p-ISSN : 2302-4216
e-ISSN : 2830-1765
Inovasi Fisika Indonesia
https://ejournal.unesa.ac.id/index.php/inovasi-fisika-indonesia
Published by:
Program Studi Fisika
Jurusan Fisika FMIPA
Universitas Negeri Surabaya
Kampus Unesa Ketintang Jl. Ketintang Gd. C3 Lt. 1
Surabaya - Indonesia
jifi@unesa.ac.id

Jurnal Inovasi Fisika Indonesia (IFI) Volume 15 Nomor 3 Tahun 2026, hal 401-409

ADSORPTION OF HYDROGEN SULFIDE (H_2S) IN OIL AND GAS INDUSTRY WASTEWATER USING HCL-ACTIVATED NATURAL ZEOLITE

[1)]Widya Ningrum, [2)] Nugrahani Primary Putri, [3)] Evi Suaebah

[1)] Department of Physics, Universitas Negeri Surabaya, Surabaya, Indonesia,
E-mail: widya.22075@mhs.unesa.ac.id
[1)] Department of Physics, Universitas Negeri Surabaya, Surabaya, Indonesia,
E-mail: nugrahaniprimary@unesa.ac.id
[1)] Department of Physics, Universitas Negeri Surabaya, Surabaya, Indonesia,
E-mail: evisuaebah@ unesa.ac.id

Abstrack

Dissolved sulfide is a corrosive contaminant commonly found in oil and gas industry wastewater. One treatment method that can be used to reduce sulfide content is adsorption using porous materials such as natural zeolite that has been modified through acid activation. This study aims to evaluate the effect of adsorbent mass and particle size of 1 M HCl-activated natural zeolite on the efficiency of sulfide removal from oil and gas industry wastewater. The adsorbent masses tested were 10, 20, 30, 40, and 50 g, while the particle sizes tested were 4, 10, and 40 mesh. The adsorption process was conducted at room temperature, with the final sulfide concentration measured using the iodometric titration method. The results showed that an increase in adsorbent mass and a decrease in particle size led to an increase in sulfide removal efficiency. The best conditions tested were achieved with an adsorbent mass of 50 g and a particle size of 4 mesh, resulting in a removal efficiency of 89.10%. The improvement in adsorption performance is related to the increased contact area between the adsorbent and the adsorbate due to the increase in mass and the reduction in zeolite particle size.

Keywords: zeolite, hydrogen sulfide (H_2S), adsorption, oil and gas wastewater.

I. INTRODUCTION

Fossil fuels, particularly oil and gas, remain the primary energy source for various applications, including the industrial sector, transportation, power generation, and households [1]. Indonesia's vast oil and gas resources have driven exploration, exploitation, and the processing of gas and oil. One example is the Cepu Oil and Gas Training Center (PPSDM Migas Cepu), the site of Indonesia's first oil well. However, oil and gas exploration and production activities generate various types of waste. These wastes originate from a number of activities, such as production, drilling, and re-drilling, as well as gas facilities and laboratories [2]. These wastes generally contain hydrocarbon and carbon compounds such as benzene, toluene, ethylbenzene, and xylene, along with phenolic compounds and hydrogen sulfide (H_2S).

Hydrogen sulfide (H_2S) is a gas produced by the decomposition of organic matter by bacteria in an oxygen-free oxygen [2]. This gas is colorless, hazardous, soluble in water, and has a distinctive rotten-egg odor [3]. At high concentrations above 1,000 ppm, H_2S gas can cause chemical asphyxia by inhibiting the enzyme cytochrome c oxidase, which disrupts oxygen transport in the body, leading to hypoxia and ultimately death.

Petroleum waste is considered hazardous if discharged directly into water bodies because it contains risky and toxic chemicals [4]. Results of an analysis conducted by the Bojonegoro Regency Environmental Agency

In 2025, the Bengawan Solo River was observed to have a blackish-brown color, was turbid, and was foamy due to oil waste pollution [9]. This wastewater threatens the survival of aquatic organisms. For this

reason, the government has established wastewater quality standards that must be met before wastewater is discharged into the environment to prevent damage to the ecosystem's balance [10]. Therefore, wastewater treatment is essential before discharge into the environment.

