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Isolation of Sillica from Pantai Cermin Sand and Modification with Sodium Lauryl Sulfate and Ligan Ethylendyamine Through Coating Method as Absorption of Pb Metal

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ABSTRACT

Research on the formation of synthesis and modification coating of silica and obtained silica with a quartz structure used to absorb lead metal ions. This isolation was performed by extracting silica from sand with the co-precipitation method using NaOH 7M and then adding HCl 2M. The resulting silica was coated with sodium lauryl sulfate and ethylenediamine to increase adsorption. The FT-IR spectrum on silica shows the presence of Si-O-Si, Si-O-, Si-OH, S–O, SO_3, CH_2, and NH—Sillica coating, which sodium lauryl sulfate and ethylenediamine ligands were carried out by coating method at pH 2. The results obtained before and after coating on silica were characterized by FT-IR, XRD, and SEM EDX analysis. The FT-IR spectrum on sillica showed the presence of Si-O-Si, Si-O, and Si-OH functional groups. After the coating, there was a change in the spectrum, indicating new functional groups in the S-O, SO_3, CH_2, and NH spectra. Characterization using XRD shows the diffraction peaks were of 2θ 27,6297°, which indicates the amorph. After coating, the diffraction peaks appeared at an angle of 2θ in the area of 19.62 °, 20.47°, 21.04 °, 25.56 °, 26.50 °, and 29.73 ° with a high enough intensity indicating increased crystallinity. The morphology, composition, and size of the silica produced before and after coating were observed by SEM-EDX. Where there is a change in the size before and after a modification, that is 100,788 nm to 85,3773 nm. Silica before and after coating is used as an adsorbent to reduce levels of heavy metal lead (Pb). This analysis showed that the adsorption of Pb^{2+} with silica was 4,5162 ppm, while the adsorption of Pb^{2+} after coating was 1,1146 ppm.

Keywords: Adsorbent, Coating, Ethylenediamine, Sodium Lauryl Sulfate, Silica

ABSTRAK

Telah dilakukan penelitian mengenai pembuatan dan pelapisan silika didapatkan silika yang memiliki struktur kuarsa yang digunakan sebagai penyerap ion logam timbal. Pembuatan silika dilakukan dengan mengekstraksi silika dari pasir dengan metode kopresitasi menggunakan NaOH 7 M kemudian ditambahkan HCl 2M. Pelapisan silika dilakukan menggunakan natrium lauril sulfat dan ligan etilendiamin dilakukan dengan metode coating pada pH 2. Hasil silika yang diperoleh sebelum dan susudah coating dikarakterisasi dengan analisa FT–IR, XRD, SEM EDX. Spektrum FT-IR pada silika menunjukkan adanya gugus fungsi Si-O-Si, Si-OH. Setelah dilakukan coating terjadi perubahan terhadap spektrum yang menunjukkan adanya gugus fungsi baru pada spektrum S–O, C–N, CH_2, dan NH. Hasil karakterisasi menggunakan XRD menunjukkan puncak difraksi yang melebar pada 2θ pada daerah 27,6297° yang menunjukkan fasa amorph. Setelah dilakukan coating muncul puncak difraksi pada sudut 2θ di daerah 19,62°, 20,47°, 21,04°, 25,56°, 26,50°, 29,73° dengan intensitas yang cukup tinggi menandakan kritalinitas meningkat. Morfologi, komposisi, dan ukuran partikel silika yang dihasilkan sebelum dan sesudah coating diamati dengan SEM-EDX. Dimana terjadi perubahan ukuran rata rata sebelum dan sesudah coating yakni 100,788 nm ke 85,7337 nm. Silika hasil coating digunakan sebagai adsorben untuk menurunkan kadar logam berat Timbal (Pb). Tujuannya untuk membandingkan daya adsorpsi dengan dan tanpa dilakukan coating natrium lauril sulfat dan ligan

etilendiamin. Hasil Penelitian ini menunjukan bahwa penyerapan logam Pb2+ dengan silika sebesar 4,5162 ppm, sedangkan penyerapan logam Pb2+ dengan silika yang sudah terlapisi sebesar 1,1146 ppm.

