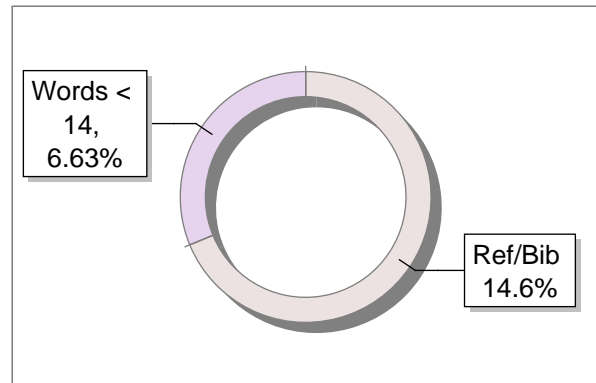
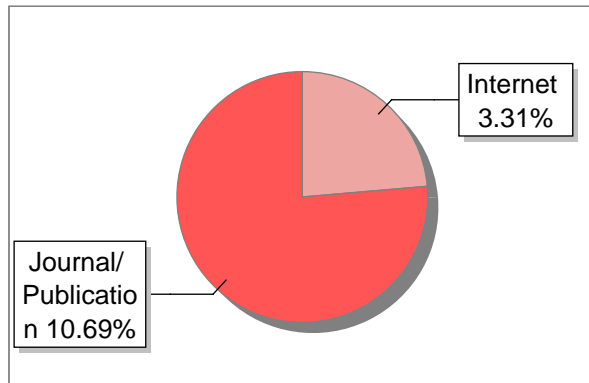
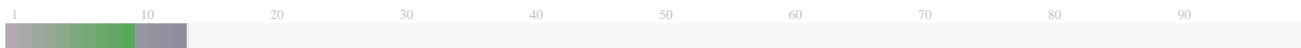


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## REMOVAL OF LEAD AND IRON IN LEACHATE WASTEWATER OF THE TPST PIYUNGAN BY ACTIVATED NATURAL ZEOLITE

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**Abstract:** Landfill generates a problem of soil and water pollution, with leachate wastewater contaminating heavy metals. Wastewater treatment with natural zeolite adsorbents can remove heavy metals from wastewater. We have analyzed the leachate wastewater in the last pond at the wastewater treatment plant at the TPST Piyungan contains lead (Pb) ions of 2.13 mg/L and iron (Fe) of 9.94 mg/L, greater than the maximum levels allowed for sanitary hygienic water. according to the regulations of the Minister of Health. The concentration of Pb and Fe metal ions in wastewater can be removed by activated local natural zeolite adsorbents. The adsorbent was prepared from local natural zeolite by activation with ammonium nitrate solution and calcined at 300 °C for 2 hours. Characterization by XRD, BET-adsorption analysis, and SEM showed that activation can increase natural zeolite's surface area and pore size without being followed by a change in the crystal structure. The leachate adsorption experiment was carried out by varying the weight percentage of activated natural zeolite adsorbent and adsorption time. The concentration of Pb and Fe ions in wastewater can be reduced by activated natural zeolite adsorbents. Adsorption with 10% weight of natural zeolite and an adsorption time of 60 minutes delivered the best results. Under these adsorption conditions, the concentration of Pb ions decreased by 38.50% and that of Fe decreased by 67.10%. The adsorption treatment with activated natural zeolite can significantly reduce the concentration of Pb and Fe metal ions in the waste leachate of TPST Piyungan.