Waste treatment processes can utilize various methods such as coagulation [5], electrocoagulation [6], photocatalysis [7], filtration, and adsorption [8]. Among these, coagulation, electrocoagulation, and photocatalysis require high energy input, involve excessive use of chemicals, and exhibit reduced efficiency at low concentrations. The adsorption method is often used to treat wastewater because it is efficient, simple, and capable of absorbing pollutants without producing hazardous byproducts [9]. Adsorption is a process in which molecules of a dissolved substance gather and attach to the surface of an adsorbent [10]. Various materials are used as adsorbents, such as fly ash [11], activated carbon [12], coconut fiber [13], and coconut shell charcoal [14]. However, these adsorbent materials have limited efficiency and stability. In addition, inorganic materials such as zeolites are also used in the adsorption process due to their abundant availability, chemical stability, and physical properties that support the absorption of contaminant molecules.

Zeolites are aluminosilicate minerals with a three-dimensional crystalline structure that forms cavities and microporous channels. This porous structure, combined with a negative charge on the aluminosilicate framework that is balanced by cations, gives zeolites high ion-exchange capacity and affinity for polar molecules such as H_2S. In the H_2S adsorption process, adsorption occurs through electrostatic interactions and chemical reactions between H_2S and metal cations within the zeolite cavities, resulting in stable metal sulfide compounds and enabling regeneration without structural degradation [15].

However, before use, zeolite requires an activation process because it still contains mineral impurities and clogged pores, so its adsorption capacity is not yet optimal. The purpose of activation is to increase the specific surface area, open the pores, and improve its adsorption properties [16]. Various activation methods can be used, such as heating, treatment with a base (NaOH) [17], and acid activation [18]. Activation using HCl was chosen because it effectively removes interfering metal ions and increases the number of active sites compatible with the acidic nature of H_2S, thereby enhancing the interaction and adsorption capacity for H_2S [18].

Therefore, this study was conducted using activated zeolite as an H_2S adsorbent to enhance the effectiveness of H_2S adsorption. A previous study by Georgiadis et al. (2021) [18] employed iodometric titration to evaluate H_2S adsorption capacity across various zeolite types and demonstrated that zeolites possess significant adsorption potential as well as good thermal and chemical stability. Accordingly, this study employed the iodometric titration method, taking into account the variables of adsorbent mass, particle size, and pH to determine the optimal conditions for the adsorption mechanism of sulfides by activated zeolite.

II. METHODS

A. Study Design

This study aims to analyze the adsorption of sulfide (H_2S) in oil and gas industry wastewater using HCl-activated natural zeolite. The research stages include zeolite preparation, zeolite activation, the adsorption process with variations in zeolite mass and particle size, and the analysis of sulfide concentration sulfide concentration using the iodometric method. Waste samples were collected using sealed containers and analyzed within less than 24 hours to maintain the stability of the sulfide.

B. Operational Variables of the Study

Control variables included sulfide volume, stirring time, stirring speed, operating temperature, and settling time. Independent variables consisted of zeolite mass at 10 g, 20 g, 30 g, 40 g, and 50 g, as well as zeolite particle sizes of 4, 10, and 40 mesh. Dependent variables were final sulfide concentration (mg/L) and removal efficiency (%).

C. Data Collection Method

This study used zeolite obtained from a local supplier in Indonesia. Homogenization was performed by grinding with a mortar and pestle, followed by sieving using 40-mesh, 10-mesh, and 4-mesh sieves. The

samples used were the fractions that passed through each sieve. Activation was performed chemically using a 1 M HCl solution. A total of 90 g of zeolite from each fraction size was added to 50 mL of distilled water and 1 mL of 1 M HCl. The mixture was stirred using a magnetic stirrer at a speed of 300 rpm for 30 minutes. After activation, the solution was washed twice with 100 mL of distilled water until the pH reached neutral. The solid residue from the washing was filtered using Whatman No. 42 filter paper and then dried in a furnace at 500°C for 3 hours. After cooling, the activated zeolite was stored in a sealed polyethylene container to prevent contamination by atmospheric moisture prior to use in the adsorption testing phase.