Kata Kunci: Adsorben, Coating, Etilendiamin Natrium Lauril Sulfat, Silika,

1. Introduction

Indonesia is an island with a very long coastline and abundant natural resource potential [1]. Coastal sand is one of the minerals that is found in abundant amounts. The beach sand has a variety, including black and white beach sand [2]. Coast sand tends to contain material such as quartz sand or silica sand. Silica sand is composed of top silica crystals (SiO_2). It contains toxic compounds such as iron oxide, calcium oxides, alkaline oxides, and magnesium oxides, as well as clay and organic residues from animals and plants [3]. Therefore, silica sand needs to be further purified to obtain pure silica that can be used in various applications.

Silica sand has been widespread, both as a primary raw material and a mixture. As a primary raw material, for example, used in the glass industry, cement, ceramic mosaics, silicone fero raw materials, silicon carbide abrasive materials (glass and sandblasting), while as a mixture material, e.g., in the car industry, oil and mining industry, fire-resistant bricks (refractory), and so on [4]. Silica sand can also be used in nanotechnology, such as nano-silica synthesis.

Silica nanoparticles can be synthesized in several modes: sol-gel, gas phase, co-precipitation, etc [5]. The most commonly used method is co-precipitation. This method is one of the methods of synthesis of inorganic compounds based on the sedimentation of more than one substance simultaneously when passing through the saturation point. The co-recipitation method has several advantages, including a process that uses low temperatures in a relatively shorter time and a simple and easy-to-do way [6-7].

Surface modification of silica relates to the whole process aimed at changing the chemical composition of surfaces. It has increased at the modification of the functional group because some of the silanol groups are transformed into other functional groups [8]. Modification of function groups on silica surfaces can be done by various techniques, including modification impregnation by forming covalent bonds and coating [8]. Coating is a process of coating the surface of a nanoparticle material using an agent coating to coat one nanoparticle with another [9]. Organic components play an essential role in forming pores and the size of particles of a compound. This is due to the difference in size, type of shape, group of functions, and charge of the surface agent molecules that produce different micelles due to different interactions between the inorganic component and the organic ingredient [10]. Sodium lauryl sulfate is an anionic surfacing agent that acts as a coating for ligan attachment to positive-charged SiO_2. The increased ligan linkage to SiO_2 by sodium lauryl sulfate increases the absorption of heavy metals [11].

Many studies on silica, among them, Rahma Hayati (2015) has synthesized silica nanoparticles from the coastal sand of Purus Padang Padang West Sumatra with the method of co-precipitation obtained silica in the form of hexagonal crystals of the size of 25 nm–80 nm [12]. Surahmat Hadi (2011) synthesized silica based on Bancar sand with a wet method using NaOH 7 M, obtaining silica with a size of ~58 nm. Some studies on the modification and functionalization of silica [13], among them, Sari (2015) has modified the Mesopori silica Mobil Crystalline of Material Number-48 (MCM-48) with 1.5 diphenyltiocarbazone ligands, where there is no significant porous change [14]. Still, there is an increase in the absorption of Zn2+ metal ions. Ali Mirabi (2012) modified the silica nanoparticles with sodium lauryl sulfate as a coating and diphenylcarbazone as an accurate and sensitive Cr_3+ metal ion adsorbent at very small concentrations. Compared to diphenylcarbazone, ethylenediamine has several advantages as a ligan in the research, namely that it has a slightly better solubility in polar solvents [11].

The above factors also contribute to the obtained logam absorption capacity. Based on previous research, this study will make silica from Pantai Cermin sand and silica coating using sodium laureth sulfate and ethylenediamine as coating and ligan. Pantai Cermin sand is purified/leached using HCl and NaOH, then HCl to form co-creation. Silica crystal solid is subsequently reacted with sodium lauryl sulfate, an anionic surface agent as a coating, and ethylenediamine as a ligan to modify the morphology and size of particles that affect the adsorption properties of metal ions of the resulting material.

The researchers wanted to make carbon fiber derivatives from the PVA precursor by adding NPP based on the above exposure. PVA nanoparticles were made using an electrospinning method that was subsequently diodinated to increase the carbon yield by more than 40%. NPP simultaneously formed in PVA solution and completely assembled at the nanoparticle stabilization process at 180ºC. Ag nanoparticle addition is expected to accelerate/increase the graphitization process so carbon fibers can form at lower temperatures below 1200°C.

