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Synthesis of Nanosilica Gel Based on River Sand and Its Use as Water Treatment

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Abstract: The synthesis of silica gel derived from magnetic minerals of Lombok Island river sand has been carried out, which is used as an adsorbent for heavy metals in water. The synthesis method used is the sol-gel method. Water samples came from the Babak River, located 40 m from Kebon Kongok landfill. Water quality tests include physical parameters (total dissolved solution (TDS), conductivity, pH, and temperature) and chemical parameters (manganese, copper, iron, and lead content). The results showed that water conductivity was reduced by 13.05%, TDS was decreased by 8.51%, and the pH of the water changed from acidic to neutral after filtering using silica gel. Based on chemical parameters, heavy metal content is reduced, with manganese content reduced by 30.50% and copper content reduced by 25.58%. Iron's most minor decrease occurred because Fe has the smallest atomic radius, so it can easily pass during filtration. The grain size of silica influences the screening process that occurs and is not affected by the magnetic properties of silica. However, in general, it is found that silica gel is very efficient to be used as a heavy metal adsorbent to improve water quality.

1. Introduction

Pollution is a phenomenon that often appears in people's lives. Water pollution is one type that often appears (Syuzita *et al.*, 2022). The existence of pollution in water can cause a lot of damage to humans and also to the surrounding environment. One of the pollution that often occurs is water pollution. The entry of unwanted substances into the water will cause pollution, so water quality will be disturbed and affect public health (Nurhidayati *et al.*, 2021).

Water quality requirements can be reviewed from physical and chemical parameters, regulated in Permenkes RI Number 416/Menkes/Per/IX/1990 concerning water requirements and quality (Syuzita *et al.*, 2022). Water quality based on physical parameters is indicated by TDS, pH, conductivity, and physical properties of water, such as odorless, colorless, and tasteless. Meanwhile, based on chemical parameters, unpolluted water is water that does not contain heavy metals above the specified threshold limit and is not toxic (Sadashiva *et al.*, 2023). If the water is found to have physical and chemical properties above the specified threshold limit, the water is said to be polluted (Nurhidayati *et al.*, 2021). Some treatment methods to improve water quality include adsorption neutralization, biosorption, ion exchange, and precipitation (Awodi *et al.*, 2023; Bakri & Yushananta, 2023). The adsorption process is a recommended method to improve water quality. In the process, adsorption involves chemical bonds, ion exchange, and attractive forces between molecules (Hardyanti *et al.*, 2023). The process of improving water quality in absorption involves adsorbent materials. Some materials that have been used include zeolite (Zhou *et al.*, 2022), bentonite (Hardyanti *et al.*, 2023), kaolin (Zhang *et al.*, 2022), sargassum (Song *et al.*, 2022), and nano-silica (Liu *et al.*, 2022).

Nanomaterials received more attention of researchers and inductrials that have been found to be effective in elimination of several pollutants from wastewater such as heavy metals, organic and inorganic solvents (Aaddouz *et al.*, 2023). amorphous silica has small pores so it has a large absorption cross-sectional area. large absorption cross-sectional area is very effective in absorption due to its large absorption field (Ershad *et al.*, 2021).

Nanosilica is an exciting material as absorption material because it can be synthesized from natural materials such as bagasse (Panyo *et al.*, 2023), rice husk (Han et al., 2022; Jyoti et al., 2021), bamboo (O. Olawale, 2020), glass powder (Tran *et al.*, 2022) and iron sand (Meiliyadi *et al.*, 2022). The application of nano-silica as an absorbent is an exciting study today due to its economical nature (Liu *et al.*, 2022). 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 sent down iron to humans because iron can benefit humans. What is meant by iron here is all forms of metal, including nano-silica.

Previous research on synthesizing and utilizing nano-silica based on iron sand magnetic minerals has been carried out. Ananda & Aini (2021) have synthesized mesoporous nano silica based on magnetic sand nano silica by sol-gel method. Meiliyadi *et al.*, (2022) successfully synthesized nano-silica based on beach sand and river sand with varying concentrations of NH_4OH. The results showed that the concentration of NH_4OH influenced the particle size of nano-silica. The greater the attention, the smaller the particle size (Meiliyadi *et al.*, 2022). The sol-gel technique is mainly used to the synthesis of various of kind of compounds (Navas *et al.*, 2022; Maarouf *et al.*, 2021).

