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The Synthesis of Triamine Group Modified Silica Hybrid Material Coated on *Muara Kencan* Iron Sand for Hg (II) Adsorption

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Abstract

Hg metal was toxic and harmful to the environment because it does not biodegradation, can accumulate in living things, and pollute waters. One method that can be used was adsorption by inorganic-organic hybrid materials. This study aims to synthesize a triamine group modified silica hybrid material coated on iron sand or Magnetic Materials@Silica@Triamine (MM@SiO2@TA) to adsorb Hg(II) metal ions. The study began with the preparation of the iron sand of Muara Kencan Beach using magnets, washing using a solution of 1 M HCl and 0.5 M HF, and coating the material using sodium silica (SiO 25.5-28.5%) modified by a 99% triamine group with co-precipitation. The XRF characterization results showed that the magnetic material of the iron sand of Muara Kencan Beach has an iron oxide (magnetite) content of 73.31%. The iron oxide content increased after washing with HCl solution to 79.12% and HF solution to 81.81%. The results of FTIR, XRD, and TEM characterization showed that the MM@SiO2@TA was successfully synthesized with a size of 63.43 nm. The MM@SiO2@TA was tested to adsorb metal ion Hg(II) at a concentration of 100 ppm, pH 6, and a contact time of 1 hour. The AAS results showed that MM@SiO2@TA could adsorb metal ions Hg(II) as much as 60.82 ppm or 60.82%.

Keywords: *Iron Sand; Silica; Triamine; Muara Kencan Beach; Hg(II) metal ions*

Introduction

Heavy metals are harmful substances to human health and the environment because they are not biodegradable and can accumulate in living organisms over time (Rostamian et al., 2011). Heavy metals have a density ≥ 5 g/cm^3 (Powell et al., 2009). The heavy metal category includes mercury (Hg), which has a density of 7.6 g/cm^3 (Gautam et al., 2014). Because it can contaminate the survival of aquatic life, mercury is a harmful and toxic metal. The metal Hg is made up of waste from the chemical, printing, and gold mining industries (Adhani and Husaini, 2017). Therefore, dealing with the heavy metal Hg necessitates a method. Adsorption is one method that can be used. The adsorption method has several advantages, including the feature that it is inexpensive, environmentally friendly, does not produce hazardous waste, can be used repeatedly, and does not require a large number of solvents (Chung et al., 2012).

As heavy metal adsorbents, materials such as organic-inorganic hybrids can be used (Wu et al., 2008). Silica gel has several advantages, including being inert to redox reactions, being stable in acidic conditions, and being able to be modified with functional groups such as Mercapto (-CH_3) dan amine (–NH_2) which can increase the adsorbent's selectivity that make it suitable for use as a matrix of an inorganic compound in inorganic-organic hybrid materials (Wu et al., 2008).

Adsorbents with amine groups have better adsorption capacity than Mercapto groups modified adsorbents (Mohammad-Beigi et al., 2013). The research findings on silica modified with monoamine and diamine groups to adsorb metal ions were reported by Lakay (2009), which stated that the silica modified with the diamine group produced two times higher adsorption capacity than the silica modified with the monoamine group. As a result, silica modification with three amine groups was carried out in this study to boost its adsorption capacity in the adsorption of heavy metal ions.

In the batch system of adsorption process utilizing amine functional group modified silica, the difficulty of separating the adsorbent from the solution becomes an obstacle. Therefore, it is required to develop a means to make the separation of the adsorbent from the solution system easier, such as by using magnetic materials to modify the adsorbent (Zhang et al., 2013). An external magnet can be used to separate magnetically modified adsorbents from the solution system (Susanto et al., 2017).

Magnetite (Fe_3O_4) formed from natural iron sand can be used as a source of magnetic material (Prasdiantika and Susanto, 2017). Iron sand is found along the beach in Indonesia, such as in *Lansilowo* beach in Konawe Regency (Prasdiantika and Susanto, 2017), *Masang* beach in West Sumatra (Mufit et al., 2013), the *Pantai Selatan* in Kebumen (Ansori, 2013) and the beach in Kendal regency, Central Java (Dinas ESDM Jawa Tengah, 2018).