2. Materials and Methods

2.1. Equipment

In this study, the tools used glass, Analytical Balance (Radwag), Hot plate (Cimarec), Oven (Carbolite), Magnetic stirrer, aluminum & Passenger, Father 100 mesh, vacuum, filter paper, FTIR, XRD, SEM EDX, and UV Vis.

2.2. Materials

Materials used in this study were NaOH, HCl, HNO_3, sodium lauryl sulfate, ethylenediamine, distilled water, and deionized water.

2.3.Sand Preparation

Pantai Cermin sand preparation was washed repeatedly with distilled water, then washed, dried at air temperature, smoothed with foam and float, and refined using 100 mesh pads.

2.4. Manufacturing of Silica from Sand

A sample of 100 mesh sand is soaked 30 g in 1M of HCl for 12 hours while stirring to dissolve the resin on the sample. The sample is then washed with distilled water to purify it again and dried in the oven. Subsequently reacted with NaOH 7 M, then filtered. The filter escape solution is driped with HCl gradually by controlling the final pH 2. The resulting drip is washed with deionized water to remove NaCl up to five times with 300 ml aquades. After that, it is dried in the oven at a temperature of 110°C for 5 hours. After the water level has disappeared, the grinding is carried out with a pass to obtain silica powder. The results are characterized by FT-IR, XRD, and SEM-EDX.

2.5. Silica Modification

A total of 1 g of SiO_2 nanoparticles and 5 mL of HNO_3 4M are added to the Erlenmeyer, and then the Erlenmeyer is mixed mechanically for 10 minutes. Then 20 mL of a solution containing 200 mg of sodium lauryl sulfate as a coating and 2 mL ethylenediamine solution as a ligan were added to Erlemneyer pumpkin. The pH of the solution was adjusted to a pH of 2 with 1 M HCl solution, and then Erlenmeyer pumpkins were mechanically coated for 10 minutes and then filtered. Then, the containers were vacuumed so that silica was coated. Results are characterized by FT-IR, XRD, and SEM-EDX.

2.6. *Pb2+ solution generation 10 ppm*

10 mL of Pb_2+ 100 ppm solution was inserted into 100 mL of dried pumpkin. Then, it dissolved with distilled water to the bounder line and homogenized.

2.7 *Characterization*

Fourier Transform Infrared Shimadzu IR Prestige 21 function group observation analysis was performed in the 4500-450 cm^{-1} wave range. Crystality analysis was done with the Miniflex X-ray diffractometer. Morphology, diameter, and elemental measurements of silica were analyzed using SEM EDX Tensilon. UV-VIS spectrophotometry analysis using the Shimadzu UV Mini 1240 Lambda 243 instrument at a 200-800 nm wavelength range.

3. Result and Discussion

3.1 *Sand Preparation*

The initial treatment of this study was sample preparation; the sand from Pantai Cermin was washed and dried. Then, the sand was smoothed with clay and floats, then refined with 100 mesh of fine powder with a gray-white foam shade, as shown in Figure 1.

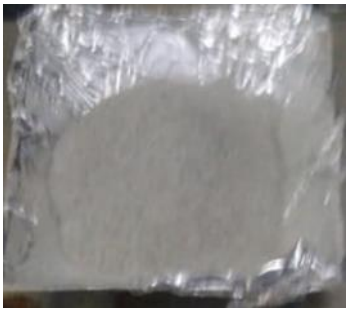


Figure 1. Pantai Cermin Sand with 100 mesh

3.2. *Making silica from sand*

Silica from Pantai Cermin Sand was obtained by 100 mesh sand samples soaking as much as 30 g in 1M of HCl for 12 hours while stirring. It's done to dissolve the resin that's on the sample. Then, the sample was washed with distilled water to purify it and dried in the oven (Figure 2).

Figure 2. Leaching Process Results

It is then reacted with 300 mL of NaOH 7 M coated with a magnetic stirrer for 24 hours while heated to a temperature of 130°C. This is done to extract silica silica from coastal sand since silica is soluble in NaOH according to the reaction can be seen in Figure 3.

$SiO_{2(aq)} + 2NaOH_{(aq)} \rightarrow Na_2SiO_{3(aq)} + H_2O_{(l)}$

Figure 3. Results of silica extraction processes using NaOH

The heating and condensation process was carried out using a hotplate stirrer that aims to increase the occurrence of intermolecular collisions in the solution to speed up the dissolution process [15]. The heated mixture was then filtered to obtain a sodium silicate solution as a filter with a mix of metal oxides and other residues. The filter is gradually pressed with HCl by controlling to pH 7 (Figure 4).