However, from previous studies, the utilization of nano-silica based on beach sand and river sand as a heavy metal adsorbent to improve water quality has not been carried out. At the same time, nanosilica is a magnetic material that can bind heavy metals from polluted water. Therefore, it is necessary to study and research the characteristics of the absorbance properties of nanosilica in gel form based on Lombok Island river sand used as a heavy metal adsorbent.

2. Methodology

2.1 Preparation of Nanosilica Gel

Purification of river sand is done by soaking the sand in 1 liter of 10 M HCl solution for 20 hours to reduce other metal oxides contained in the sand. SiO_2 is not reactive with HCl, so adding HCl will not lessen the SiO_2 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 at 60 ^oC for 5 hours in the oven (Meiliyadi *et al.*, 2022). The research scheme is shown in **Figure 1.**

The next step is synthesizing sodium nanosilicate solution from 50 grams of purified sand. Sand samples that have been purified with 100 mL of NaOH solution with a concentration of 9 M. The model was heated using a hotplate magnetic stirrer with a temperature of 80 ^oC and a rotating speed of 1000 rpm to accelerate the reaction of sodium nanosilica formation and to reduce precipitation. The sodium nanosilica sample was filtered using filter paper to obtain a nano-silica gel precursor.

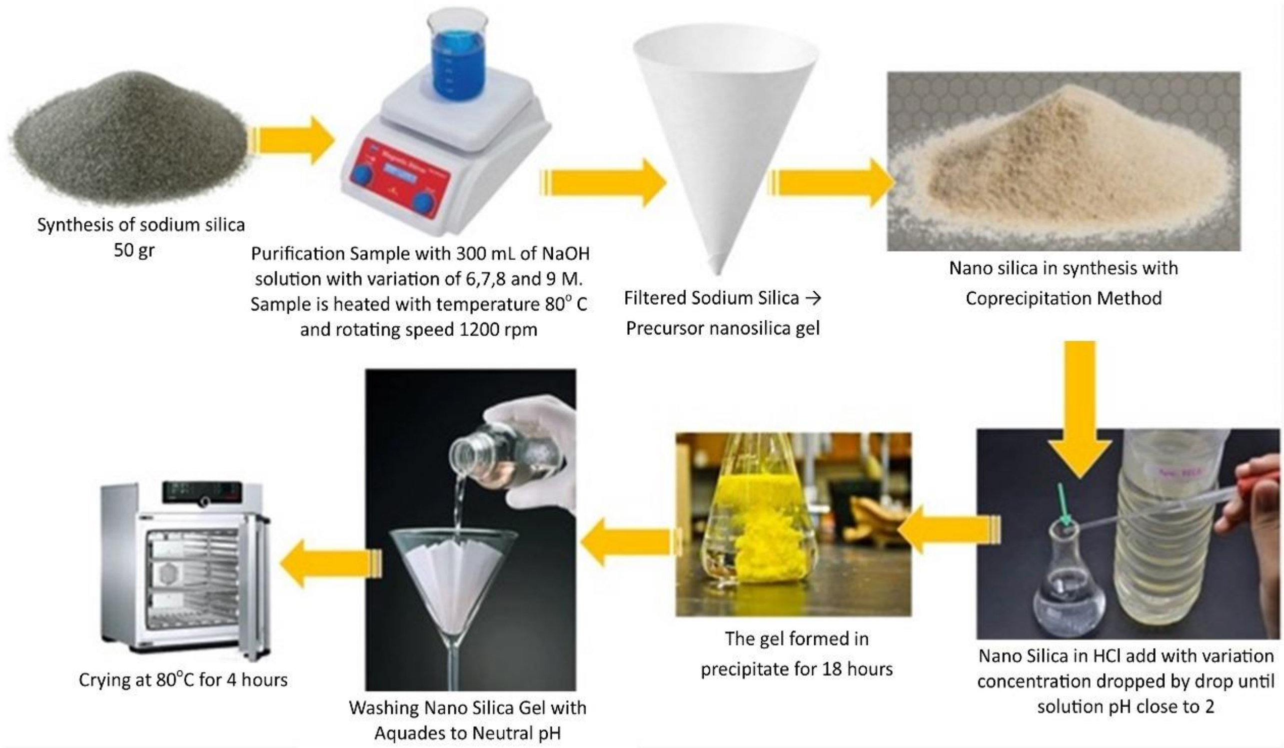


Figure 1. Research scheme of Nanosilica Gel Synthesis

Nanonanosilica gel was then synthesized using the coprecipitation method. Precursor nano-silica gel was measured for pH first to get the initial pH value. The sample was then added with 9 M HCl drop by drop while stirring until a gel was formed. After the gel is created, the solution is still dripped with HCl without going until the pH of the sample is close to 1. The gel formed is then deposited for 18 hours before being filtered. The gel is then washed with distilled water until the pH is neutral and removes the NaCl formed from the reaction of $NaSiO_2$ and HCl. The samples were dried at 80 ^{o}C for 4 hours in the oven (Yu *et al.*, 2022).

