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Application of zeolite-y based on sidrap clay and rice husk ash as the adsorption of copper (Cu) and lead (Pb) metals

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ABSTRACT

This research examines zeolite-Y made from sidrap clay and rice husk ash as the adsorption of copper (Cu) and lead (Pb) metals. The method used is hydrothermal, using an autoclave with variations of Pb and Cu metals, namely 200, 400, and 600 ppm. The results showed that zeolite-Y was able to adsorb Cu and Pb well, as evidenced by the presence of Pb and Cu in the mapping results and EDS (Energy Dispersive X-Ray) and SEM (Scanning Electron Microscopy) showed the morphology of each sample of Cu and Pb metals. Pores indicate the absorption of Cu and Pb metals in the zeolite. The results of AAS (Atomic Absorption Spectrophotometer) show that the greater the levels of Cu and Pb produced, the greater the absorbent level.

Keywords: adsorption; copper (Pb); lead (Pb); zeolit-Y

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INTRODUCTION

As the industry grows, some advantages and disadvantages arise. Benefits include the production of equipment to support daily life, such as batteries, textiles, household appliances, etc. Meanwhile, the disadvantage is in the form of industrial waste that contains hazardous and toxic materials (B3) such as heavy metals (Anrozi & Trihadiningrum, 2017; Raharjo & Kuswadi, 2020). The effects arising from heavy metal pollution encourage search efforts to overcome them so that the benefits obtained from their use are not followed by risks that are accepted by humans, and the environment is non-biodegradable (Javanbakht et al., 2016; Nicoleta Popa, 2015). Sources of heavy metal ions in wastewater include mining and industries such as paper, textiles, etc (Suherni, 2010). Heavy metal contamination from wastewater generated by industries such as mineral processing, petrochemical activities, and metal plating facilities has caused worldwide concer (Shi et al., 2013). Heavy metals harm human health, resulting in reduced growth and development, as well as diseases such as cancer, damage to organs and nervous systems, and ecosystems. Therefore, various techniques, including ion exchange, chemical precipitation, membrane separation, and adsorption, have been developed to remove heavy metals from wastewater (Liu & Zhang, 2018). One way to reduce heavy metal pollution is using adsorbents to adsorb heavy metals. Several methods of removing heavy metals can be carried out by ion exchange, precipitation, phytoextraction ultrafiltration, reverse osmosis, electrodialysis, and adsorption techniques. (Mier et al., 2001). The essential thing in the adsorption process is selecting a suitable type of adsorbent. One of the most potential adsorbents is zeolite (Lehman & Larsen, 2014; Vishnu et al., 2021). Zeolite has several properties: dehydration, high cation exchange, a suitable catalyst, and an adsorbent for other compounds (Hartmann et al., 2016; Kundari et al., 2010; Setiawan et al., 2020; Solikah & Utami, 2014; Wei et al., 2015). In addition, the strength of the zeolite acid can also be controlled.

About 20% of the weight of rice is rice husk, varying from 13 to 29% of the composition of the husk is husk ash which is always produced every time the husk is burned. The most common silica (SiO2) content value in rice husk ash is 94 – 96% (Prasetyoko et al., 2006). The high content of silica in rice husk ash that it can be used in the manufacture of zeolite-based materials (Deviani et al., 2018; Saceda et al., 2011; et al., 2018). Clay minerals are layered silicates; the crystal structure of these minerals is composed of layers of SiO4 tetrahedron. In the center of the 6-banded SiO4 tetrahedron, there is usually a hydroxyl ion (OH). Clay minerals consist mainly of aluminum and/or iron, and magnesium silicates. Some of them also contain alkaline or alkaline earth as basic components (Setiawan et al., 2014). The synthesis of zeolite has been widely studied before, such as by Putri Hawa Syaifie (2019), who succeeded in synthesizing zeolite from kaolin and rice husk ash for the adsorption of copper and chromium metal in industrial wastewater. (Syaifie et al., 2019). Citra Deliana Dewi Sundari (2018) synthesized Synthesis of zeolite L using rice husk ash silica for methylene blue adsorption: kinetic and adsorption isotherms (Sundari et al., 2018). Lisanti Emelda (2013) used activated Natural Zeolite for Cr^{3+} metal adsorption (Emelda et al., 2013). Santasnachok dkk. (2014) synthesized zeolite from rice husk ash as an adsorbent for heavy metal Cd (II) in the Zn mining industry. The maximum Cd(II) removal capacity obtained from Na-A and Na-X zeolites were 736.38 and 684.46 mg/g, respectively (Santasnachok et al., 2014).