$Na_2SiO_{3(aq)} + 2HCl_{(aq)} \rightarrow H_2SiO_{3(l)} + NaCl_{(aq)}$

$H_2SiO_{3(s)} \rightarrow SiO_2.H_2O_{(s)}$

Figure 4. Co-creation process

The resulting sediment is washed with aquades to remove NaCl. After that, it is dried in the oven at a temperature of 110°C for 5 hours, as shown in Figure 5.

Figure 5. Silica

3.3. Silica Modification

The resulting silica is added to HNO_3 4M., which aims to remove the resin that covers the silica pores so that silica becomes open. The silanol cluster was bound to the surface of silica free from impurities and to diminish the size of the particles. Sodium lauryl sulfate is a coating agent for ligan attachment to positive-charged SiO_2. Ethylenediamine serves to increase adsorption. The reaction condition at pH 2 is intended for sodium lauryl sulfate to adhere to the silica's surface through self-aggregates. When the solution is inserted, sodium lauryl sulfate will form hemi-misal on silica with strong absorption [11]. Then, it was filtered, and the deposits were vacuumed to obtain modified silica. The removal process that occurred can be shown in the following reaction (Figure 6).

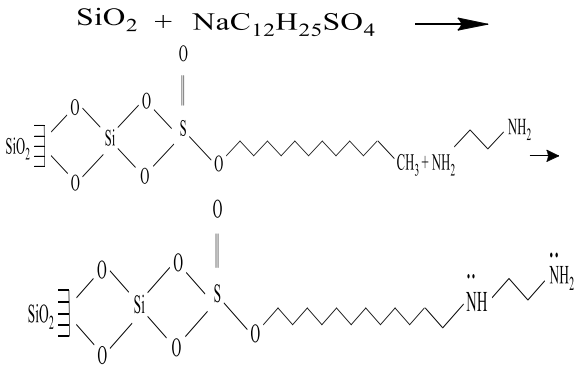


Figure 6. The process of removing the torque from the silica pores with HNO_3

a) b) c)

Figure 7. The mix of Sodium Lauryl Sulfate SiO_2 and Ethylenediamine Ligand

3.4. FT-IR Analysis

Silica before and after coating was analyzed using FT-IR to determine the presence of functional groups. The FT-IR results obtained are shown in Figure 8. The FT-IR spectrum in the wave number Figure 8 a shows the functional groups of silica with characteristics. The absorption peaks at wave numbers 3749.62 cm^{-1}, 3425.58 cm^{-1} show the Si-OH functional group on Si-OH, 1635.64 cm^{-1} shows Si-OH on Si-OH, 424.34 cm^{-1} shows Si-O-Si, 864.11 cm^{-1} shows Si-O-Si, 1010.70 cm^{-1} shows Si-O- Si or siloxane [16].

In Figure 8, the addition of sodium lauryl sulfate and ethylenediamine shows the presence of new functional groups that appear in the range of wavenumbers 1087.85 cm^{-1} indicating C-N functional groups, wave numbers 1219.01 cm^{-1} indicating S-O functional groups , wave numbers at 2924.09 cm^{-1} indicating C-H functional groups, wave numbers at 2854.65 cm^{-1} and 1465.90 cm^{-1} indicating CH groups from sodium lauryl sulfate. The functional group of ethylenediamine appears in the range of wave numbers 1504.48 $cm^{-1,}$ showing the NH functional group [16].

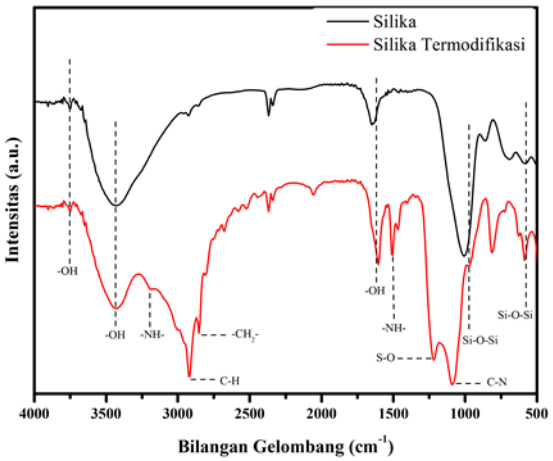


Figure 8. FT-IR spectra a) Before the addition of NaLS and ligand en b) addition of NaLS and ligand en