**Keywords:** landfills; leachate; wastewater; lead; iron; natural zeolite.

**Abstrak:** Tempat Pembuangan Sampah (TPS) menimbulkan masalah polusi air dan tanah dengan air limbah lindi yang mengandung logam-logam berat. Perlakuan dengan adsorben zeolit alam dapat mengurangi logam berat dalam air limbah. Analisis dengan AAS menunjukkan bahwa air limbah pada kolam terakhir instalasi pengolahan air limbah di TPST Piyungan mengandung ion logam timbal (Pb) 2,13 mg/L dan besi 9,90 mg/L. Kadar kedua logam berat ini melebihi ambang batas yang dibolehkan dalam air untuk sanitasi higien menurut regulasi Kementerian Kesehatan. Konsentrasi ion logam Pb dan Fe dapat dikurangi dengan adsorben zeolit alam lokal yang diaktivasi. Adsorben dibuat dengan aktivasi zeolit alam lokal dengan larutan ammonium nitrat dan dikalsinasi pada 300°C selama 2 jam. Hasil analisis adsorben dengan XRD, analisis adsorpsi BET, dan SEM menunjukkan bahwa aktivasi dapat menaikkan luas permukaan zeolit dan ukuran pori tanpa diikuti perubahan struktur kristal. Eksperimen adsorpsi limbah lindi dilakukan dengan variasi persentase berat adsorben zeolit alam dan waktu adsorpsi. Konsentrasi ion Pb dan Fe dalam limbah lindi dapat dikurangi dengan adsorben zeolit

alam teraktivasi. Hasil terbaik diperoleh dengan penggunaan 10% berat adsorben zeolit dan waktu adsorpsi 60 menit. Pada kondisi ini konsentrasi ion Pb berkurang 38,50% dan Fe turun sebesar 67,10%. Perlakuan adsorpsi dengan zeolit alam teraktivasi dapat mengurangi secara signifikan konsentrasi ion logam Pb dan Fe dalam limbah lindi TPST Piyungan.

**Kata kunci:** TPST; air limbah; lindi; timbal; besi; zeolit alam.

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## Introduction

The Piyungan Integrated Waste Disposal Site (TPST Piyungan) is the disposal site for urban waste originating from the City of Yogyakarta, Sleman Regency, and Bantul Regency. In waste management, the TPST applies a landfill system (Ariyani et al., 2019). Besides being useful for waste management, the existence of the TPST also hurts the surrounding environment, especially soil pollution through leachate. In leachate, heavy metals from waste such as Pb, Cu, Zn, and Cd are potentially hazardous to health (Lokahita et al., 2018; Sihombing et al., 2020). More piles of waste cause more leachate production so the potential for soil and water pollution in the area around the TPST is getting bigger.

Several previous studies have shown that there has been heavy metal pollution in the soil layer around the TPST (Harjito et al., 2018; Sartohadi et al., 2017; Parhusip et al., 2016; Parhusip et al., 2017; Muyassar & Budiman, 2021). Soil contamination with heavy metals occurs through the intrusion of leachate into the soil layer. Garbage is buried directly on the land without an impermeable layer underneath so that all contaminants including heavy metals have the potential to intrude into the soil layer. Pollutant migration through the subsoil is accelerated by the presence of high rainfall, which causes the infiltration of heavy metals into groundwater (Muyassar & Budiman, 2021). As a result, it is suspected that the resident's water sources around the TPST are polluted by heavy metals. So far there has been no research to analyze heavy metal contamination in residents' water sources around the TPST and how to solve the problem.

The results of previous research showed that the soil around the TPST site was contaminated with heavy metals Pb, Cu, Zn, and Pb (Almasoud et al., 2020). Although the structure of the soil layers there is capable of holding heavy metal contaminants from leachate sources, the results of other studies identified the movement of leachate infiltration into the soil with a depth of 5-20 meters to the northwest of the TPST. The movement of leachate infiltration into the soil causes contamination of groundwater in residential areas, as evidenced by the results of the physicochemical analysis of water (Parhusip et al., 2017; Muyassar & Budiman, 2021). This study has examined the water quality of 29 dug wells around the TPST.

As a result, all the well water studied exceeded the normal threshold in terms of TDS, DHI, and salinity parameters. Another research group (Mancinelli et al, 2020). identified contamination of the heavy metals in the soil around the TPST with light to heavy levels of contamination.