The adsorption test used 500 mL of waste sample from the PPSDM Migas Cepu refinery in each experiment. The zeolite was added to a glass beaker containing the waste sample, then stirred using a flocculator at a speed of 120 rpm for 30 minutes at a temperature of 30 °C. After the stirring process was complete, the mixture was allowed to stand for 10 minutes so that the solid particles could settle naturally; the filtrate was then separated to determine the final H_2S concentration. The H_2S concentration was determined using the iodometric titration method in accordance with SNI 6989.75:2009. Zinc acetate solution was added to the filtrate sample was treated with zinc acetate solution to bind the H_2S, then reacted with an excess of iodine (I_2) solution in an acidic environment. The residual iodine is titrated using a sodium thiosulfate ($Na_2S_2O_3$) solution until the endpoint, marked by the disappearance of the blue color following the addition of an amylose indicator. The H_2S concentration is calculated based on the difference between the initial amount of iodine and the residual iodine.

D. Data Processing Techniques

The effectiveness of the adsorption process is determined based on the decrease in H_2S concentration before and after treatment using the following equation:

$$\eta = \frac{C_0 - C_t}{C_0} \, x \, 100\% \quad (1)$$

Each experiment was conducted three times to obtain the mean and standard deviation and to reduce measurement errors. Another parameter considered in this study was the final pH, which supports the success of the adsorption process. After treatment, there was an increase

III. RESULTS AND DISCUSSION

A. Materials and Adsorbent Preparation

This study utilized zeolite as the adsorbent material. Preparation involved grinding and sieving to obtain a uniform particle size. Once a uniform size was achieved, the zeolite was washed with distilled water until the pH approached neutral to remove impurities, dust, and dissolved ions that could interfere with the adsorption process. The solid resulting from this washing process was then dried in an oven to remove residual water from the pore structure. The washings were filtered and dried to remove the water trapped within the zeolite pores during the washing process.

Zeolite activation is performed to modify the surface properties of the adsorbent and reduce the content of impurities covering the pores. The addition of hydrochloric acid aims to dissolve some of the impurity components present on the zeolite surface, thereby increasing the availability of active sites. Activation is carried out by soaking the zeolite in a 1 M HCl solution, which is then stirred using a magnetic stirrer.

$$\text{Zeolite} + \text{HCl} \rightarrow \text{Zeolite} + M+ + Cl- \quad (2)$$

where M^+ is an alkali or alkaline earth metal ion naturally present in the zeolite. After the mixture has been soaked, it is filtered and washed until the pH of the filtrate becomes neutral. The that has been neutralized is then calcined at 400 °C for 3 hours to remove any remaining water. The color change in the zeolite—from dark brown to a lighter, grayish hue—indicates the removal of some metal impurities and organic compounds. This is consistent with research by Rahmayani et al. on the increase in the Si/Al ratio of zeolite, where the color of natural zeolite—which was initially dark brown

before activation—became lighter after activation and calcination due to the loss of water bound within the zeolite structure.

B. Effect of Zeolite Mass on Adsorption Efficiency

The determination of H_2S concentration is based on the principle of redox reactions in the iodometric method, in which H_2S reacts with iodine to form sulfur and iodide ions according to the reaction:

$$H_2S + I_2 \rightarrow 2HI + S \quad (3)$$

where the remaining I_2 is then back-titrated using $Na_2S_2O_3$ according to the reaction:

$$I_2 + 2S_2O_32- \rightarrow 2I- + S4O2- \quad (4)$$

The decrease in the amount of iodine titrated indicates that some of the iodine has reacted with H_2S, so it can be used to determine the concentration of sulfide in the sample. The smaller the volume of thiosulfate used, the higher the concentration of sulfide that reacts in the system. The initial sulfide concentration was determined from a control sample without adsorbent and was found to be 18.73 (mg/L).

