Synthesis and Characterization of Triamine modified coated Iron Sand Hybrid Nanomaterials originating from Kendal Coast

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1. Introduction

Nanomaterials have broad and unique applications in various fields. In this research, synthesis of Iron Sand Magnetic Hybrid nanomaterials coated with propyldiethylenetriamine modified silica (PB@SiO\textsubscript{2}@TA) originating from the coast of Muara Kencan, Kendal Regency. This study began with iron sand preparation, continued with activation, dispersing iron sand, then coating iron sand with propyldiethylenetriamine modified silica. The resulting product was characterized by X-Ray Fluorescence (XRF), Fourier Transform Infrared (FT-IR) Spectrophotometer, X-Ray Diffractometer (XRD), and Transmission Electron Microscope (TEM). The characterization results show that the iron sand of the coast of Muara Kencan Beach has a high iron oxide content (81.66%) with minerals in the form of magnetite. The characterization results also showed that the hybrid iron sand coated with propyldiethylenetriamine modified silica (PB@SiO\textsubscript{2}@TA) was successfully synthesized with a crystal size of 36.21 nm, with better particle dispersion than the prepared iron sand.

In order to improve the ability to adsorb heavy metals, an organic compound with three amine groups of N-(3-trimethoxysilylpropyl) diethylenetriamine (TMSPDETA) was used to modify silica-coated iron sand. The pH factor influences the type of interaction that occurs between the metal and the adsorbent surface. At low pH (pH < 4.9), the protonated amine group (-NH\textsubscript{3}) forms -NH\textsubscript{4}\textsuperscript{+} as Lewis acid so that it can bind to a metal...
anion complex [18]. However, at pH > 4.9, the -NH₂ group tends to be a Lewis base so that it can bind metal cations [18]. Thus, it is expected that the magnetized silica hybrid nanomaterial modified triamine group can be utilized as an adsorbent in further research. The purpose of this study was to synthesize and characterize the triamine modified silica-coated iron sand hybrid material (Pb@SiO₂@TA). The benefit of this research is the production of a hybrid triamine modified silica-coated iron sand material (Pb@SiO₂@TA).

2. Methodology

The stages of this research consisted of a) Preparation of iron sand magnetic material treated with water and characterization of the preparation material. b) Coating of iron sand material by adding NaSiO₃ and TMSPDETA solution and continued with a characterization of the synthesized material.

2.1. Equipment and Materials

The equipment for preparation consists of a measuring cup, beaker glass, volumetric flask, porcelain mortar, sonicators, ovens, analytical balance, external magnets (Niobium), and shakers. While the analysis equipment consists of X-Ray Fluorescence (PAnalytical Minipal 4) for identification of the constituent elements of materials, Fourier Transform Infrared Spectrophotometer (FT-IR, Shimadzu Prestige 21) for identification of functional groups of materials, X-Ray Diffractometer (XRD, XRD-6000 Shimadzu) for identification of mineral types, material size and crystallinity, and Transmission Electron Microscope (TEM, JEOL TEM-1400) for observing the morphology and dispersion of materials.

The material used consisted of iron sand from Muara Kencan Beach, Kendal Regency, Central Java, 37% HCl (Merck), Sodium Citrate (Aldrich), NaSiO₃ with SiO content of 25.5–28.5%, N-(3-trimethoxysilylpropyl) diethylenetriamine (TMSPDETA) 99% (Aldrich), and universal pH paper

2.2. Iron Sand Magnetic Material Preparation

The essential ingredients of iron sand were separated using an external magnet. Solids attracted by external magnets were dried in an oven at a temperature of 70–80°C for 24 hours, then crushed and weighed. The obtained iron sand powder was characterized by XRF, FT-IR spectrophotometer, XRD, and TEM.

2.3. Activation and Dispersion of Iron Sand Magnetic Materials

Five grams of magnetic iron sand from the preparation was then put into a beaker. Then washed with distilled water and sonicated for 30 minutes and repeated three times. After that, the magnetic material in the beaker was washed using 10 mL HCl solution and sonicated for 30 minutes, then rinsed using distilled water to neutral pH. An external magnet separated the mixture obtained in the beaker, and the sediment is taken. The activated magnetic material was then added with 100 mL of 0.5 M sodium citrate solution and allowed to stand for 24 hours. The obtained solid was separated with an external magnet and dried in an oven at a temperature of 70–80°C for 24 hours. Then the solid was crushed, weighed, and characterized with XRF, FT-IR spectrophotometer, XRD, and TEM.