3.5. XRD Analysis

The coated silica and silica that had been obtained were characterized by XRD analysis to identify the phase of the material at 2θ angles as shown in Figure 9 below: The XRD diffractogram of silica is shown in Figure 9. a, where silica has a peak of 2θ in the region of 27°. The presence of diffraction peaks that widen and have low intensity in the 2θ range between 22° and 34° indicates that silica is amorphous [17].

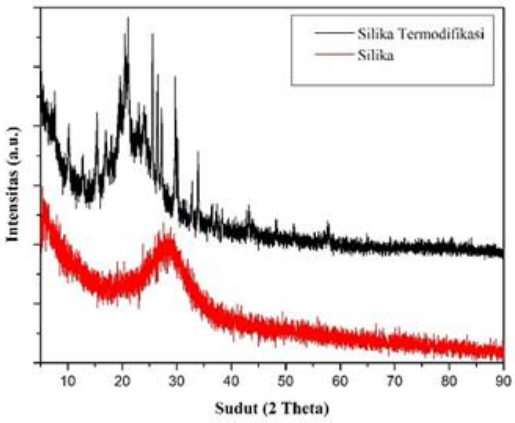
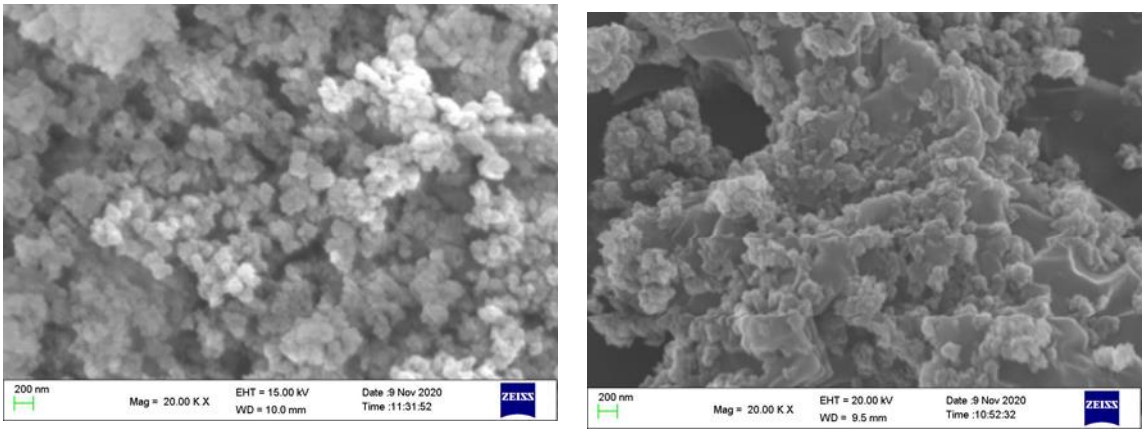


Figure 9. XRD of Silica a) Before adding NALS and ligand en b). addition of NALS and ligand en

The modified silica, Figure 9.b, was characterized by XRD analysis to identify the phase of the material at 2θ angles, as shown in Figure 4.2 b. The figure shows the success of the coating on silica in the form of the appearance of new diffraction peaks with a fairly high intensity in the areas of 19.62°, 20.47°, 21.04°, 25.56°, 26.50°, 29.73° [18]. In other words, the coating process causes the crystallinity of the coated silica to increase.