In this research, the application of zeolite as adsorption of copper Cu and lead Pb will be carried out using the basic ingredients of rice husk ash and clay, which are widely available in the Sidrap area of Sulawesi.

RESEARCH METHODS

The clay used as the primary material for zeolite comes from Sidrap Regency, South Sulawesi. The clay is cleaned of impurities and soaked with Aquadest to separate large lumps of clay particles. The results of the clay bath were filtered and then dried in the oven at 100℃ for 2 hours. The dry clay was ground to smaller particle size and then sieved through a 200 mesh sieve. The sieve results were then dehydroxylated for 4 hours at a temperature of 750℃.

Rice husk ash used as a mixture for making zeolite comes from Sidrap Regency, South Sulawesi. Rice husk ash taken from the rice mill factory was first soaked in 0.1 mol HCl solution for 4 hours. The HCl solution is intended to release impurity minerals attached to rice husks. The immersion results were washed with distilled water several times until the remaining HCl solution was lost and dehydroxylated at 850℃ for 4 hours.

The method used in the manufacture of zeolite is hydrothermally utilizing an autoclave. The hydrothermal process is a crystallization process, namely the arrangement of atoms to obtain a stable structure/crystal. This stage determines the type of zeolite. The length of time and temperature of crystallization will affect the results of the arrangement (Sriatun et al., 2017). Clay and rice husk ash were activated using NaOH and various Pb and Cu solutions.
Pb and Cu solutions with concentrations of 200, 400, and 600 ppm were taken as much as 19.3 grams, respectively.

The addition of NaOH serves as an alkaline condition during the synthesis of Na-Y zeolite. Also, it forms a soluble sodium alumina salt to convert it into the zeolite. The Na+ cation from NaOH is used to stabilize the charge of Al3+ ions in the zeolite framework, but it is also needed to synthesize zeolite under hydrothermal conditions (Ojha et al., 2004).
After everything is mixed then, poured into a cylindrical mold and put into an autoclave, and heated in the oven for 3 hours at a temperature of 100℃.

RESULTS AND DISCUSSION

X-ray Diffraction (XRD) Characterization

XRD characterization was carried out to identify the phase, lattice parameters, and degree of crystallinity contained in each sample.