2.2 Characterisation of nano-silica gel

Characterization of nano-silica gel morphology using Scanning Electron Microscope while analyzing its mineral content using Energy Dispersive X-Ray (EDX) type Jeol 700—vibration between nanosilica atoms and other metals using Fourier Transform Infra-Red Spectroscopy (FTIR) (Sumari *et al.*, 2023).

2.3 Water quality based on physics parameters

Physical water quality parameters include temperature, pH, Total Dissolved Solution (TDS), and Conductivity. The four quantities were measured using a TDS meter (Mengstie *et al.*, 2023; Moeinzadeh *et al.*, 2023; Nurhidayati *et al.*, 2021).

2.4 Water quality based on chemical parameters

Chemical parameters include the content of heavy metals contained in water, including iron (Fe), copper (Cu), lead (Pb), and manganese (Mn). Mineral content analysis using Atomic Absorption Spectroscopy (AAS) (Syuzita *et al.*, 2022).

2.4 Treatment of Wastewater

Two grams of nano-silica gel was added to the water sample. Water samples that have been treated are then measured for quality based on physical parameters (Temperature, pH, TDS, and Conductivity) and chemical parameters (Awodi et al., 2023). The synthesized nano-silica sample used is shown in **Figure 2**.

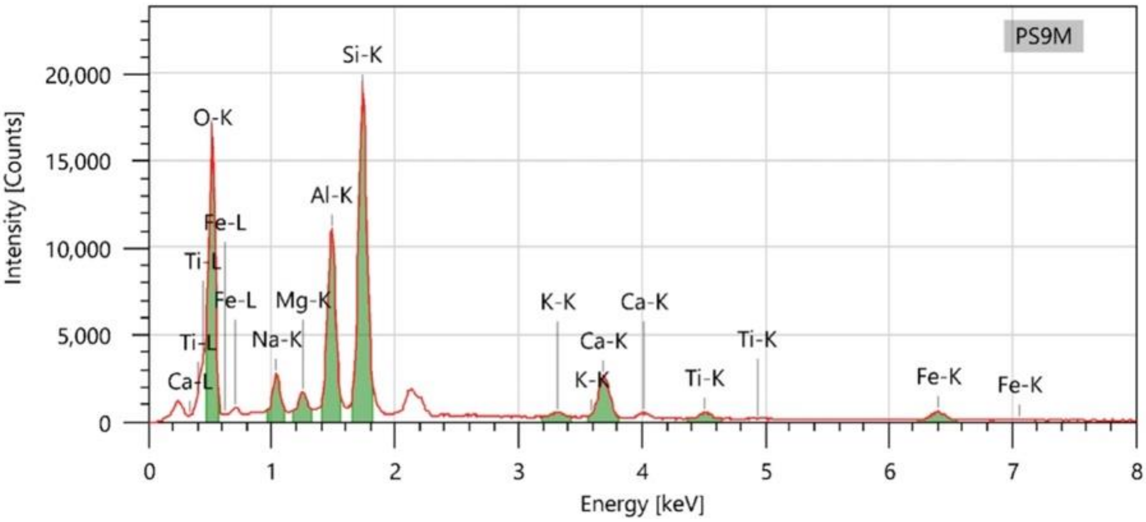
Figure 2. Nano-silica gel based on river sand

The water samples were taken in the Babak River adjacent to the Kebon Kongok landfill. The water was taken at a distance of 40 m from the landfill. This position was taken because of the location of the river water, which has the closest space to the kebon kongok landfill.

3. Results and Discussion

3.1 Slika Gel Mineral Content

Characterization of the mineral content of nano-silica gel using Energy Dispersive X-ray (EDX) analysis. The spectrum of EDX analysis results is shown in **Figure 3**.



