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FUNCTIONAL GROUP ANALYSIS OF SILICA GEL BASED ON RIVER SAND MAGNETIC MINERAL AS HEAVY METAL ABSORBANCE

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Abstract

Functional group analysis of silica gel synthesized from river sand magnetic minerals using the sol-gel method was conducted. River sand magnetic mineral samples were taken from Sompang River, Lombok. The synthesis method used is a sol-gel method with variation of HCl acid 10 M and NH4OH base 10 M. Morphological characterization using a Scanning Electron Microscope (SEM) and mineral content analysis using Energy Dispersive X-ray (EDX). The morphology of the resulting silica gel is granular, showing crystalline properties with a silica concentration of 77.99 ± 0.17 atomic per cent. Silica functional groups before absorption showed three stretching vibrations, namely OH group stretching vibrations in Si-OH and H2O at an absorption wavelength of 3669.77 cm-1, Si-O stretching vibrations from siloxane (Si-O-Si) at an absorption wavelength of 2858.51 cm-1 and Si-O asymmetric stretching vibrations from silanol (Si-OH) at an absorption wavelength of 962.88 cm-1. While the results of the analysis of silica gel after absorption show that almost all samples have vibrational absorption in the range of 400-500 cm-1. Other vibrational absorptions in silica samples used as water filters indicate that the bonding between nano-silica as an absorber and heavy metals is due to the magnetic properties of nano-silica

INTRODUCTION

Lombok Island is one of the islands in the province of West Nusa Tenggara. Lombok Island has many rivers. However, it has yet to be widely utilized by the surrounding community because many rivers on the island of Lombok are polluted. Polluted water can interfere with human health in everyday life [1]–[8].

The Government of the Republic of Indonesia also considers water pollution to be something that needs attention. The Indonesian government issued a policy on water pollution through the Regulation of the Minister of Health of the Republic of Indonesia (Permenkes RI) number 32 of 2017. Based on Permenkes RI No 32 of 2017, it is explained that water is polluted if the water exceeds the specified parameter threshold [9]–[11].

Water pollution parameters are divided into two, namely physical and chemical parameters. Physical parameters include total dissolved solution (TDS), conductivity, acidity and temperature [10].

Heavy metal content parameters include lead, manganese, iron, and copper. Significant manganese content is usually caused by water contaminated with laundry soap. While the content of lead and copper usually occurs in waters close to the dock. The high manganese and lead content near the dock is caused by the paint of passing ships [11].

There are many ways to reduce heavy metal content in water, such as absorption, ion exchange and precipitation. Absorption is the easiest method to reduce heavy metal content in water. Water purification by absorption method requires absorbent material. Widely used absorbent materials are iron and silica [1], [12].

Silica is a fascinating material as an absorption material because it can be synthesized from natural materials such as bagasse [13], rice husk [14], [15], bamboo [16], glass powder [17] and iron sand [1], [18], [19]. The application of nano-silica as an absorbent is an exciting study today due to its economical nature [20].

The use of iron sand magnetic material has been explained in the Qur'an Surah Al-Hadid verse 25. The verse explains that Allah SWT sends iron to humans because iron can benefit humans. What is meant by iron here is all forms of metal, including silica [21].

Many studies have been conducted on the synthesis of silica gel. Meiliyadi et al. have synthesized silica gel based on river sand magnetic minerals to Bramantya et al. synthesized silica aerogel from sea sand as an oil spill absorbent and produced nano-silica absorbents that can absorb an average oil of 13.98 g per mass of silica aerogel [22]. Meiliyadi et al. successfully synthesized nano-silica based on beach sand and river sand with varying concentrations of NH4OH. The results showed that the concentration of NH4OH influenced the particle size of nano-silica. The greater the concentration, the smaller the particle size [18].

However, there still needs to be research that analyzes the absorption method carried out by silica on heavy metals. Therefore, it is necessary to research the formation of other functional groups on silica gel after filtering using Fourier Transform Infrared (FTIR).