The beach in Kendal regency has more iron sand mineral content than others, which is around 3 million tons (Dinas ESDM Jawa Tengah, 2018). One of the beaches in Kendal Regency is *Muara Kencan* beach in Pidodo Kulon Village.

Iron sand needs to be prepared with an external magnet to get the magnetic material. The prepared magnetic material tends to still have impurities such as aluminum oxide, silica oxide, and other oxides (Prasdiantika and Susanto, 2017). The iron sand must be rinsed with an acid solution to remove the impurity oxides. Acid solutions that are often used are HF solutions, HCl solutions, or H_2SO_4. Prasdiantika dan Susanto (2020); Prasdiantika et al. (2019) conducted a study on the effect of the acid solution on the cleaning of magnetic material in iron sand at *Lansilowo* beach and *Jomblom* beach. The results showed that cleaning by using an acid solution could reduce the oxide composition of impurities, increase the composition of iron oxides, and increase the crystallinity of the material.

pH is one of the factors that influence the type of interaction between the adsorbent surface and the metal during the adsorption process. The amine group in the form of -NH_2 will be protonated to form -NH_3^+ as Lewis acid at pH 4.9. Thus, the amine group can bind a metal anion.

During the adsorption process, pH is one of the elements that might affect the sort of interaction between the adsorbent surface and the metal. The -NH_2 amine group will be protonated to generate –NH_3^+ as a Lewis acid at < pH 4.9, allowing the amine group to bind a metal anion. Meanwhile, the -NH_2 tends to be a Lewis base at pH >4.9, allowing it to bond to a metal cation complex (Lam et al., 2008)s. As a result, researchers hope to use the modified silica hybrid material with triamine groups coated on iron sand as an adsorbent in further studies. The purpose of this study is to synthesize triamine-modified iron sand hybrid material (MM@SiO_2@TA) and test its adsorption to heavy metal ions such as Hg (II). The benefits of this research include the production of MM@SiO_2@TA hybrid material as an adsorbent for heavy metal ions such as Hg (II), as well as the understanding of the adsorption ability of MM@SiO_2@TA for Hg (II) metal ions.

Research Methodology

Materials

The tools were used include beaker glass, porcelain mortar, mortar, measuring flask, sonicator, external magnet, analytical balance, oven, and shaker.

The materials were used include iron sand from *Muara Kencan* Beach in Pidodo Kulon Village, Kendal sub-district, 37% HCl (Merck), Sodium Citrate (Aldrich), Na_2SiO_3 with a SiO content of 25.5-28.5%, N-(3-trimethoxysilylpropyl) diethylenetriamine 99% (Aldrich), pH meter, $Hg(NO_3)_2$ (Merck), potassium hydrogen phthalate (Merck) and HF (Merck).

Preparation

The basic ingredients of natural iron sand from *Muara Kencan* beach are separated by external magnets and then dried in an oven at 80°C. Then it was ground until smooth, and the magnetic material powder was characterized by XRF, XRD, FT-IR, and TEM.

Activation

The prepared magnetic material was rinsed with distilled water and sonicated three times for each of 30 minutes. After that, it was rinsed with 10 mL of 1M hydrochloric acid solution and sonicated for 30 minutes. The magnetic material was rinsed by using distilled water until the pH was neutral. The solids were separated by magnets, then dried in an oven at 80°C and ground. The powder material obtained was characterized by XRF.

The magnetic material was then rinsed in 10 mL of 0.5 M HF solution, sonicated for 30 minutes, and then rinsed in distilled water until the pH was neutral. The activated magnetic material was added to 100 ml of 0.5 M citrate solution for 24 hours. External magnets were used to separate the solids, which were then dried at 80 ℃. XRF, FT-IR, spectrophotometer, and XRD were then used to characterize the iron sand magnetic material powder.