Figures 3. Spektrum analisis EDX silica gel berbasis pasir sungai

Figure 3 shows the spectrum of silica gel analysis using EDX. EDX is used to determine the chemical composition of the sample surface, and most SEM tools are equipped with energy-dispersive X-ray

(EDX) capabilities. EDX is generated from X-rays by firing X-rays at the position you want to know the composition. After firing at the desired position, specific peaks representing an element contained will appear. EDX can also create elemental mapping by giving different colors to each part on the sample's surface. EDX can be used to analyze the percentage of each element quantitatively. SEM-EDX can provide information about the analyzed sample's topography, morphology, and composition. Topography is the ability to interpret surface and texture. Morphology is the ability to analyze the shape and size of the sample. Composition is the ability to analyze the composition of the object's surface quantitatively and qualitatively (Didik et al., 2021). The mineral content of nano-silica gel is shown in **Table 1**.

Table 1. Mineral content silica gel based on river sand

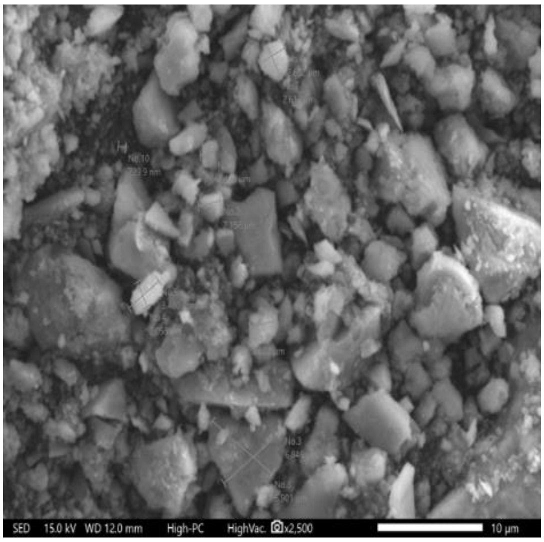
S/N	Element	%Atomic (%)
1	Sodium	3.35±0.14
2	Magnesium	1.64±0.09
3	Aluminum	11.09±0.19
4	Silicon	72.90±0.28
5	Potassium	1.53±0.07
6	Calcium	3.20±0.11
7	Titanium	1.03±0.07
8	Ferum	5.35±0.20

After conducting research using SEM, it can be seen that several contents exist in the 9 M river sand sample above. The content is in the form of several elements, including Na, Mg, Al, Si, K, Ca, Ti, and Fe. The highest mineral content in this river sand sample is Nanosilica (Si) (72.90 ± 0.28)%.
Based on Table 2, it can be seen that silica is the compound with the largest percentage. this indicates that the nanosilica sample has been formed. Silica is a chemical compound with the molecular formula SiO2 (silicon dioxide) which can be obtained from mineral, vegetable and crystal synthesis silica. Mineral silica is a compound that is commonly found in mining/excavation materials in the form of minerals such as quartz sand, granite, and fledsfar which contain silica crystals (SiO_2).
Silica gel is a form of silica produced by agglomerating sodium silicate sol ($NaSiO_2$) (Meiliyadi *et al.*, 2022). Silicate solution reacted with acid will produce silicic acid. In silicic acid systems, gel formation is described as the condensation of $Si(OH)_4$ into siloxane chains, which then branch and further crosslink to form a three-dimensional framework. These agar-like sols can be dehydrated to turn into inelastic glass-like solids or granules (Liu *et al.*, 2022). Silica gel is shaped like a soft and spongy solid but a certain temperature range can be shaped like a fluid. Usually the gel has the property of thixotropy, that is, it becomes liquid when shaken and re-solidifies when left calm.

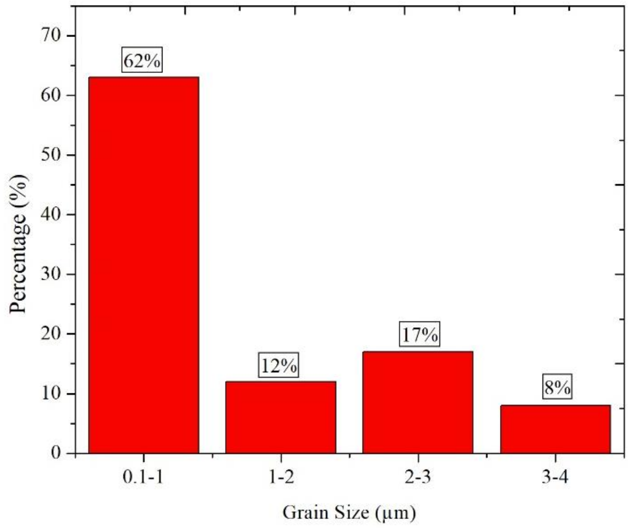
3.2 Morphological Characteristics of Silica Gel

