0.01 M percent of desorption is 12.0684% and the concentration of HCl is 0.02 M percent of desorption of 11.7602%. However, at the highest concentration of HCl, which is 0.02 M, the effectiveness of desorption decreased slightly to 11.7602%. This is due to a combination of excessive protonation factors of the chitosan functional group, the formation of a stable Cu-Cl complex, as well as changes in chemical balance and possible changes in the structure of the adsorbent. Therefore, although an increase in HCl concentration initially increases desorption, at concentrations that are too high the effectiveness actually decreases slightly. According to research by Yanti and Oktavia (2022), it shows that an increase in acid concentration does not change the efficiency of desorption, because at certain concentrations the adsorbent can be damaged and if the concentration used is lower, the ions to be desorpted are slightly separated from the adsorbent.

CONCLUSION

Based on the results of the study, it can be concluded that the highest desorption effectiveness reached 10.11% at HCl concentration of 0.02 M. Meanwhile, in the process of desorpation of Cu2+ metal with silica adsorbents, the desorption ability also increased as the HCl concentration increased, with the highest desorption effectiveness of 0.0753% at HCl concentration 0.02 M.

The use of silica-chitosan adsorbents, the effectiveness of Pb2+ metal desorption is low and inconsistent. The highest value was 0.0298% at an HCl concentration of 0.01 M, which shows that the Pb2+ bond on the silica-chitosan surface is stronger than that of silica adsorbents. Meanwhile, in Cu2+ metals with silica-chitosan adsorbents, the effectiveness of desorption is also low and inconsistent. However, the highest value obtained was higher, which was 12.0684% at an HCl concentration of 0.01 M. Nevertheless, this result still shows that the Cu2+ bond on the silica-chitosan surface is stronger than that of silica adsorbents..

RECOMMENDATIONS

This suggestion requires further research. Further research can explore other types of desorption solutions other than HCl, such as alkaline solutions or different acid mixtures, to see the more optimal desorption effectiveness of Pb^{2+} and Cu^{2+} metals.

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