Coating

The sol-gel procedure is used to coat iron sand magnetic material with silica gel and bind propyldiethylenetriamine groups to the silica surface. 1 mL of 1 M hydrochloric acid solution was used to acidify 3 grams of magnetic material, and the precipitate was obtained (Mixture 1). Then 3.0 mL of Na_2SiO_3, 1.8 mL of TMSPDETA, and 1.2 mL of distilled water were added, followed by 30 minutes of sonication (Mixture 2). Furthermore, mixture 2 was sonicated for 10 minutes in a container containing mixture 1. Then, drop by drop, 1 M HCl solution was added to make a gel with a pH of 7.

The gel was then sonicated for 30 minutes before being aged overnight. After aging, the gel was separated by a magnet and rinsed with distilled water to pH 7. The precipitate was then baked in an oven at 80°C until it was completely dry, then characterized by using a spectrophotometer, XRD, and TEM.

Adsorption

A plastic bottle was filled with 10 mg of $MM@SiO_2@TA$ material. The adsorption process was carried out in batches, with 10 ml of $Hg(NO_3)_2$ at 100 ppm and pH 6 being placed in a plastic bottle and shaken for 1 hour. An external magnet is used to separate the sorted material from the supernatant. The supernatant obtained was then analyzed using Atomic Absorption Spectroscopy (AAS) to determine the concentration of unadsorbed Hg (II) metal ions.

Results and Discussion

The iron sand obtained from *Muara Kencan* Beach was prepared with an external magnet and activated by using an acid solution to reduce the impurity oxide content in the iron sand magnetic material.

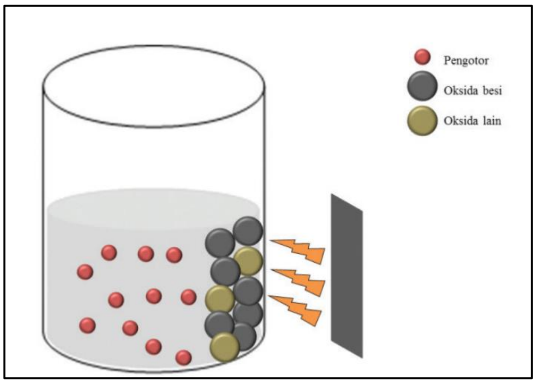
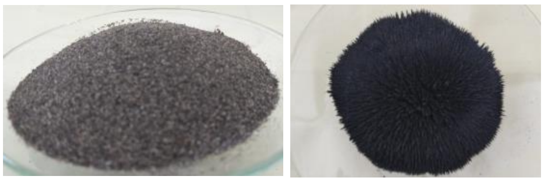


Figure 1: Illustration of Magnetic Material Separation using External Magnet (Prasdiantika and Susanto, 2017)

The obtained magnetic material produces a solid black color. This indicates that the iron sand material is dominated by magnetite (Fe_3O_4). This is in accordance with the characteristics of magnetite proposed by Cornell and Schwertmann (2003). This iron sand magnetic material will be used to synthesize a silica-triamine hybrid material.



(a) (b)

Figure 2: (a) Iron Sand, (b) Solid Black Iron Sand as Preparation Result

To determine the success of the synthesis, the magnetic properties of the prepared iron sand, activation results, and triamine group modified silica hybrid material coated on the iron sand magnetic material (MM@SiO2@TA) were characterized.

Results of Material Content Analysis (XRF Test)

The results of XRF characterization were carried out to determine the magnetic material content of iron sand as shown in Table 1. In Table 1, it can be seen that magnetite (Fe_3O_4) and other oxides such as elements SiO_2, TiO_2, Al_2O_3, Bi_2O_3, CaO, V_2O_3, and other minor oxides dominate the magnetic material of iron sand from Muara Kencan Beach. After the magnetic material was rinsed by using a 1 M hydrochloric acid solution, the iron oxide content increased by 5.81%, from 73.31% to 79.12% because some of the impurity oxides dissolved in the HCl solution. To optimize the iron oxide content, the magnetic material is also rinsed with an HF solution.

The iron oxide content increased by 3.69 % after rinsing with HF solution, going from 79.12 % to 82.81 %. Furthermore, impurity oxides such as silica oxide have lowered by 3.60 %, from 6.70 % to 3.10 %., It can happen because the HF solution dissolved silica. This is in accordance with the findings of Prasdiantika et al. (2019), who found that rinsing iron sand magnetic material with HF solution can reduce silica oxide levels.