2.4. Coating of iron sand magnetic material by triamine modified silica

The process of coating iron sand material by silica gel and the attachment of a group of propyldiethylenetriamine to the surface of silica was conducted through the sol-gel process. Three grams of iron sand material were acidified with 1 mL 1 M HCl solution, and the precipitate was taken (Mixture 1). Then as much as 3.0 mL of NaSiO₃ solution added with 1.2 mL of distilled water and 1.8 mL of TMSPDETA, then sonicated for 30 minutes (Mix 2). Then mixture 2 was put into a container containing mixture 1 and sonicated for 10 minutes. After that into the mixture, 1 M of HCl solution was added dropwise to pH of 7, and a gel was formed. Then, the gel formed was sonicated for 30 minutes and left for 24 hours. After aging, the gel was washed with distilled water to a neutral pH, separated with an external magnet, and the sediment was taken. The precipitate obtained was dried in an oven at 80°C for 24 hours. After drying, the solid was crushed and separated using an external magnet. Solids attracted by magnets were weighed and characterized by FT-IR, XRD, and TEM spectrophotometers.

2.5. Data analysis

Data analysis of iron sand material coating by silica gel and clustering was performed by interpreting the data obtained from the results of the characterization of the synthesized material.

Iron Sand Material Content. XRF analysis was used to identify the magnetic material content of the iron sand in Muara Kencan Beach, Kendal Regency, Central Java.

Functional Group. FT-IR spectrophotometer analysis is used to qualitatively identify the presence of functional groups contained in a sample compound.

Crystallinity and particle size. The XRD analysis method is used to obtain X-ray diffraction patterns. The characteristic peak diffraction angle of magnetite is at 2θ=30.0°; 35.4°; 43.0°; 53.4°; 56.9°; and 62.5° with Miller’s index values of 220, 311, 400, 422, 511 and 440, respectively [19]. Through XRD data, crystal size can be determined using the Debye-Scherrer equation [20]:

\[
D = \frac{0.9 \cdot \lambda}{\beta \cos \theta}
\]

where D = Crystal size (nm), λ = wavelength of the metal atom used (nm), β = half the width of the peak FWHM (radians), θ = Bragg diffraction angle (degrees). The crystallinity of the magnetic material can be seen from the intensity of the X-ray diffraction peak. A sharper peak in XRD indicates higher crystallinity [21]. Based on the XRD pattern, magnetite coated with silica has lower crystallinity compared to magnetite coated with citrate, due to the amorphous nature of silica [22].
Amorphous silica has a characteristic peak at $2\theta = 20 - 26^\circ$ [23].

Morphology. TEM is used to determine the dispersion of magnetite nanomaterials [11] and see the image of the coated material. The magnetite part is black, while the silica layer is gray [15].

**Table 1.** FT–IR wave numbers of sodium citrate dispersed magnetite nanomaterials and amine-modified FeO$_x$@SiO$_2$.

| Wavenumber (cm$^{-1}$) | Identification | Reference |
|------------------------|----------------|-----------|
| 466                    | Si–O–Fe stretching vibrations | [24] |
| 540                    | Vibrational stretches of Fe–O bonds | [25] |
| 805                    | Si–O–Si stretching vibrations | [22] |
| 1092                   | Si–O–Si stretching vibrations | [24] |
| 1307–1463              | –COO– symmetry stretching vibrations | [11] |
| 1458–1645              | N–H bending vibration | [26] |
| 1628–1636, 3441–3487   | –OH vibration stretching groups of Fe–OH and Si–OH | [11, 16] |
| 2900                   | C–H asymmetrical stretching vibrations | [8] |
| 2850                   | C–H symmetry stretching vibrations | [8] |

3. Results and Discussion

Iron sand samples were obtained from the Muara Kencan Beach, Kendal Regency, Central Java Province. Iron sand was prepared using an external magnet to separate the material content of the iron sand from its impurities; then, it is washed with distilled water, then dried. Then the iron sand was activated using 1 M HCl solution and 0.5 M sodium citrate solution. The iron sand magnetic material obtained had a black color, which indicated that the iron sand was dominated by the magnetic iron oxide content (FeO$_x$). This result was following the characteristics of magnetite proposed by Cornell and Schwertmann [9].