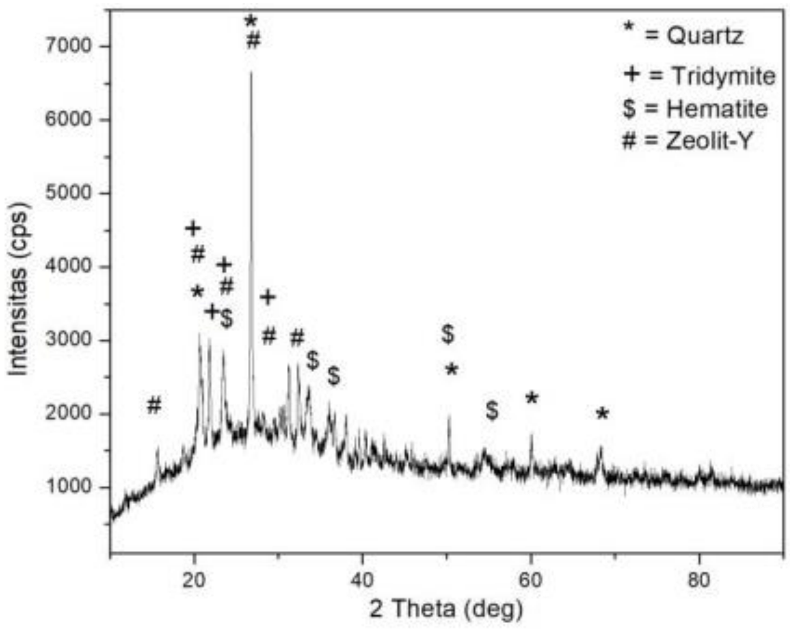


Figure 1. X-Ray Diffraction (XRD) characterization results in Zeolite-Y (Armayani et al., 2020)

From the XRD results, it can be seen in Figure 1. The manufacture of zeolite from the results of mixing sidrap clay and rice husk ash with the highest peak content obtained quartz and zeolite-Y with the highest intensity at 2θ = 15.61^0; 20,950^0; 21,794^0; 23.44^0; 26,737^0; 33.59^0;

35.97^0; and 41.15^0. The percentages of compounds in table 1. are in the form of zeolite type Y, Quartz, Trydimite, and Hematite.

Table 1. XRD qualitative analysis data

Phase name	Content (wt%)
Tridymite, syn	4,5
Quartz low HP, syn	58,0
Zeolte Y (Na)	28,0
Iron diiron(III) Oxide magnetite low	9,6

Type Y zeolite is used in the application of Cu and Pb adsorption.

Atomic Absorption Spectophotometer (AAS)

AAS characterization was carried out to determine how much adsorption power Cu and Pb metals were absorbed by zeolite Y. Each absorbent used was 200, 400, and 600 ppm, which were directly mixed in the process of making zeolite-Y using sidrap clay and risk husk ash as the basic ingredients.

a) Cu adsorption Zeolite

Based on the results of the AAS test for Cu adsorption zeolite using ppm variations of 200, 400, and 600 ppm, it can be seen from table 2 that the more Cu contained in the zeolite indicates that, the greater the adsorption power is absorbed. It can be seen from the 200 ppm Cu sample with 47.56 ppm Cu content that can be fascinated by the adsorption power of 0.0830. Samples of Cu 400 ppm with a concentration of Cu with levels of 38.17 ppm can be absorbed by the adsorption power of 0.0213. and Samples of Cu 600 ppm with Cu content of 57.43 ppm can be absorbed by the adsorption power of 0.0840.

In addition, the ability of the adsorbent to adsorb substances is influenced by several factors, namely the surface area of the adsorbent, the type of adsorbate, the concentration of the adsorbate, the molecular structure of the adsorbate, temperature, stirring speed, contact time is also influenced by the porosity of the adsorbent (Syauqiah et al., 2011).

Table 2. Results of AAS Testing for Metal Cu adsorption Zeolite samples

Code Sample	Absorbance	[Cu] (mg/L)	Fp (times)	[Cu] x fp (mg/L)	B. Sample (G)	V.Sample (mL)	Grade Cu (mg/kg=ppm)
Cu 200	0,0830	0,9522	1	0,95	1,0011	50	47,56
Cu 400	0,0213	0,2326	1	0,23	0,3047	50	38,17
Cu 600	0,0840	0,9638	1	0,96	0,8392	50	57,43

b) Pb adsorption Zeolite

Based on the results of the AAS test for Pb adsorption zeolite using ppm variations of 200, 400, and 600 ppm, it can be seen that the more Pb contained in the zeolite indicates that, the greater the adsorption power is absorbed. It can be seen from the 200 ppm Pb sample with Cu content of 1491.76 ppm that can be absorbed by the adsorption power of 0.0113. Samples of Pb 400 ppm with Pb levels with levels of 842.65 ppm can be absorbed with an adsorption capacity of 0.0057, and Pb samples of 600 ppm with Pb levels of 488.91 ppm can be absorbed in an adsorption capacity of 0.0047.