Table 1. XRF Test Results of Magnetic Iron Sand from *Muara Kencan* Beach

Oxide	Content (%)		
	MM	MM + HCl	MM + HF
Fe_3O_4	73.31	79.12	82.81
SiO_2	7.30	6.70	3.10
TiO_2	6.78	6.63	5.31
Al_2O_3	6.15	5.50	4.20
Bi_2O_3	1.53	1.20	0.77
CaO	1.12	1.53	1.16
V_2O_5	1.10	0.61	0.59
Eu_2O_3	0.87	0.61	0.58
MnO	0.71	0.57	0.41
P_2O_5	0.23	0.24	0.11
K_2O	0.20	0.26	0.08
Re_2O_7	0.19	0.20	0.20
Cr_2O_3	0.18	0.17	0.12
Rb_2O	0.16	0.31	0.30
Br	0.12	0.26	0.19
ZnO	0.05	0.09	0.07

* MM = Magnetic Material

Results of Functional Group Analysis (FTIR Test)

The results of the FTIR test of the prepared magnetic material, the activation product, and the MM@SiO_2@TA synthesis product can be seen in Figure 3. Based on Figure 3, it can be seen that the prepared magnetic material has an absorption peak of

FTIR waves at (347, 394, 463, 571, 1034, 1636, and 3426) cm^{-1}. The magnetic material that is activated by acid solution has an absorption peak of FTIR waves at (324, 355, 471, 571, 1034, 1404, 1636, and 3449) cm^{-1}. The MM@SiO$_2$@TA product has an absorption peak of FTIR waves at (355, 463, 571, 795, 957, 1080, 1404, 1474, 1636, 2338, 2932, and 3449) cm^{-1}.

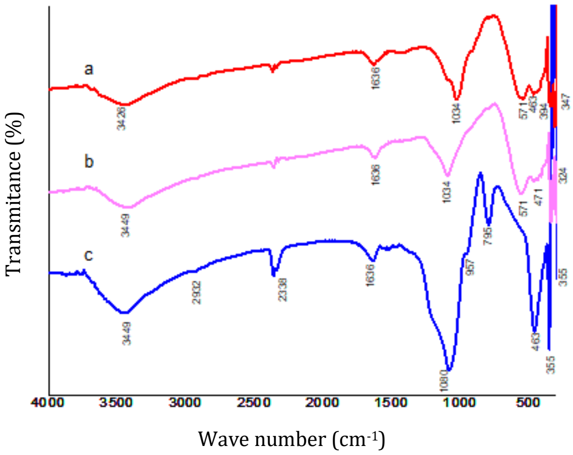


Figure 3. Absorption of FTIR wave peaks in the synthesized material: (a) MM preparation (b) MM activation, and (c) MM@SiO$_2$@TA

There were wave numbers that appeared at 463 cm^{-1} and 471 cm^{-1}, which were Si-O-Fe stretching vibrations; at 571 cm^{-1}, which is Fe-O bond stretching vibrations of magnetite. At 1636 cm^{-1} and 3426–3449 cm^{-1}, which were the stretching vibration of the –OH group of Fe-OH and Si-OH, indicated that the prepared and activated magnetic material, and MM@SiO$_2$@TA had –OH functional groups and strong Fe–OH bonds. It also indicated the presence of iron oxide in the magnetic material.

Impurities in the form of silica are still present in the sand preparation and activation results, as demonstrated by the asymmetric stretching vibration of Si-O-Si at 1034-1080 cm^{-1}. A –COO– group and a C–H bond from the citrate group can also be found in the activated iron sand. It indicated that the iron sand dispersed in citrate was successfully synthesized in the presence of wavenumber of 1404 cm^{-1}.