The removal of heavy metals in wastewater can be done with several adsorbents, including materials based on activated carbon and natural zeolites. Previous studies have been carried out to investigate the adsorption efficiency of natural zeolite on heavy metal ions of  $\text{Cu}^{2+}$ ,  $\text{Pb}^{2+}$ , and  $\text{Cd}^{2+}$  in industrial wastewater [Tasic et al., 2019; Elboughdiri, 2020; Mancinelli et al., 2020). The adsorption efficiency of Cu, Pb, and Cd metal ions increased proportionally with the addition of zeolite weight, the mixing speed of the batch system, the initial concentration of the waste, and the initial pH of the waste solution. The highest heavy metal adsorption rate occurred in the first hour of treatment. As a comparison, synthetic zeolite type ZSM-5 has been studied as an adsorbent for filtering industrial wastewater (Krol, 2020). The results showed that ZSM-5 has a fairly good adsorption efficiency to reduce contaminants in groundwater. This zeolite functions as an adsorbent that can remove a wide range of contaminants, including heavy metals, chlorinated solvents, and aromatic hydrocarbons. Another research group has modified the natural zeolite type clinoptilolite by impregnating sulfur from  $\text{Na}_2\text{S}$  at  $150^\circ\text{C}$  with the aim of increasing the adsorption of mercury Hg (II) in solution (Ugrina et al., 2021). Adsorption of Hg (II) ions in sulfur-impregnated zeolite is affected by pH, adsorbent/solution ratio, initial concentration of Hg(II), and contact time. Optimal adsorption conditions were reached at pH 2, adsorbent/solution ratio of 10 g/L, and contact time of 800 minutes. The maximum adsorption capacity of sulfur-impregnated zeolite against Hg (II) was 1.02 mmol/g. Sulfur impregnation on zeolite increased Hg (II) adsorption 3.6 times higher than natural zeolite. Furthermore, the leaching test indicated that the separation of Hg (II) from the zeolite structure occurred over a wide pH range so that the sulfur-impregnated adsorbent could be a potential material for the remediation of mercury-contaminated environments. Montmorillonite natural zeolite can be used to remove the radioactive element Ra-226 in groundwater. The efficiency of zeolite in removing Ra-226 is higher than active biochar (Almasoud et al., 2020).

There are abundant natural zeolite deposits in the Gunung Kidul district, that have not been properly utilized (Muzwar et al., 2018). Based on its abundant availability, low cost, and high adsorption efficiency, zeolite can be a promising adsorbent for removing heavy metals from wastewater solutions and groundwater remediation (Jagaba et al., 2021; Wingfelder et al., 2005). However, so far the removal of heavy metals from leachate from landfills with natural zeolite has not been studied. In this paper, we report the capacity of activated local natural zeolite to remove lead and iron ions in dilute leachate wastewater from the TPST.

## Method

### Materials

Local natural zeolite (Figure 1) was obtained from a deposit in the area of Hargomulyo, Gunung Kidul, Yogyakarta. Natural zeolite is an aluminosilicate compound with a unique tetrahedral structural framework ( $x\text{AlO}_4 \cdot y\text{SiO}_4 \cdot n\text{H}_2\text{O}$ ). Wastewater samples were taken from the last pond at the leachate treatment plant at TPST Piyungan, Bantul, Yogyakarta (Figure 2). The activating solution was prepared from solid ammonium nitrate pro-analysis grade (E. Merck, Germany).

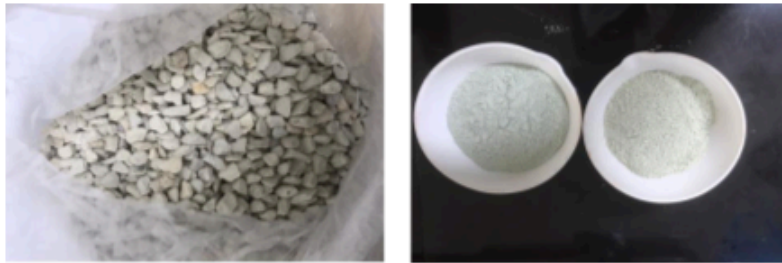


Figure 1. Original natural zeolite (left) and its powder (right)