To provide a more comprehensive picture of the effect of mass on concentration and adsorption efficiency based on iodometric titration analysis, the results are presented in **Table 1**

Table 1. Experimental Results: Effect of Mass on Adsorption Efficiency

Mass (g)	Particl e Size (mesh)	Final H_2S Concentr ation (mg/L)	Adsorption Efficiency (%)
	40	3.72 ± 0.10	**80.12 ± 0.43**
10	10	7.43 ± 0.14	**60.34 ± 0.35**
	4	9.26 ± 0.16	**50.56 ± 0.45**
	40	3.29 ± 0.08	**82.45 ± 0.44**
20	10	7.02 ± 0.08	**62.50 ± 0.41**
	4	9.03 ± 0.14	**51.80 ± 0.55**
	40	2.87 ± 0.08	**84.70 ± 0.55**
30	10	6.58 ± 0.13	**64.85 ± 0.55**
	4	8.80 ± 0.12	**53.02 ± 0.45**
	40	2.44 ± 0.09	**86.95 ± 0.42**
40	10	6.14 ± 0.13	**67.20 ± 0.42**
	4	8.60 ± 0.12	**54.10 ± 0.37**
	40	2.04 ±0.09	**89.10 ± 0.43**
50	10	5.72 ± 0.13	**69.45 ± 0.43**
	4	**8.39 ± 0.14**	**55.20 ± 0.36**

As the mass of zeolite increases, the efficiency of the adsorption process also increases due to a rise in the number of active sites capable of interacting with sulfide molecules. This finding is consistent with research conducted by Asilian H. et al. [20], which demonstrated a positive correlation between adsorbent mass and sulfide adsorption capacity. The results showing the effect of increasing zeolite mass on the reduction of sulfide concentration are presented in Figure 1.

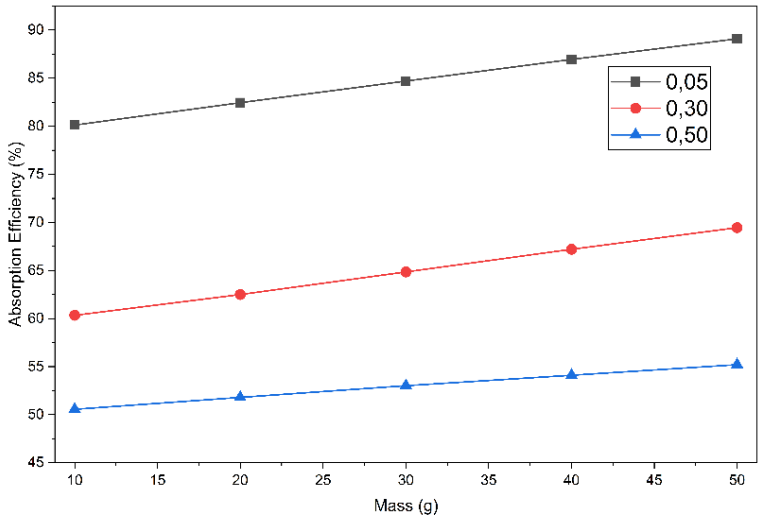


Figure 1. Effect of adsorbent mass on adsorption efficiency for various particle sizes

The adsorption mechanism, which involves the uptake of molecules by the adsorbent, occurs through processes including the transfer of molecules from the gas phase to the adsorbent surface, diffusion into the adsorbent pores, and finally adsorption at active sites on the surface [21]. An increase in zeolite mass leads to an increase in the number of active sites, resulting in improved adsorption efficiency. This is consistent with Figure 1, which shows that an increase in zeolite mass results in a significant improvement in adsorption efficiency. The reduction in final H_2S concentration, measured via iodometric titration, indicates that more H_2S molecules are adsorbed as the mass of the adsorbent increases.

An increase in zeolite mass has an impact on H_2S adsorption efficiency. Based on the observations made, it is evident that adsorption efficiency increases in line with an increase in adsorbent masfrom 10 g to 50 g. At an initial mass of 10 g, the adsorption efficiency was still in the moderate range. However, when the mass was increased to 20 g–30 g, a more pronounced increase was observed because the number of active sites on the zeolite surface capable of interacting with H_2S molecules increased. When the mass increases to 40 g and 50 g, the efficiency reaches its peak, indicating that the adsorption capacity increases in proportion to the amount of active material present. Increasing the adsorbent mass can shorten the diffusion path of gas molecules toward the pore centers, which accelerates the adsorption process while simultaneously strengthening the physical and chemical interactions taking place [20].