EXPERIMENTAL METHOD

This research was conducted at the Physics Laboratory of UIN Mataram. The nano-silica synthesis method used in this study was the sol-gel method. Mineral magnetic beach sand was taken from a distance of 2 m from the lip of the Sompang River, East Lombok [18]. In addition, the materials used are HCl with a concentration of 10 M Physical Analysis (99%) from Sigma Aldrich. NH4OH with a concentration of 10 M Physical Analysis (99%) and deionized water. The sampling location is shown in Figure 1.

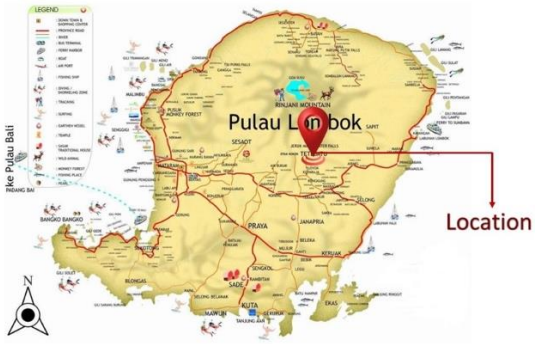


Figure 1. Sampling location

Mineral magnetic river and beach sand are then dried in the sun for 2 days to dry A permanent magnet separates mineral magnetic river sand and beach from impurities. Furthermore, washing is done using distilled water 5 times and dried. After washing, the magnetic minerals of river sand and beach sand are dried in an oven at 80 oC for 12 hours to remove the water content [24].

Purification of the river sand was carried out by immersing the sand in 1 litre of 10 M HCl solution for 20 hours to reduce other metal oxides contained in the sand. SiO2 is not reactive with HCl, so adding HCl will not reduce the SiO2 content in the sand but will reduce other metals. The

sample is washed with distilled water until it reaches a neutral pH of 7. The samples were filtered with filter paper and dried in an oven at 60 oC for 5 hours.

The next stage synthesises sodium silicate solution from 50 grams of purified sand. Sand samples that have been purified with 300 mL of NH4OH solution with a concentration variation of 10 M. The sample was heated using a hotplate magnetic stirrer with a temperature of 80 oC and a rotating speed of 1200 rpm to accelerate the reaction of sodium silica formation and to reduce precipitation. The sodium silica sample was filtered using filter paper to obtain a nano-silica gel precursor. The samples were then evaporated at 100 oC for 2 hours.

The morphology and mineral content of silica gel were characterized using SEM-EDX type jeol 700. Analysis of physical parameter characteristics was carried out using TDSmeter type COM3000. The chemical parameters (Pb and Mn metals) were analyzed using AAS-type Shimadzu AA-6200.

RESULT AND DISCUSSION

Characterization of the mineral content of silica gel using Energy Dispersive X-ray (EDX) analysis. Most SEM tools are equipped with energy-dispersive x-ray (EDX) capabilities to determine the chemical composition of the sample surface. The results of mineral content characterization using EDX are shown in Table 1.

Table 1. Mineral content of silica gel using EDX

Element	% atom
Na	3.57 ± 0.09
Mg	1.35 ± 0.05
Al	10.34 ± 0.12
Si	77.99 ± 0.17
K	0.39 ± 0.02
Ca	3.76 ± 0.08
Ti	0.70 ± 0.04
Fe	1.90 ± 0.08

Table 1 shows that the 7M beach sand sample contains Na, Mg, Al, Si, K, Ca, and Fe elements. The element that has the highest value of other elements contained in river sand samples is Silica (Si) 77.99 ± 0.17 %. EDX uses secondary electrons fired at the sample to determine the composition of the mineral content in the sample. The data read in the form of intensity peaks on each element, as shown in Figure 2.

Figure 2 shows the maximum intensities produced by each element due to the reflection of secondary electrons in EDX. Based on Figure 1, silica produces the maximum intensity, followed by oxygen. This indicates that the silica gel sample formed mainly consists of SiO2 material. The formed siO2 material can be amorphous or crystalline. Usually, crystalline SiO2 can be seen from the material's morphology in the form of grains [25]. Meanwhile, amorphous materials usually have a morphology in the form of fibres. Therefore, it is necessary to analyze morphology using SEM. The results of morphological analysis using SEM are shown in Figure 3.