Figure 2. Leachate wastewater from the last pond of TPST Piyungan

### Preparation and characterization of natural zeolite adsorbents

Solid natural zeolite samples were cleaned of impurities and ground into powders with a size of 90 mesh. The activation process was carried out by mixing natural zeolite powder into a 1 molar ammonium nitrate solution with a weight/volume ratio of 1:2. The mixture was stirred with a magnetic stirrer at medium speed at room temperature for 1 hour. The mixture was separated by filtering. The zeolite solids were dried in an oven at 110°C for 2 hours, then ground into 90 mesh size powder. Zeolite powder was calcined at 350°C for 2 hours to



produce activated zeolite adsorbent. The characterization of the zeolite adsorbent was carried out in Integrated Laboratory Universitas Islam Indonesia (UII) by XRD to determine the crystalline phase, by BET-sorption to determine the surface area, pore size, and BET-isotherm type, and SEM analysis to image the surface morphology.

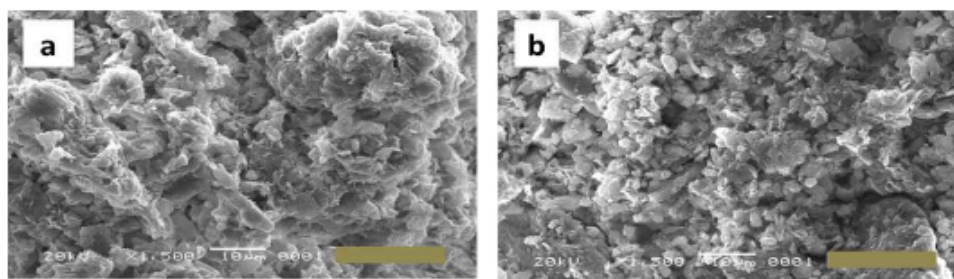
#### *Removal of Pb and Fe ions in leachate wastewater*

Leachate wastewater is diluted with distilled water in a volume ratio of 1:1. The initial concentration of Pb and Fe ions in leachate wastewater was analyzed by AAS Perkin Elmer PinAAcle 900T equipped with flame or graphite furnace. Adsorption experiments were performed by adding 2 g of activated natural zeolite powder (4%) into 50 mL of dilute leachate wastewater. The mixture was stirred with a magnetic stirrer for 30 minutes, and then filtered. After adsorption with activated natural zeolite, the filtrate obtained was analyzed with AAS to determine the concentration of Pb and Fe ions. To study the optimum adsorption conditions, the experiment was repeated with variations in the amount of 10% and 20% zeolite adsorbent and the stirring time was 60 minutes and 90 minutes.

## **Result and Discussion**

#### *Properties of activated natural zeolite adsorbent*

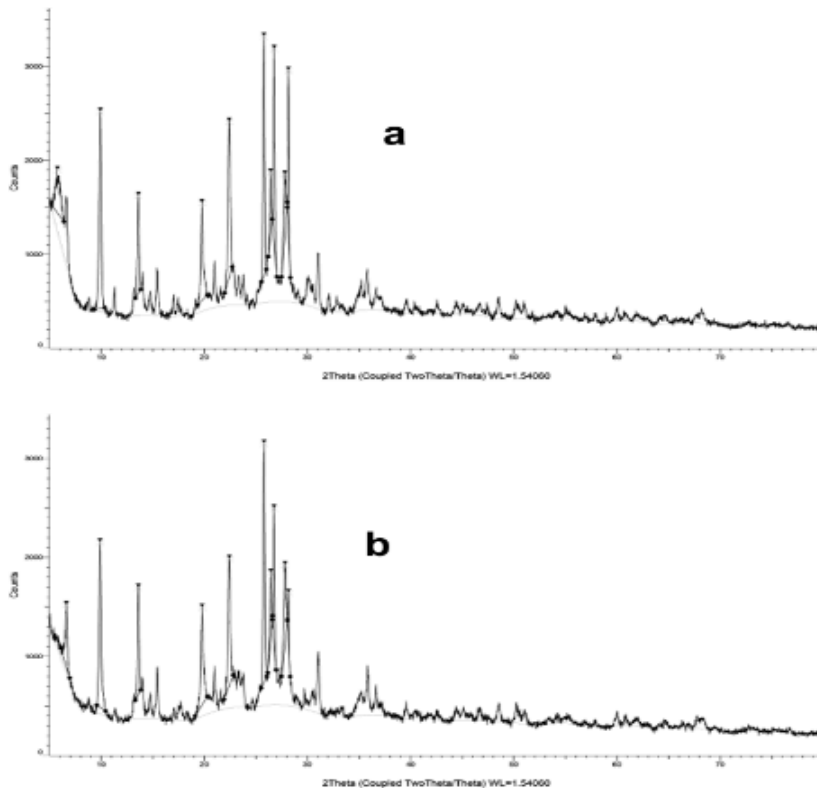
The activation process of natural zeolite with ammonium nitrate solution and calcination at high temperatures does not significantly change or damage the structure and porosity of natural zeolite. The surface morphology of natural zeolite before and after activation did not change significantly as shown in Figure 3. Original natural zeolite and activated natural zeolite are porous amorphous mixed crystalline materials.