Magnetic material for iron sand preparation, activated iron sand material, and the synthesized product in the form of hybrid triamine modified silica-coated iron sand material (PB@SiO$_2$@TA) were characterized to find out the success of synthesis with a) analysis of material contained in iron sand with X-Ray Fluorescence (XRF), b) functional group analysis with Fourier Transform Infrared (FT–IR) Spectrophotometer for analysis of functional groups in materials, c) analysis of crystallinity and crystal size with X-Ray Diffractometer (XRD) for analysis, and morphological analysis with Transmission Electron Microscope (TEM).

1.1. Results of analysis of material contained in iron sand (XRF test)

The results of XRF characterization on iron sand from the preparation and activation results are shown in Table 2. Based on Table 2, it is known that the magnetic material of the iron sand coast of Muara Kencan Beach has a content that is dominated by iron (Fe) and a minority content of impurities such as Ti, Si, Al, Ca, V, Mn, Eu, Rb, Re, K, P, Cr, and Zn. Meanwhile, activated iron sand showed an increase in iron content compared to prepared iron sand, from 81.66% to 82.64%. This is because some of the impurities have dissolved in the HCl acid solution used in the activation process.

**Table 2.** Results of XRF characterization in prepared and activated iron sands.

| Elements | Prepared Iron sand | Activated iron sand |
|----------|--------------------|---------------------|
| Fe       | 81.66              | 82.64               |
| Ti       | 6.75               | 6.80                |
| Si       | 4.1                | 2.6                 |
| Al       | 2.3                | 3                   |
| Ca       | 1.21               | 0.96                |
| V        | 0.64               | 0.64                |
| Mn       | 0.62               | 0.56                |
| Eu       | 0.54               | 0.36                |
| Rb       | 0.41               | 0.29                |
| Re       | 0.3                | 0.59                |
| K        | 0.24               | 0.08                |
| P        | 0.2                | 0.19                |
| Cr       | 0.16               | 0.16                |
| Zn       | 0.09               | 0.08                |

3.1. Iron Sand and PB@SiO$_2$@TA functional groups

The results of FTIR characterization on magnetic iron sand were prepared, activated iron sand, citric acid dispersed iron sand, and the PB@SiO$_2$@TA were shown in Figure 1.
From Figure 1, the prepared iron sand has the peaks of FTIR spectra at 471, 571, 1034, 1636, and 3449 cm⁻¹. Activated iron sand has peaks of FTIR spectra at 463, 571, 1041, 1404, 1443, 1627, 2855, 2924, and 3449 cm⁻¹. Meanwhile, hybrid trimine modified silica-coated iron sand material has FTIR spectra peaks at 463, 623, 772, 1049, 1474, 1582, 1627, 2862, 2924, and 3349 cm⁻¹.

Based on Figure 1 and Table 1, wave numbers at 463 cm⁻¹ and 471 cm⁻¹ indicate the existence of Si–O–Fe stretching vibrations. Wave numbers at 571 cm⁻¹ and 623 cm⁻¹ indicate the existence of stretching vibrations of the Fe–O bond of magnetite. The wavenumber at 772 cm⁻¹ indicates the symmetric stretching vibration of Si–O–Si from propyldiethylenetriamine modified silica groups. Wave numbers at 1034–1049 cm⁻¹ indicate the existence of Si–O–Si asymmetric stretching vibrations. Wave numbers at 1404 cm⁻¹ and 1443 cm⁻¹ indicate the presence of symmetric stretching vibrations –COO– of the citrate group. Wave numbers at 1474 cm⁻¹ and 1582 cm⁻¹ indicate the presence of N–H buckling vibrations from the propyldiethylenetriamine group. Wave numbers at 1627–1636 cm⁻¹ and 3449 cm⁻¹ indicate the existence of stretching vibrations of the –OH group from Fe–OH and Si–OH. Wave numbers at 2855–2866 cm⁻¹ and 2924 cm⁻¹ indicate symmetry stretching vibrations and C–H asymmetries.

Based on Figure 1 shows that the prepared and activated iron sands and PB@SiO₂@TA have a –OH functional group and a Fe–OH bond that shows the iron oxide content of magnetic material. The prepared and activated iron sands also still have impurities in the form of silica. The iron sand activated by HCl and sodium citrate also has a –COO– group and a C–H bond from the citrate group that demonstrates that the citric dispersed activated iron sand was successfully synthesized. In PB@SiO₂@TA, there is also a Si–O–Si bond with symmetric stretching vibrations, N–H and C–H bonds of the propyldiethylenetriamine group, the –OH group from Si–OH which shows that the activated iron sand was successfully coated with a propyldiethylenetriamine modified silica group. Thus, PB@SiO₂@TA was synthesized successfully.