3.6. Function Analysis with SEM EDX

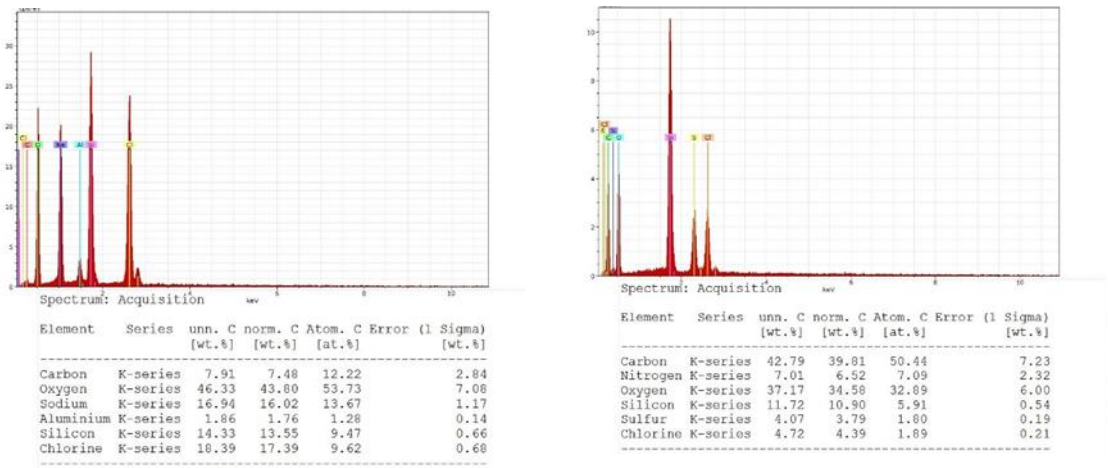
Morphological analysis of silica produced using the co-precipitation method using SEM at 20,000 times magnification can be seen in Figure 10. The silica produced from the co-precipitation method has a round shape that tends to be a homogeneous oval with sizes ranging from 63.58 nm to 119.675 nm with an average of 100.788 nm [19].



a) b)

Figure 10. a). SEM of Silica, b). SEM coated Sodium Lauryl Sulfate and Ethylenediamine

The silica coating results from sodium lauryl sulfate with ethylenediamine ligand has a size of 58.357 nm to 105.062 nm with an average of 85.3773 nm, as seen in Figure 11. Where sodium lauryl sulfate and ethylenediamine are homogeneously and evenly distributed on the surface of silica without aggregation [20].



Spectrum: Acquisition

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
Carbon	K-series	7.91	7.48	12.22	2.84
Oxygen	K-series	46.33	43.80	53.73	7.08
Sodium	K-series	16.94	16.02	13.67	1.17
Aluminium	K-series	1.86	1.76	1.28	0.14
Silicon	K-series	14.33	13.55	9.47	0.66
Chlorine	K-series	18.39	17.39	9.62	0.68

Spectrum: Acquisition

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Error (1 Sigma) [wt.%]
Carbon	K-series	42.79	39.81	50.44	7.23
Nitrogen	K-series	7.01	6.52	7.09	2.32
Oxygen	K-series	37.17	34.58	32.89	6.00
Silicon	K-series	11.72	10.90	5.91	0.54
Sulfur	K-series	4.07	3.79	1.80	0.19
Chlorine	K-series	4.72	4.39	1.89	0.21

Figure 11. Energy Dispersive X-Ray Spectroscopy of a). silica, b). silica coated with sodium lauryl sulfate and ethylenediamine ligand

In the results of the Energy Dispersive X-Ray Spectroscopy spectrum of silica coated with sodium lauryl sulfate and ethylenediamine ligand (Figure 11), 10.90% silicon, 34.58% oxygen, 39.81% carbon, 3.79% sulfur, 6.52% nitrogen, 4.39% chlorine were obtained. EDX results showed an N spectrum of 6.52%, proving ethylenediamine as the source has been attached. Likewise, the C and S spectra were found to be 39.81% and 3.79%, respectively, indicating sodium lauryl sulfate was attached to silica. The presence of Cl is an imperfection in the washing process [21].

3.8. UV-Vis Analysis

UV-Vis analysis was used to determine the percentage concentration of Pb metal absorption before and after silica coating. The results of UV-Vis analysis include maximum wavelength, standard curve (regression equation), and concentration determination, such as Figure 12 and Table 1. From the research that has been done, the maximum wavelength of the PbNO standard solution3 obtained is 243 nm. The equation of the calibration curve of the $Pb(NO_3)_2$ standard solution comparison obtained is Y = 0.07032 x + 0.36468 with a correlation coefficient value (r) = 0.99578, making the standard curve of $Pb(NO_3)_2$ is done to fulfill the Lambert-Beer law, which is a straight line [20].