**Figure 3.** SEM images (x 1500) of natural zeolite (a) and activated natural zeolite (b)

The original natural zeolite XRD diffractogram pattern before and after activation shows that the main phase of natural zeolite consists of the mineral montmorillonite mixed with quartz (Figure 3). The similarity of the diffractogram patterns in Figure 4 shows that activation and calcination also do not change the phase and crystalline structure of the natural zeolite. The thermal and chemical

stability of zeolite is a requirement for zeolite applications as adsorbents and catalysts (Krol, 2020).

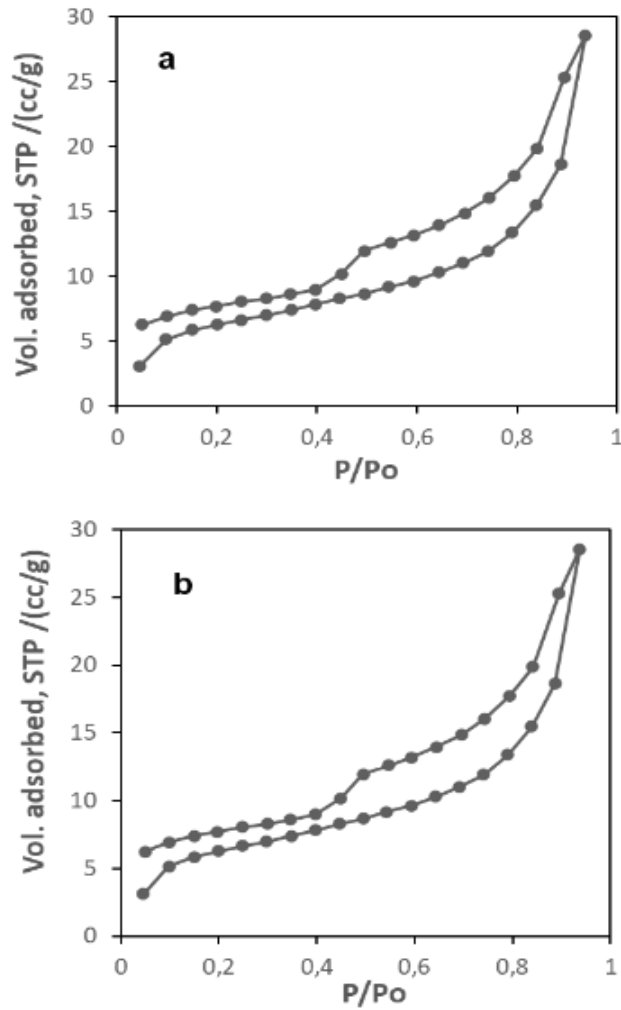


**Figure 4.** Diffractogram patterns of original natural zeolite (a) and activated natural zeolite

Other important properties that must be possessed by adsorbent materials are adequate surface area and porosity. Figure 5 shows the BET isotherms of the natural zeolite before and after activation. The two BET isotherm patterns of zeolite samples which are similar to type IV can be interpreted that natural zeolite is an amorphous crystalline mixed solid material with microporosity. This is supported by the data in Table 1, which shows the diameter of the micro-sized pores. Although activation and calcination do not significantly affect the structure and morphology of natural zeolite, activation can slightly increase the surface area of zeolite from about 23 to 35 cm<sup>2</sup>/g. The presence of pores with sufficient surface area will allow molecules and/or ions of the appropriate size to enter the pores and be adsorbed physically and chemically in the adsorbent (Krol, 2020).