The morphology of nano-silica gel was analyzed using a Jeol 70-type Scanning Electron Microscope (SEM). The detector in the SEM detects the reflected electrons and determines the location of the reflected beam with the highest intensity. The direction gives information on the surface profile of the object. SEM is a type of electron microscope that uses electron beams to draw the surface profile of things (Garcia de Abajo *et al.*, 2021; Ben Hmamou *et al.*, 2012). The morphology analysis using SEM is shown in **Figure 4**.

Figure 4 (a) shows the morphology of nano-silica gel synthesized using the sol-gel method. The morphology of nano-silica gel appears to be granular, with the grain size shown in **Figure 4 (b)**. Based on **Figure 5(b),** the largest grain size of silica gel is on the order of 0.1-1 micrometers, indicating that the samples produced are already on the order of nanometers. The average grain size of nano-silica gel is 988.82 nm.



(a)



(b)

Figure 4. Morphological Characteristics of Silica based on Coastal Sand (a) SEM Results with 2500x Magnification, (b) Percentage Distribution of Grain Sizes of Costal Iron Sand

SEM is an electron microscope that uses electron beams to draw surface profiles. The working principle of SEM is to shoot the surface of an object with a high-energy electron beam. The object's surface subjected to the shaft will reflect the beam or produce secondary electrons in all directions. But there is one direction where the beam is reflected with the highest intensity. The detector inside the SEM detects the reflected electrons and determines the location of the highest-intensity reflected beam. This direction provides information on the surface profile of the object (Didik et al., 2021).

When an electron beam hits a solid specimen, several interactions occur that can provide information about the structure of the material. Some of the shaft is dissipated, and some penetrates the sample. The interaction between scattering electrons and atoms in the sample results in the release of low-energy electrons, X-ray photons, and Augger electrons, all of which can be utilized to characterize the material, especially the material's morphology (Meiliyadi et al., 2022; Shaheen et al., 2022).

3.3 Water quality before and after treatment based on physical and chemical parameters

After gel filtering, the water sample is examined for quality based on physical parameters. The water samples and after filtering are shown in **Figure 5.** After filtering the water sample, its quality was then measured based on physical parameters, including temperature, pH, conductivity, and TDS, and chemical parameters, including iron (Fe), copper (Cu), lead (Pb), and manganese (Mn) content (Syuzita et al., 2022). **Table 2** shows that there is a quality improvement. Decrease in the value of both physical and chemical water quality parameters. Temperature can be considered not to have decreased because the matter is still around 31 ^oC. The acidity level has a significant change in the water sample. Water samples that initially had high acidity (low pH) became neutral (pH between 6.5-7). The acid content is reduced due to filtering, which can be caused by the reduced value of heavy metal in water. The magnitude of the decrease in physical values can be seen in **Figure 6**.

(a) (b)

Figure 5. Water sample (a) before treatment by using silica, (b) after treatment by using silica

Table 2. Water quality before and after treatment

No.	Element	Unit	Water quality		
			Before treatment	After treatment	Different (%)
	Physical parameters				
1	TDS	ppm	527.10	482.23	8.51
2	conductivity	µS/cm	687.12	597.42	13.05
3	Temperature	ºC	31.90	31.50	1.25
4	pH	no unit	5.41	6.80	20.44
	Chemical Parameters				
1	Fe Content	ppm	0.2035	0,1797	11.70
2	Cu Content	ppm	0.0043	0.0032	25.58
3	Pb Content	ppm	0.0221	0.0183	17.19
4	Mn Content	ppm	0.0141	0.0098	30.50