Table 3. Results of AAS testing for metal Pb adsorption Zeolite samples.

Code Sample	Absorbance	[Pb] (mg/L)	Fp (times)	[Pb] x fp (mg/L)	B. Sample (G)	V.Sample (mL)	Grade Pb (mg/kg=ppm)
Pb 200	0,0113	0,6233	10	16,23	0,5441	50	1491,76
Pb 400	0,0057	0,8052	10	8,05	0,4778	50	842,65
Pb 600	0,0047	0,6624	10	6,62	0,6774	50	488,91

Zeolite is an adsorbent (absorbent) for the binding of certain compounds and molecules that only occurs on the surface. This process occurs due to physical interactions by van der Walls forces and chemical interactions with electrostatic properties (Atmono et al., 2017). The properties of zeolite include dehydration, ion exchange, adsorption, catalyst, and filtering/separation.

Characterization of Scanning Electron Microscopy (SEM) and Mapping

SEM characterization was carried out to determine the surface morphology of the zeolite samples formed.

a) ***Zeolite-Y***

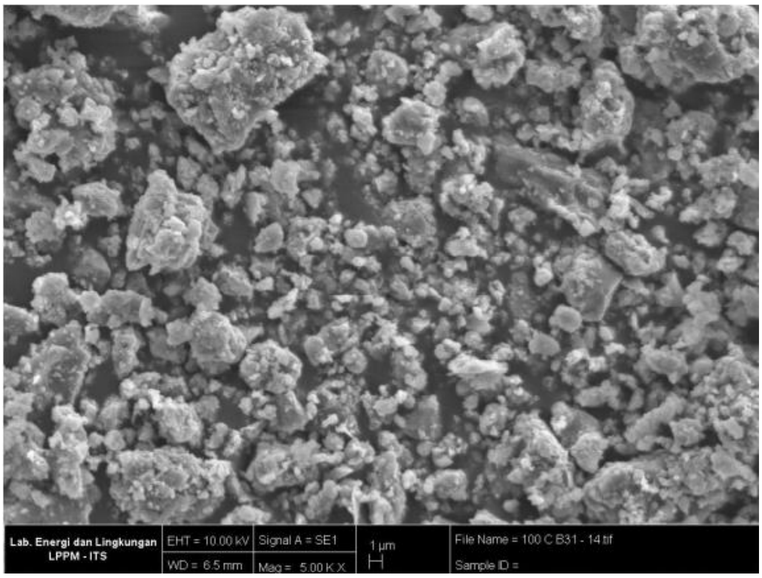


Figure 2. Results of SEM Image (Scanning Electron Microscopy) of Zeolite-Y sample (Armayani et al., 2020)

The morphology of zeolite-Y using clay and rice husk ash as a base material with a magnification of 5,000 times shows grains of varying or irregular size with a range of approximately 1 m. According to Nicoleta and Maria (2015), the surface area of zeolite is influenced by particle/pore size, pore shape, and the arrangement of pores in the particles (Nicoleta Popa, 2015).

b) Metal Cu Adsorption Zeolite

Figure 3 below shows that the morphology of the zeolite containing Cu levels of 200 ppm is rectangular in shape with different size variations using a magnification of 5,000 times. There is a reasonably large pore on the sample's surface, which absorb Cu metal content in the zeolite.