**Table 1.** BET-surface area and pore size of natural zeolite

Zeolite sample	Surface area / (m <sup>2</sup> /g)	Pore diameter / nm	Pore volume / (cc/g)
original	23,27	3,81	0,04
activated	34,68	5,48	0,06



**Figure 5.** BET-isotherm of original natural zeolite (a) and activated natural zeolite (b)

*Removal of Pb and Fe ions in wastewater by activated natural zeolite*

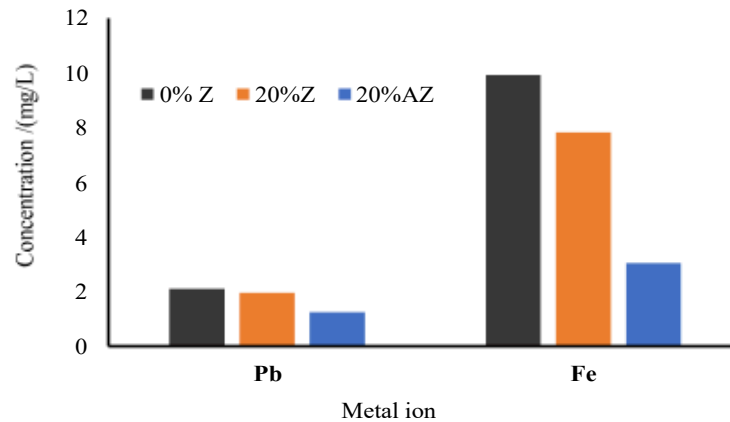
The results of the analysis with AAS showed that leachate wastewater in the last pond at the TPST waste treatment plant contained metal ions of Pb 9.94 mg/L and of Fe 2.13 mg/L. The concentration of these heavy metal ions is greater than the maximum allowed value for hygienic water sanitation according to the regulation of the Minister of Health of the Republic of Indonesia number 32 of 2017, namely 0.05 mg/L Pb and 1 mg/L Fe. There is a potential for heavy metal contamination into river water and groundwater sources for the surrounding community if the leachate wastewater is discharged into the river. We have analyzed samples of river water where leachate was disposed of and some water from community wells adjacent to the TPST Piyungan. The result is presented in Table 2. The concentration of Pb ions in river water and some well water around the landfill was greater than the threshold value of 0.05 mg/L, while the concentration of Fe ions was lower than the threshold value of 1 mg/L. This indicates that some of the residents' well water around the TPST has been infiltrated by lead (Pb) metal, which has the potential to harm health if it is used for the consumption of drinking and cooking water.

**Table 2.** The existence of Pb and Fe ions in the river and communal water around the TPST Piyungan

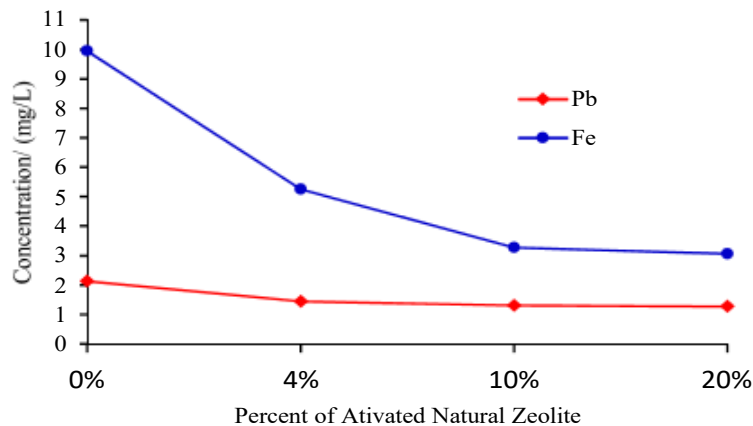
Water sample	Concentration / (mg/L)	
	Fe	Pb
IPAL-leachate	9,94	2,13
Well 1	0,56	0,44
Well 2	0,52	0,40
Well 3	0,56	0,50
River	0,55	0,51