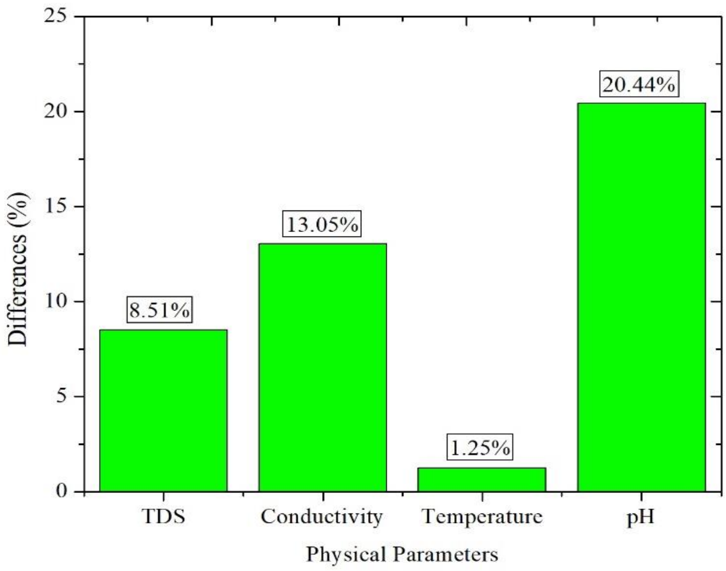


Figure 6. Differences in water quality before and after treatment based on physical parameters

Based on **Figure 6**, pH has the most considerable percentage change. Conductivity and TDS also have significant differences. The amount of TDS and conductivity is influenced by the amount of metal content in the water—the reduced value of heavy metal content in water, especially conductive heavy metals such as copper. The decrease in the value of chemical parameters is shown in **Figure 7**. Based on **Figure 7**, it can be seen that Mn has a large percentage of metal absorption, accompanied by Cu. In contrast, Fe has the smallest absorption percentage because Fe has the smallest atomic radius, so it can easily pass during the screening process. Shows that the grain size of silica influences the screening process that occurs and is not affected by the magnetic properties of silica (Rianto et al., 2018; Meiliyadi et al., 2022; Shaheen et al., 2022). Proses adsorpsi merupakan teknik pemurnian dan pemisahan yang efektif dipakai dalam industri untuk mengurangi ion logam berat dalam air limbah. Silika memiliki gugus aktif Si-O-Si dan Si-OH yang dapat digunakan sebagai adsorben logam Fe (Hardyanti et al., 2023). Permukaan silika bermuatan negatif, sehingga mampu mengadsorpsi ion-ion logam yang bermuatan positif seperti Fe, Mn, Cu dan Pb.

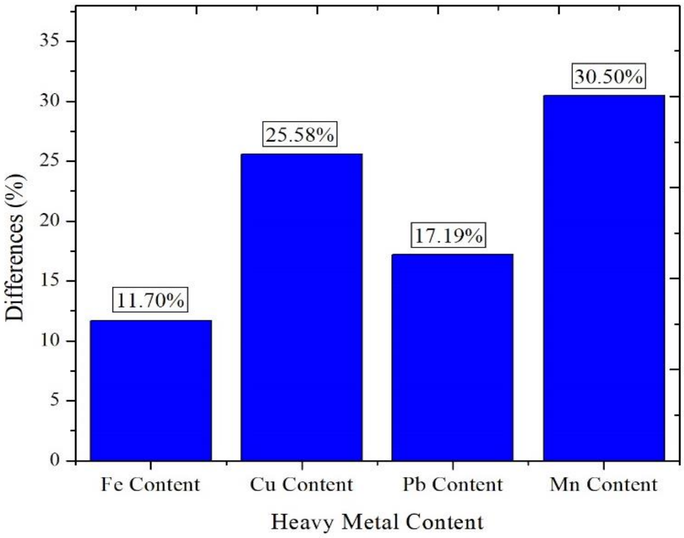


Figure 7. Differences in water quality before and after treatment based on chemical parameters

Conclusion

This study uses silica derived from river sand magnetic minerals and synthesized using the sol-gel method as a water treatment. Water quality tests were conducted based on physical parameters and chemical parameters. Based on physical parameters, it was found that the conductivity of water was reduced by 13.05%, TDS was decreased by 8.51%, and the pH of water, which was initially acidic, became neutral after filtering using silica gel. Based on chemical parameters, heavy metal content is reduced, with manganese content reduced by 30.50% and copper content reduced by 25.58%. Iron's most minor decrease occurred because Fe has the smallest atomic radius, so it can easily pass during filtration. The grain size of silica influences the screening process that occurs and is not affected by the magnetic properties of silica. However, in general, it is found that silica gel is very efficient to be used as a heavy metal adsorbent to improve water quality.

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Compliance with Ethical Standards: This article contains no studies involving human or animal subjects

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