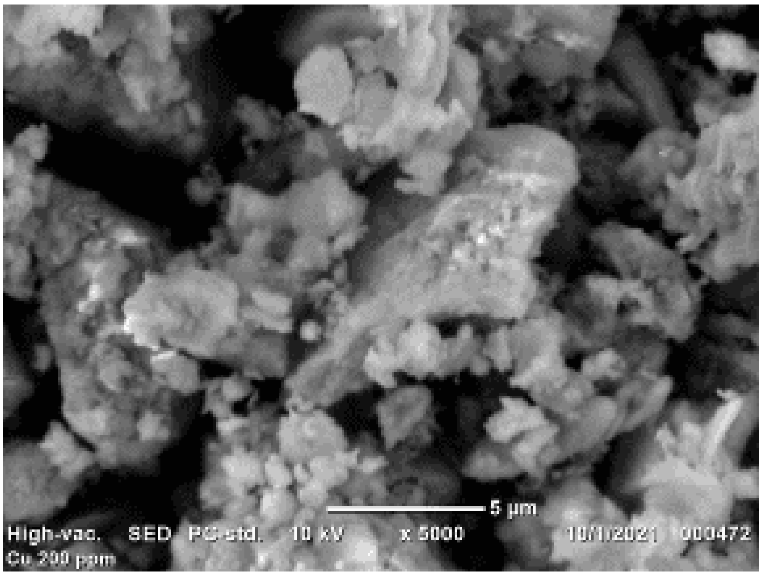


Figure 3. Results of SEM (Scanning Electron Microscopy) samples of 200 ppm Cu metal zeolite.

The morphology of the zeolite sample containing 400 ppm Cu metal is shown in Figure 4, with irregular flat shapes with varying sizes with very small pores on the surface of the zeolite with a magnification of 5,000 times.

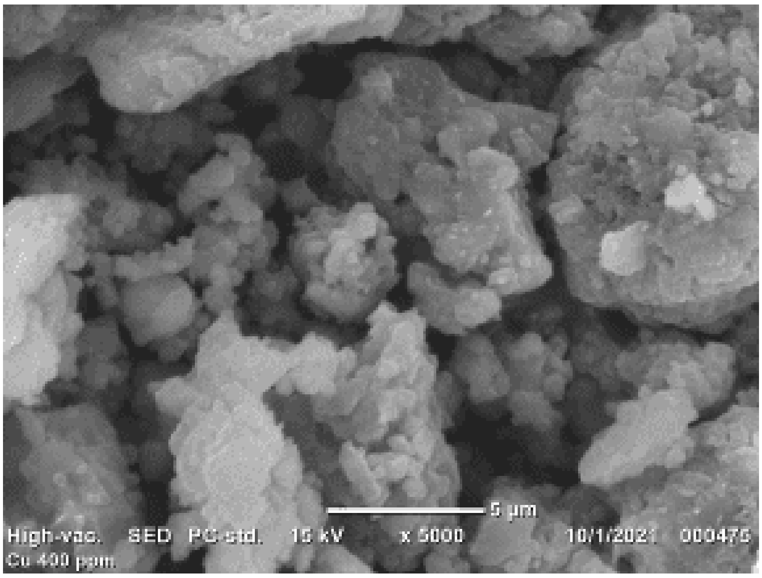


Figure 4. SEM (Scanning Electron Microscopy) image results of 400 ppm Cu metal zeolite samples.

Figure 5 can be seen from the morphology of the zeolite in the form of a flat shape with uneven pores on the surface.

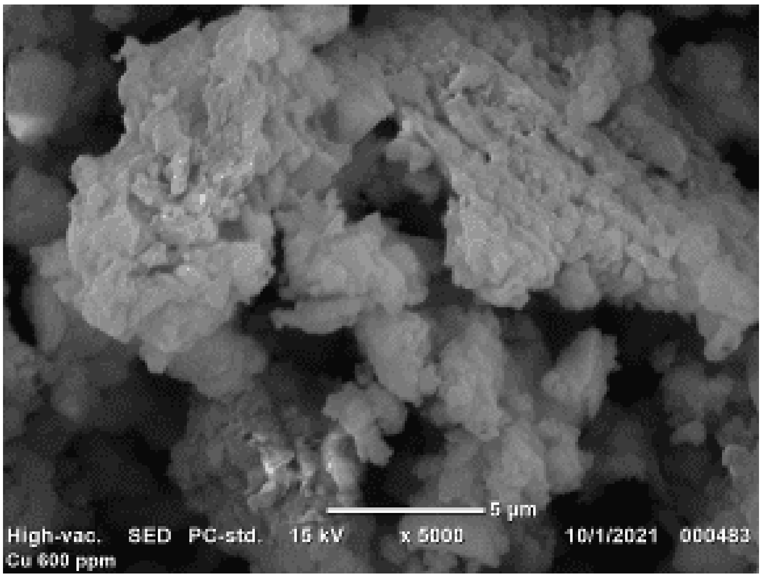


Figure 5. Results of SEM (Scanning Electron Microscopy) images of 600 ppm Cu metal zeolite samples.