Natural zeolite is a promising adsorbent due to its availability and low cost for removing metal cations from wastewater (Tasic et al., 2019; Elboughdiri, 2020). To ensure that the activation process of natural zeolite affects the adsorption capacity to metal ions, an experiment was carried out to remove Pb and Fe ions from TPST leachate wastewater with natural zeolite. Figure 6 shows a comparison of the adsorption capacity between the original natural zeolite and activated natural zeolite. Activated natural zeolite removes 38% of Pb ions in wastewater, while the original natural zeolite only removes 4% of Pb ions. A similar fact was found for the adsorption capacity of zeolite for Fe ions. This means that the activation process increases the adsorption capacity of the zeolite by 9 times greater than that of the original material. The activation process with ammonium nitrate solution followed by calcination at 350°C is believed to improve the properties of the zeolite such as porosity, morphology, and surface area; which plays an important role for adsorbents (Wingenfelder et al., 2005). The adsorption capacity to remove metal ions in wastewater depends on several factors, such as the amount of adsorbent, initial metal concentration, adsorption contact time, and agitation speed. In this

work, we only study the factors of the adsorbent amount and adsorption contact time.



**Figure 6.** Comparison of Pb and Fe ions decreasing capability between original natural zeolite (Z) and activated natural zeolite (AZ); adsorption condition: 10%-weight of zeolite and time of 60 minutes.

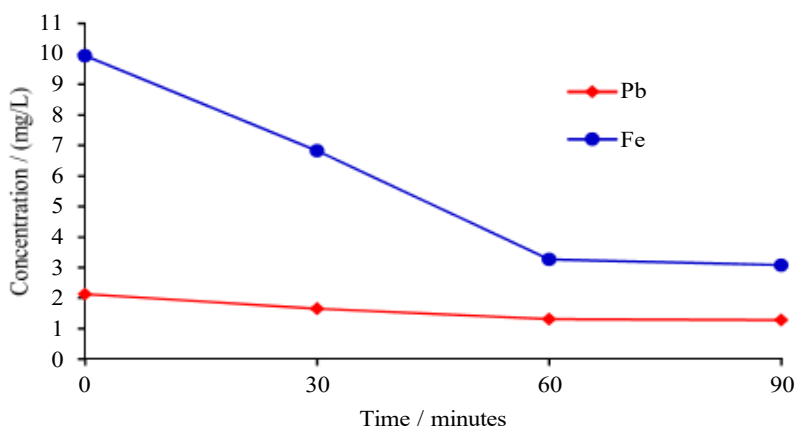


**Figure 7.** Effect of zeolite adsorbent percentage on decreasing of Pb and Fe ions in wastewater of the last pond of waste treatment installation at the TPST Piyungan for 60 minutes.

The effect of the amount of activated natural zeolite adsorbent on decreasing the concentration of Pb and Fe ions in leachate wastewater is presented in Figure 7. Adsorption for 60 minutes with the use of 4 to 10% activated natural zeolite can reduce the concentration of Pb ions by 38% and Fe by 67%. Increasing the amount of adsorbent up to 20% only slightly increases the adsorption capacity. This follows the results of previous studies, which stated that the metal ion removal efficiency tended to decrease or remain constant as the adsorbent dose increased above 10% (Wingfelder et al., 2025). It can also be stated that the use of 10% activated

natural zeolite adsorbent is the optimum dose for removing Pb and Fe ions from leachate wastewater.