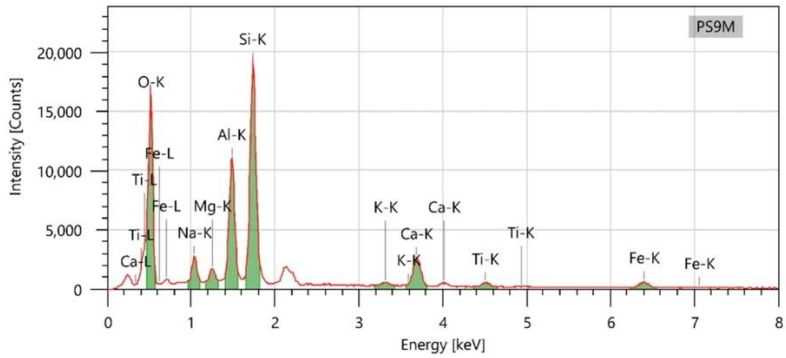
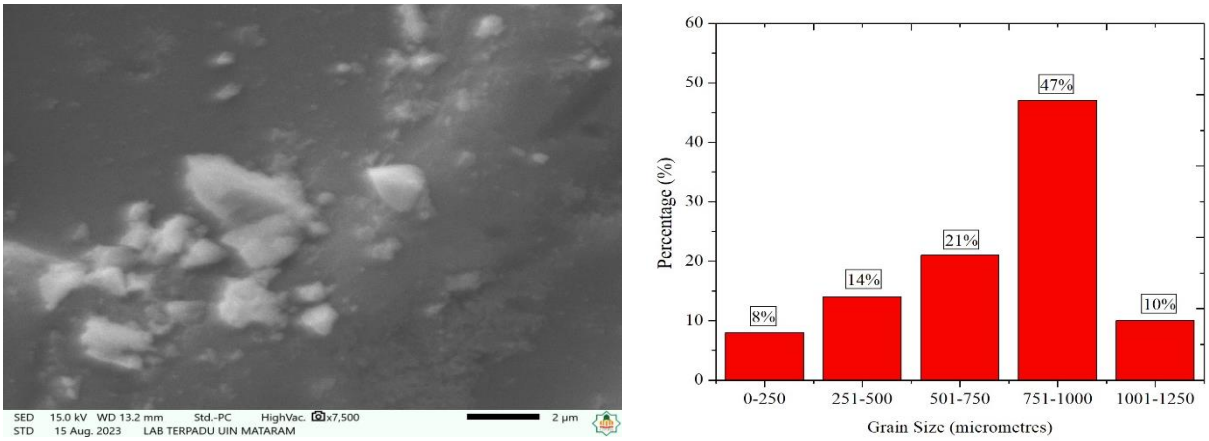


Figure 2. Maximum intensity of each element as a result of EDX analysis



(a) (b)

Figure 3. (a) morphology of silica gel using SEM with 2500 x magnification and (b) grain size distribution of silica gel particles.

Figure 3 shows the results of the morphological analysis of silica gel using SEM. The morphology of silica gel has formed in the form of granules. Morphology like this is a form of morphology owned by crystals, mainly in small clumps of solid in the form of small corals.

Table 2. Water quality before and after filtration

Parameters	Before	After	Differences
Physics Parameters			
Total Dissolved Solvent (ppm)	657.15	601.71	55.44
Conductivity (***μ***S / cm)	326.88	299.35	27.53
Chemical Parameters			
Pb (ppm)	0.0059	0.0047	0.0012
Mn (ppm)	0.0104	0.0091	0.0013

A scanning electron microscope (SEM) is a type of electron microscope that uses an electron beam to describe the surface shape of the sample being analyzed. SEM has a higher resolution than an optical microscope (OM). This is due to the de Broglie wavelength, which has shorter electrons than the OM wave. Because the smaller the wavelength used, the higher the microscope's resolution. SEM has a higher resolution than OM. The resolution that OM can produce is only 200 nm, while the resolution that SEM can produce reaches 0.1 - 0.2 nm. The working principle of SEM is to image the surface of an object or material with a beam of reflected electrons with high energy [19].