It can be seen that the surface morphology of the zeolite samples with Cu metal adsorption with various ppm variations multiple sizes vary with a size of approximately 5 µm. There is also from each morphological image of the Cu variation, and it can be seen that pores are in each sample. Of the three variations of the adsorbed Cu metal, Cu with a deviation of 600 ppm seems to have a reasonably large pore among the three, whereas if viewed from the adsorption results using AAS, it is the 600 ppm Cu variation that absorbs the most Cu where the Cu content contained in this sample of 57.43 ppm with an absorption capacity of 0.0840. In addition, the decrease in the size of the adsorbent causes an increase in the pore volume and diameter. This tends to increase the adsorption capacity of the zeolite to heavy metals (Setiawan et al., 2020).

c)Metal Pb Adsorption Zeolite

From Figure 6 below, it can be seen that the morphology of the 200 ppm Pb zeolite is flat with different sizes with the presence of pores on the surface.

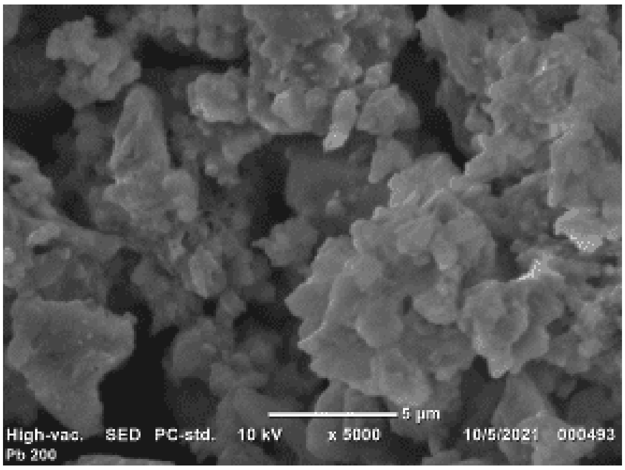


Figure 6. Results of SEM (Scanning Electron Microscopy) sample of 200 ppm Pb metal zeolite.

It can be seen from the morphology of the zeolite sample with a metal content of 400 ppm Pb in Figure 7 in the form of varying particles, and there is a cubic shape between them and the presence of pores on the surface of the sample.

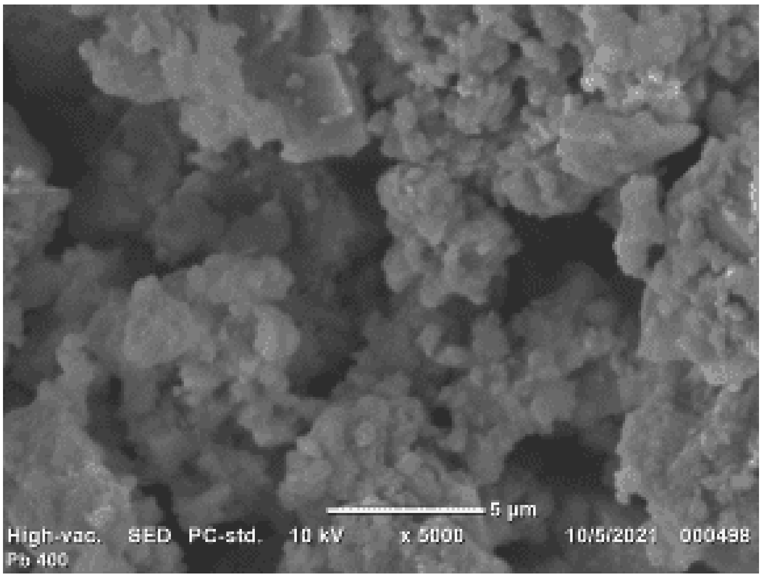


Figure 7. Results of SEM (Scanning Electron Microscopy) samples of 400 ppm Pb metal zeolite.