C. Effect of Particle Size on Adsorption Efficiency

In this study, five variations in zeolite particle size were used, namely 4, 10, and 40 mesh, with masses of 10, 20, 30, 40, and 50 g, respectively. Adsorption effectiveness was measured based on the reduction in sulfide concentration using the iodometric titration method as specified in SNI 6989.72:2009, with sodium thiosulfate ($Na_2S_2O_3$) serving as the indicator. Based on the data presented in Table 1, it can be seen that the particle size of the zeolite influences H_2S gas adsorption capacity. The smaller the particle size, the higher the adsorption efficiency achieved. This indicates that the specific surface area and the number of active sites increase as particle size decreases. Zeolites with smaller particle sizes provide a shorter diffusion path for H_2S molecules to reach the internal pores, allowing the adsorption process can proceed more quickly and efficiently [20]. The effect of particle size on H_2S adsorption efficiency is shown in **Figure 2**.

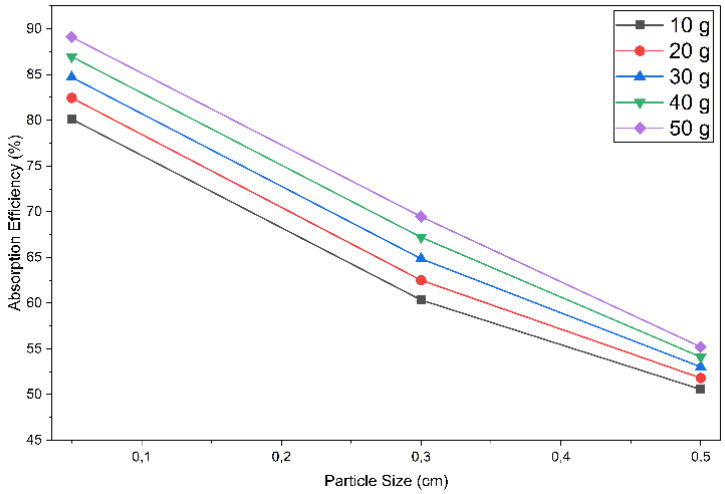


Figure 2. Effect of adsorbent particle size on adsorption efficiency for various adsorbent masses

This graph shows the correlation between particle size and adsorption efficiency, revealing a consistent decreasing trend from 40 mesh to 4 mesh. The adsorption efficiency indicates that zeolite with a particle size of 40 mesh provides the highest efficiency across all mass variations, while the 4-mesh size shows a significant decrease. This pattern confirms that reducing particle size can improve H_2S gas adsorption performance. Overall, the relationship between adsorption efficiency (η) and particle size (d) can be expressed by a simple empirical formula:

$$\eta \propto \frac{1}{d} \tag{5}$$

This equation shows that adsorption efficiency increases as particle size decreases, Furthermore, the relationship between particle size and adsorption capacity can be explained by the mechanisms of increased surface area and the ease with which molecules enter the pores of the structure. Zeolites with smaller particle sizes have a higher surface area-to-volume ratio. Particle size directly affects the surface area-to-volume ratio (SA/V). The smaller the particle size, the higher the SA/V ratio, meaning there are more active sites on the adsorbent's surface available to interact with molecules to be adsorbed, such as H_2S. Mathematically, this is described by a simple geometric relationship:

$$\text{Surface area-to-volume ratio} = \frac{A}{V} = \frac{6}{d} \tag{6}$$

This equation shows that as the particle diameter (d) decreases, the A/V value increases linearly. Consequently, the number of active sites that can interact with zeolite molecules increases, thereby also increasing the adsorption efficiency (η). In the process of sulfide adsorption on zeolite, smaller particles have shorter diffusion paths, so mass transfer occurs more rapidly. This makes it easier for sulfide molecules to reach active sites within the zeolite pores, leading to an increase in the number of adsorbed molecules in a shorter time. Additionally, a higher specific surface area allows for the formation of more contact points between the gas and the zeolite surface, which enhances adsorption efficiency.

D. Changes in pH During the Adsorption Process

The pH parameter was used as an indicator to analyze how effectively the zeolite adsorbs dissolved sulfide. Measurements were taken using a digital pH meter with an accuracy of ±0.01. Before the adsorption process, the wastewater had an initial pH of 5.48, indicating acidic conditions due to the presence of dissolved H_2S. Changes in pH values are presented in **Table 2**.