On the other hand, adsorption contact time also plays an important role in reducing the concentration of Pb and Fe ions. Adsorption experiments within 30 minutes to 90 minutes showed that the concentration of Pb and Fe ions in the wastewater samples decreased with increasing adsorption contact time as shown in Figure 8. Adsorption using 10% activated natural zeolite and stirring for 60 minutes can reduce the concentration of Pb and Fe ions. Another study has found the effect of adsorption contact time on the level of efficiency of removing some heavy metal ions in wastewater. The adsorption is a heterogeneous process as the removal rate of several heavy metal ions mostly occurred early on, but as the contact time increased, there was a decrease in the level of the effect of the contact time on adsorption and the adsorption process became slower (Elboughdiri, 2020). Based on these data it can be stated that the optimum condition for adsorption to reduce the concentration of Pb and Fe ions within this experimental limit was the use of 10% (weight/volume) activated natural zeolite adsorbent and adsorption time of 60 minutes.



**Figure 8.** Effect of adsorption contact time on decreasing of Pb and Fe ions in wastewater of the last pond of waste treatment installation at the TPST Piyungan by 10%-weight zeolite adsorbent.

The binding of heavy metal cations on the adsorbents is well described by some theoretical models, such as the geochemical equilibrium speciation model that accounts for metal complexation at hydrous oxides, ion exchange mechanism on the zeolite surfaces, as well as dissolution and precipitation processes (Wingenfelder et al., 2005). Among these models, the cation exchange mechanism is more suitable to describe the binding process of heavy metal ions on the surface of the zeolite. Recently Liu et al (2022) have modelled the adsorption mechanism

of ammonium ions ( $\text{NH}_4^+$ ) on natural zeolite layered structures. Undoubtedly, this mechanism can be adapted to explain the process of activating natural zeolite with ammonium nitrate solution and for metal cation exchange. We, therefore, adapted and modified this mechanism as a model for the binding mechanism of Pb and Fe metal ions through cation exchange in the activated natural zeolite. The modified cation exchange mechanism of Pb and Fe ions on activated natural zeolite is shown in Figure 9. Further experiments need to be carried out to explain the sorption kinetics of Pb and Fe metals on natural zeolite adsorbents.

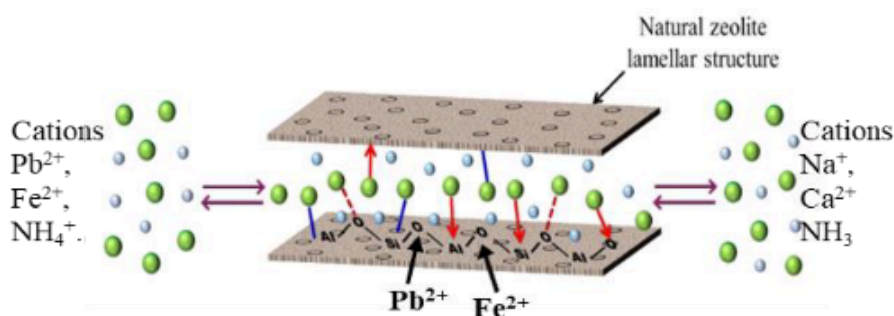


Figure 9. Pb and Fe cations exchange mechanism on activated natural zeolite adapted from Liu et al. (2022)

## Conclusion

Leachate wastewater samples in the last pond of the wastewater treatment installation at the TPST Piyungan contained 2.13 mg/L of Pb metal ions and 9.94 mg/L of Fe metal ions, which are higher than the threshold values for sanitary hygiene water. Treatment of wastewater using 10% by weight/volume of activated natural zeolite for 1 hour can remove 38.5% of Pb metal ions and 68% of iron ions respectively. Increasing the dose of zeolite adsorbent above 10% and adsorption contact time above 1 hour only slightly increased the adsorption capacity. The adsorption of Pb and Fe metal ions occurs through the mechanism of cation exchange in the layer of natural zeolite lamellar structure. The kinetic study of the adsorption of Pb and Fe metals on natural zeolite adsorbents is still under investigation.

## Acknowledgement

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