In MM@SiO$_2$@TA, there were also Si-O-Si bonds with symmetric stretching vibrations, N–H and C–H bonds from the propyldiethylenetriamine group, and the –OH group from Si-OH, which showed that the activated iron sand has been successfully coated with propyldiethylenetriamine group modified silica. This was shown at 795 cm^{-1}, which was the Si-O-Si symmetric stretching vibration of the propyldiethylenetriamine group modified silica, at 1474 cm^{-1}, which was the N–H bending vibration of the propyldiethylenetriamine group, and at 2932 cm^{-1}, which is the asymmetry and symmetry stretching vibration of the C–H. Thus, MM@SiO$_2$@TA was successfully synthesized.

The results of the analysis of the crystallinity and particle size of iron sand and MM@SiO$_2$@TA (XRD Test). The results of the XRD test on the prepared magnetic material, the activation results, and the product of the synthesis of MM@SiO$_2$@TA are shown in Figure 4. Identification of the type of oxide minerals was carried out by matching the standard diffraction pattern of JCPDS magnetite number 01-075-0449 with six characteristic diffraction peaks of magnetite at the Miller index: 220, 311, 400, 422, 511, and 440, which were produced by the synthesized material.

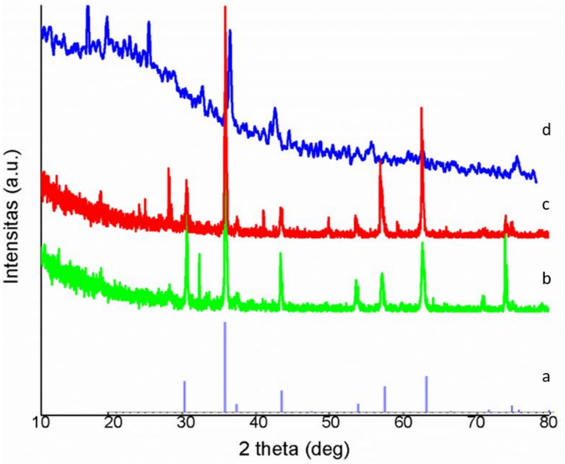


Figure 4: a) Diffraction pattern of JCPDS Magnetite 01-075-0449, b) MM preparation, c) MM activation, and d) MM@SiO$_2$@TA

Based on Figure 4, the XRD diffraction pattern on the prepared iron sand, activated iron sand, and MM@SiO$_2$@TA material have similarities with peak 2θ on the standard diffraction pattern of magnetite number 01-075-0449. This indicates that the prepared magnetic material, the activation product, and the MM@SiO$_2$@TA synthesis product contain iron oxide minerals in the form of magnetite (Fe_3O_4).

Based on the diffraction pattern in Figure 4, the particle size (Dxrd) for the prepared iron sand material, activation product, and MM@SiO$_2$@TA was determined by the Deybe-Scherrer equation as *stated by* Wu dkk (2011). In addition, the crystallinity of the material can also be determined based on the intensity of the diffraction pattern. The results of the calculation of particle size and material crystallinity are presented in Table 2. Based on Table 2, the particle size of prepared iron sand, activated iron sand, and MM@SiO2$_2$@TA have nanoparticle sizes because they have sizes in the range of 1–100 nm as stated by (Kamal, 2018).

Tabel 2. Size and Crystallinity Materials

Materials	Size (nm)	Crystallinity (%)
MM Preparation	81.20	26.85
MM Aktivation	36.11	58.48
MM@SiO$_2$@TA	63.43	48.91

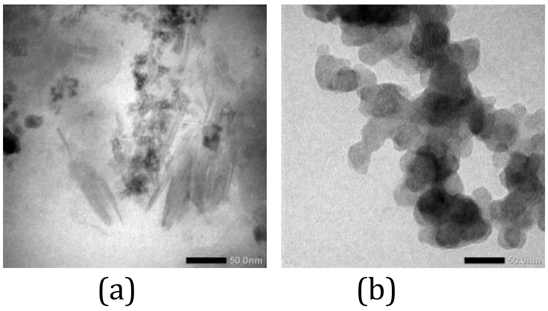
The activated magnetic material produced a smaller particle size of 36.11 nm than the prepared magnetic material, 81.20 nm. It was caused by the prepared magnetic material being only rinsed by using distilled water, so the particles were still in the form of aggregates. Meanwhile, in the activated iron sand material, some of the impurity oxides had dissolved due to rinsing with an acid solution. Besides that, the activated magnetic material was soaked in a sodium citrate solution to reduce the aggregation of magnetite particles, as stated by Susanto et al., (2016); Prasdiantika and Susanto (2016) and supported by the TEM results in Figure 5. As a result, the particle size was smaller than the prepared material.