Silica has absorption capabilities due to its magnetic properties. A magnet will attract magnetic metals, such as copper, iron, lead and manganese. This is shown from the results of water quality analysis before and after filtration, as shown in Table 2.

Table 2 shows the level of water quality before and after filtration using river sand-based silica gel. Table 2 shows that the heavy metal content in the water will be reduced due to filtration. A reduction in the TDS value and water conductivity will also follow this. The percentage reduction of each parameter is shown in Figure 4.

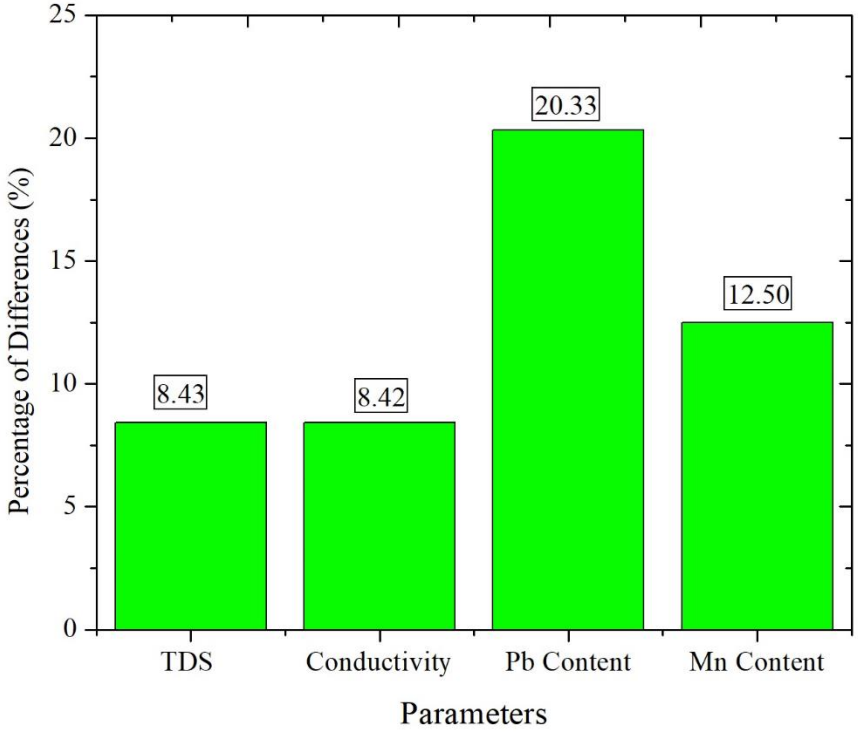


Figure 4. Changes in the values of both physical and chemical parameters before and after filtration using silica gel

Based on Figure 4, it can be seen that both physical parameters and chemical parameters have decreased. The decrease in conductivity and TDS values is due to decreased heavy metal content in the water after filtering. Metal is a material with good conductivity, so the smaller the heavy metal content in water is, the smaller the conductivity value of water will also be [26], [27].

The reduced value of metal content in water is due to silica gel filtering. The magnetic properties of silica gel result in a bond between silica and heavy metals absorbed during filtration. This is evidenced by the functional group analysis, as shown in Figure 5.

Figure 5 (a) shows the functional groups of silica gel (a) before absorption and (b) after absorption. Before absorption, it appears that there are 3 central bonds, namely OH group stretching vibrations in Si-OH and H2O at an absorption wavelength of 3669.77 cm-1, Si-O stretching vibrations from siloxane (Si-O-Si) at an absorption wavelength of 2858.51 cm-1 and Si-O asymmetric stretching vibrations from silanol (Si-OH) at an absorption wavelength of 962.88 cm-1.

Figure 5 (b) shows that almost all samples have vibrational absorption in the 400-500 cm-1 range. Indicates the presence of other vibrations in nano silica samples used as water filters. Indicates that the bond between nano-silica as an absorber and heavy metals is due to the magnetic properties of nanosilica [28].