### 3.2. Crystallinity and size of iron sand mineral and PB@SiO₂@TA

The results of XRD characterization on the prepared iron sand magnetic material, citric dispersed activated iron sand, and PB@SiO₂@TA are shown in Figure 2. Identification of the type of oxide minerals was carried out by matching the FeO₄ diffraction pattern standard of JCPDS 00-001-1111 with six diffraction peaks that are characteristic of magnetite with the Miller index of 220°, 311°, 400°, 422°, 511°, and 440°.

Based on Table 3, the sizes of iron sand crystals are prepared, citric dispersed-activated iron sand, and PB@SiO₂@TA have nanoparticle size because it has a size in the range 1–100 nm referring to Kamal [21]. Activated iron sand has a smaller crystal size and a higher diffraction pattern intensity than the prepared sand iron. This may be since the prepared iron sand is only washed with distilled water, so the particles are still in the form of aggregates and have lower crystallinity because they still have more amorphous silica impurities, as suggested by Prasadantika and Susanto [27]. Whereas in activated iron sand material, which is iron sand washed with HCl solution, impurities are lost and soaking with...
sodium citrate solution reduced aggregation of magnetite particles as suggested by [19, 28, 29] and supported the TEM results in Figure 3.

PB@SiO₃@TA material has a larger crystal size and lower intensity of diffraction pattern compared to activated iron sand. This is due to the effect of amorphous modified silica propyldiethylenetriamine groups on PB@SiO₃@TA material, as indicated by a diffraction pattern that is inflated, as stated by Hong et al. [22] and Tan et al. [23]. This shows that the activated iron sand was successfully coated with a propyldiethylenetriamine modified silica. Thus PB@SiO₃@TA was synthesized successfully.

3.3. Morphology of PB@SiO₃@TA

The images of Transmission Electron Microscopy (TEM) on prepared iron sand and citric dispersed activated iron sand with a magnification of 500 nm are shown in Figure 3.

The TEM image in Figure 3 shows that the iron sand produced from activated citrate dispersed (3b) has a more evenly dispersed particle dispersion than the prepared iron sand (3a). That is because sodium citrate in activated iron sand has succeeded in reducing the aggregation of magnetite material in iron sand. The black color in the TEM (3a) image of the prepared iron sand is a magnetite particle, while the gray part is a silica impurity element, as stated by Li et al. [15]. While the gray color in the TEM (3b) image of activated iron sand dispersed by citrate groups is a citrate layer with impurities in the form of silica.

![Figure 3. The images of Transmission Electron Microscopy (TEM) on (a) prepared iron sand and (b) citric dispersed-activated iron sand](image)

TEM images of citric dispersed-activated iron sand and PB@SiO₃@TA at a magnification of 200 nm are shown in Figure 4.

From Figure 4a, it is observed that there are many gray parts, which are the layers of citrate and silica impurities in iron sand. Whereas in Figure 4b, PB@SiO₃@TA has a gray portion derived from a modified silica layer of the propyldiethylenetriamine group and has a black part, which is a magnetite oxide in iron sand. In the modified silica coating of the propyldiethylenetriamine group, citric dispersed-activated iron sand was added with 1 M HCl, which causes the citrate to release; hence the magnetite produces a Fe-OH group which allows reacting with the propyldiethylenetriamine modified silica. The acidified iron sand exchanges ligands with silica compounds, then the silica-based ligands undergo condensation and coat the surface of the magnetite, as stated by Hong et al. [22] and Susanto and Prasdiantika [30].

![Figure 4. The images of Transmission Electron Microscopy (TEM) on (a) citric dispersed-activated iron sand and (b) PB@SiO₃@TA](image)
4. Conclusions

The results of XRF, FTIR, XRD, and TEM characterization showed that the iron sand in the Muara Kencan Beach, at Kendal Regency had a high iron oxide content (81.66%) with a type of magnetic material was magnetite. The results of FTIR, XRD, and TEM characterization also showed that the iron sand hybrid nanomaterial coated with propyl diethylenetriamine modified silica (PB@SiO₂@TA) was successfully synthesized with a crystal size of 36.21 nm and better particle dispersion compared to the prepared iron sand.

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