Table 2. Experimental Results on pH Changes and Turbidity Changes

Mass (g)	Particle Size (mesh)	Final pH	ΔpH
	40	6.90	**1.42**
10	10	6.45	**0.97**
	4	6.31	**0.83**
	40	6.93	**1.45**
20	10	6.70	**1.22**
	4	6.37	**0.89**
	40	7.00	**1.52**
30	10	6.62	**1.14**
	4	6.43	**0.95**
	40	7.15	**1.67**
40	10	6.78	**1.30**
	4	6.46	**0.98**
	40	7.20	**1.72**
50	10	6.95	**1.47**
	4	**6.49**	**1.01**

Table 2 shows that an increase in zeolite mass from 10 grams to 50 grams causes the pH to rise from approximately 5.48 to approximately 7.2. This is consistent with the increase in adsorption efficiency, where the efficiency of sulfide adsorption increases as the zeolite mass increases and particle size decreases. The rise in pH indicates that most H_2S molecules have reacted or decomposed into more stable ionic forms, as shown in the following equilibrium equation:

$$H_2S\ (aq) \rightleftharpoons HS^- + H^+ \quad (7)$$

In the pH range of 6.3–7.2, sulfides in solution generally exist as H_2S and HS^-, while the formation of S^{2-} ions is insignificant. An increase in pH from 5.48 to 6.31–7.20 indicates that the concentration of H^+ ions in the solution decreases. This occurs because sulfide is adsorbed onto the zeolite surface, causing the equilibrium in the solution to shift and resulting in a rise in pH toward neutrality. This phenomenon is consistent with the research by Sari D., et al., which states that the adsorption of acidic gases by porous materials such as zeolite tends to increase the pH. This occurs because when zeolite is introduced into water, cations in the solution can exchange with other cations present within the zeolite, such as H^+ ions. By absorbing hydrogen ions from the solution and releasing basic cations from the zeolite, the concentration of free H^+ ions insolution decreases, causing the pH to rise.

This rise in pH goes hand in hand with an increase in the efficiency of the adsorption process. This is due to ion exchange and electrostatic interactions between sulfide and the active sites within the zeolite, which increase the number of H_2S molecules trapped in the zeolite pores. This indicates that the greater the mass of adsorbent used, the more active basic sites are available to interact with ionized H_2S molecules, thereby increasing the adsorption rate and overall adsorption capacity. Additionally, smaller particles have a higher surface area-to-volume ratio , so the gas diffusion path to the active pores is shorter and the adsorption equilibrium process occurs more rapidly. This explains why the sample with a mass of 50 grams and the smallest particle size exhibited the best adsorption efficiency among those tested, as well as the most significant increase in pH. Nevertheless, it should be noted that the iodometric method used in this study is susceptible to interference from other

reducing compounds that may be present in oil and gas industrial wastewater. Therefore, the concentration values obtained in this study may not fully represent sulfides specifically, but rather may include contributions from other reducing compounds present in the system.

IV. CONCLUSION

A. Conclusion

This study demonstrates that 1 M HCl-activated zeolite, when used as an adsorbent, effectively reduces the concentration of dissolved sulfides in oil and gas industry wastewater. Removal efficiency increases with increasing adsorbent mass and decreasing particle size; the optimal conditions tested were a mass of 50 g and a particle size of 0.05 cm, yielding an efficiency of 89.10%. This improvement in adsorption performance is related to the increased contact area between the zeolite and the sulfide due to the increase in mass and the reduction in particle size, resulting in more effective interactions. Additionally, the adsorption process was accompanied by an increase in the solution's pH toward nearing neutral, indicating a decrease in the concentration of H^+ ions in the solution.

B. Recommendations

Further research is recommended to conduct additional testing on the effects of contact time, pH, and initial sulfide concentration, as well as to evaluate the zeolite's regeneration and reuse capabilities in order to analyze its performance stability after the adsorption process. In addition, further characterization of the HCl-activated zeolite is needed, such as analysis of surface area, pore structure, and morphology, to obtain more in-depth information regarding changes in zeolite characteristics and the adsorption mechanisms involved.

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