The prepared iron sand magnetic material had a lower crystallinity of 26.85% because there were still impurity oxides such as silica which were amorphous as stated by Prasdiantika and Susanto (2017). The activated iron sand magnetic material had a higher crystallinity, 58.48%, than the prepared magnetic material. It was caused by the content of impurity oxides such as silica oxide had been reduced. Therefore, the activated iron sand magnetic material had a better magnetic ability to be used as a material in the synthesis of triamine group modified silica hybrids. It was used to facilitate the process of separating the synthesized material from the solution system with an external magnet (Susanto et al., 2016).

Based on Table 2, the MM@SiO$_2$@TA material had a larger particle size, 63.43 nm, than the activated iron sand, and had a lower crystallinity (48.91%) compared to the activated iron sand. It was caused by the effect of amorphous silica on the MM@SiO$_2$@TA material detected in the swelling diffraction pattern between 2-theta 22-24 deg as stated by Susanto and Prasdiantika (2018). This indicates that the activated iron sand has been successfully coated with triamine group modified silica. Thus, MM@SiO$_2$@TA was successfully synthesized.

Results of Morphological Analysis (TEM test)

The results of the TEM test on the prepared magnetic material and MM@SiO$_2$ @TA synthesis product with a magnification of 50 nm. In the image results of TEM 5 (a), the prepared iron sand has black parts (magnetite particles), and the gray and pointed parts are impurities in the form of silica, as suggested by Li et al., (2013), In Figure 5 (b), the MM@SiO$_2$@TA synthesis product has a gray part (a triamine group modified silica) and a black part (magnetite oxide) in iron sand.



(a) (b)

Figure 5: TEM characterization image at 50 nm magnification on samples (a) prepared iron sand and (b) MM@SiO$_2$@TA

In the triamine group modified silica coating, the addition of HCl 1 M solution to the activated iron sand magnetic material that has been dispersed with citrate causes the citrate to be released and produces magnetite with a Fe-OH group, which allows it to react with triamine modified silica compounds. The acidified iron sand will exchange ligands with silica, then condense and coat the surface of the magnetic material (Prasdiantika and Susanto, 2017; Li et al., 2013).

AAS Test Results

The MM@SiO$_2$@TA adsorbent was tested for the adsorption of Hg (II) metal ions. The experiment was conducted on 10 mL of Hg(NO$_3$)$_2$ at a concentration of 100 ppm for 1 hour at pH 6. According to AAS measurements, as much as 10 mg of MM@SiO$_2$@TA adsorbent was able to adsorb 60.2 ppm of Hg (II) metal ions or as much as 60.82%. As a result, synthesized MM@SiO$_2$@TA adsorbent can adsorb the Hg (II) metal ions.

At pH 6, the Hg (II) metal ions were in the form of Hg^{2+} with the optimum fraction as stated by Zhang et al., (2013) and the triamine group as a Lewis base that can interact with cations. The charge difference between the adsorbent MM@SiO$_2$@TA and Hg (II) metal ions caused strong ionic and electrostatic interactions. This bond caused the adsorption of Hg (II) metal ions on the surface of the adsorbent.

Conclusions

The results of XRF characterization showed that the prepared iron sand of *Muara Kencan* Beach, Kendal Regency, had a high iron oxide content of 73.31% with a type of magnetic material in the form of magnetite. It increased after rinsing using HCl and HF solutions to be 81.81%. The results of FTIR, XRD, and TEM characterization showed that the triamine modified silica hybrid material coated on iron sand (MM@SiO$_2$@TA) was successfully synthesized with a size of 63.43 nm. At a concentration of 100 ppm, pH 6, and a contact time of 1 hour, the adsorbent was able to adsorb 60.82% (60.82 ppm) of Hg (II) metal ions.

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