Figure 12. Absorbance Curve of Standard Solution

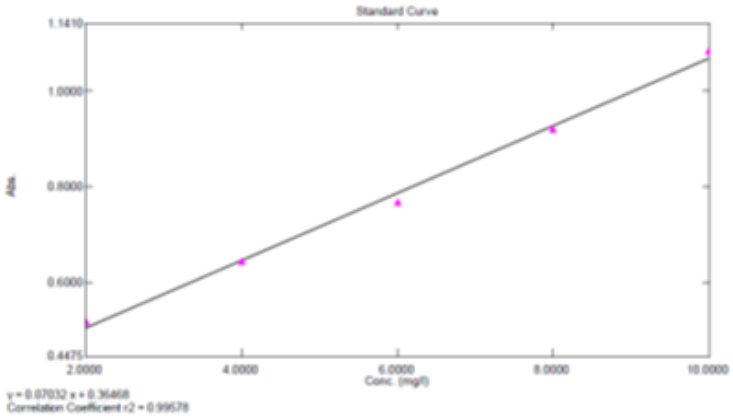


Table 1. Pressure Drop Sample

Sample	Concentration (ppm)	Absorbance
Standard Solution	2	0.5186
Standard Solution	4	0.6433
Standard Solution	6	0.7672
Standard Solution	8	0.9205
Standard Solution	10	1.0832

Standard $Pb(NO_3)_2$ solution used in the silica absorption process before and after the coating has a concentration of 10 ppm. After adsorption, concentration is reduced, as shown in Table 2.

Table 2. Pressure Drop Sample

Sample	Concentration (ppm)	Absorbance	Concentration after Absorbance
Silica	10	0.0471	4.5176 ppm
Coated Silica	10	0.2863	1.1146 ppm

Figure 13 shows SiO_2 with sodium lauryl sulfate coating and ethylenediamine ligand adsorbing lead metal ions; ethylenediamine plays a role in increasing the adsorption of lead metal ions. Lead metal ions bind physically and chemically; van der Walls bonds occur physically, and chemical bonds form complex compounds with ethylenediamine ligands [22].

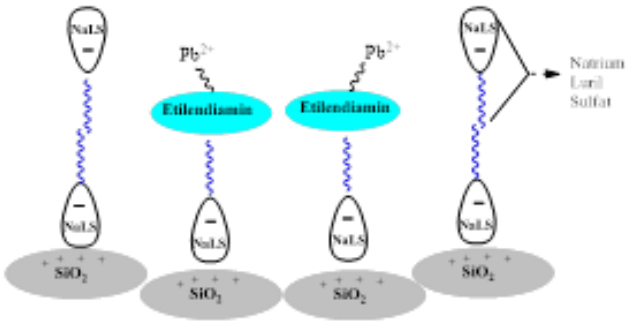


Figure 13. Illustration of NaLS coating and ligand en onto silica surface on the adsorption of metal ions

4. Conclusion

The success of silica coating is shown from the characterization results of FT-IR, XRD, and SEM-EDX analysis. FT-IR spectrum of silica shows the presence of functional groups Si-O-Si, Si-O-, and Si-OH. After coating, there is a change in the spectrum, which shows the presence of new functional groups, namely the S-O, C-N, CH_2, and NH functional groups. The results of characterization using XRD show a diffraction peak that widens at 2θ in the 27.6297 ° region, which indicates an amorph phase. After coating, diffraction peaks appear at the angle of 2θ in the areas of 19.62°, 20.47°, 21.04°, 25.56°, 26.50°, and 29.73° with high intensity, indicating increased criticism. The EDX spectrum of the silica shows that it was successfully synthesized with a silicon content of 13.55% and oxygen content of 43.80%. After coating, new contents such as carbon 39.81%, sulfur 3.79%, and nitrogen 6.52% appear. SEM results show that the silica is slightly oval and evenly round, with an average size of 100.788 nm. At the same time, silica-coated with sodium lauryl sulfate and ethylenediamine attached to the surface of silica with an average size of 85.3773 nm. Metal sorption by silica and silica materials coated with sodium lauryl sulfate and ethylenediamine ligands can reduce Pb^2 metal levels. With silica, there was a change in concentration from 10 ppm to 4.5162 ppm, while with coated silica, the concentration changed from 10 ppm to 1.1146 ppm.

5. Acknowledgements

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6. Conflict of Interest

Authors declare no conflicts of interest.

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