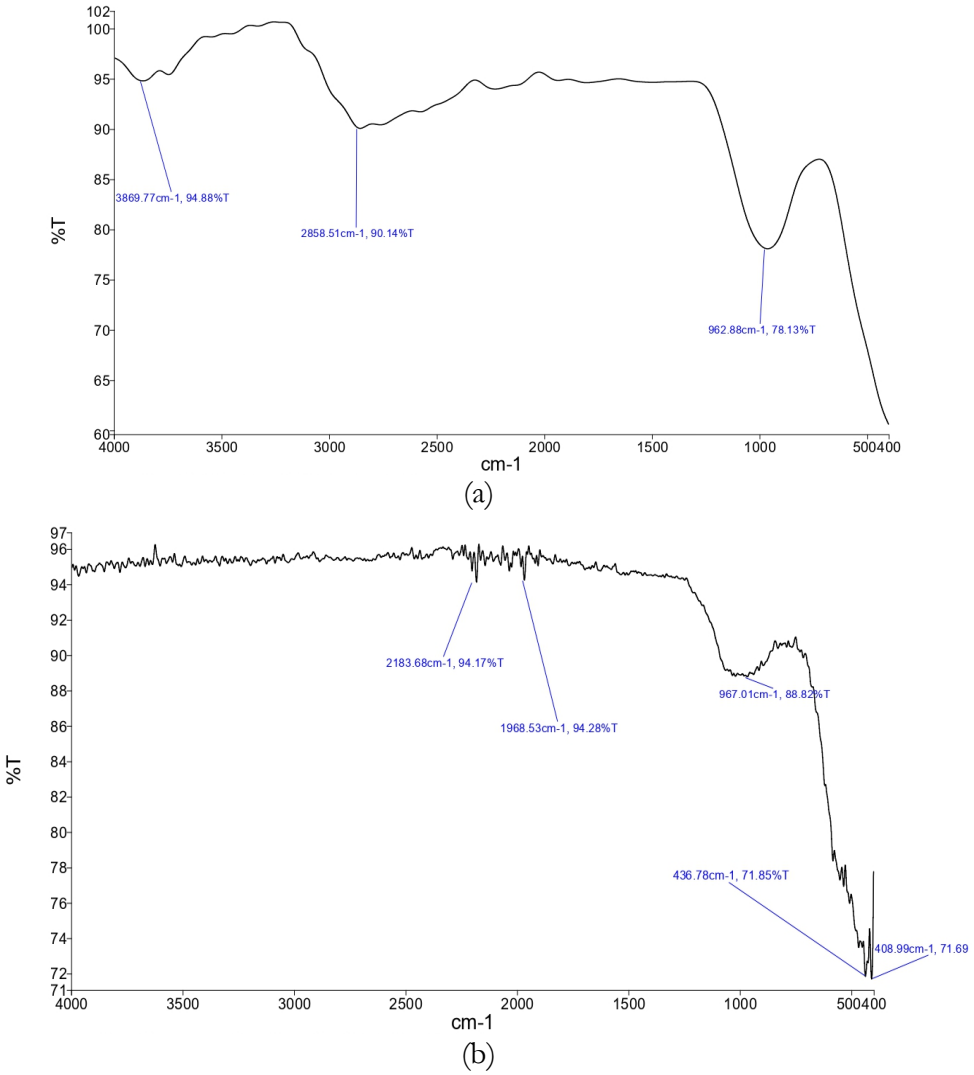


Figure 5. Functional group analysis of silica gel (a) before absorption and (b) after absorption

CONCLUSION

Based on the study's results, silica gel based on river sand magnetic minerals has been successfully synthesized using the sol-gel method. The resulting silica gel's morphology is granular, showing crystalline properties with a silica concentration of 77.99 ± 0.17 atomic per cent. The functional groups of silica before absorption showed three stretching vibrations, namely OH group stretching vibrations in Si-OH and H2O at an absorption wavelength of 3669.77 cm-1, Si-O stretching vibrations from siloxane (Si-O-Si) at an absorption wavelength of 2858.51 cm-1 and Si-O asymmetric stretching vibrations from silanol (Si-OH) at an absorption wavelength of 962.88 cm-1. The analysis of silica gel after absorption shows that almost all samples have vibrational absorption in the 400-500 cm-1 range. Indicates the presence of other vibrations in nano-silica samples used as water filters. Indicates that the bond between nano-silica as an absorber and heavy metals is due to the magnetic properties of nanosilica.

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