Figure 8 shows that the morphology of the 600 ppm Pb metal zeolite sample contained a needle-shaped growth caused by the dominant silica content in the sample and several pores.

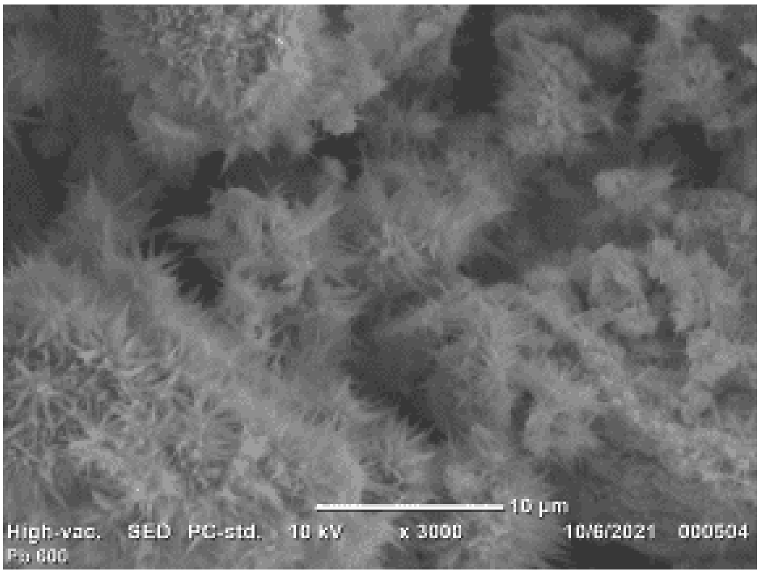


Figure 8. SEM (Scanning Electron Microscopy) image results of 600 ppm Pb metal zeolite samples.

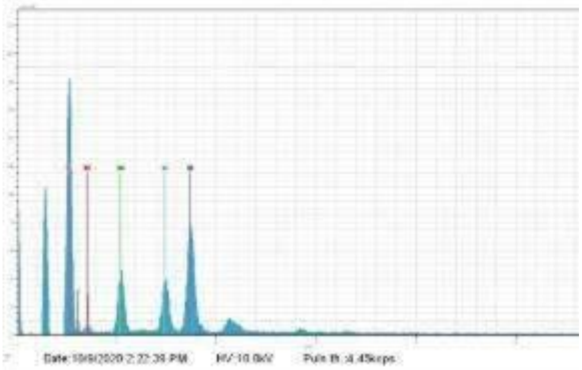
Based on the morphological results of the variation of the given Pb content, various types of sample sizes were seen where there is a flat shape and the shape of the needles surrounding the zeolite sample with a Pb content of 600 ppm. It is also seen in the presence of pores in each sample. It can be seen that with the number of pores or the more extensive the pores formed, the more power is absorbed by the Pb metal.

The pore structure of this zeolite can be applied as an adsorbent. With a finer mesh, the pores that are owned are also increasing, and the absorption surface area is also getting bigger, affecting the adsorption activity. The smaller the size of the zeolite, the greater the potential for particle aggregation so that it can close the adsorbent's playful side in heavy metals' adsorption and reduce the adsorption capacity of the adsorbent. (Hossain et al., 2012).

EDS-Mapping Results

a) Zeolite Y

EDS analysis is needed to determine the elemental composition of zeolite Y content from the synthesis of rice husk ash and sidrap clay shown in Figure 9. It shows that the Si element from zeolite Y has a higher percentage than Fe of 7.95%, Na of 6. .16%, and Al of 4.80%.



El	AN	Series	Unn.C [wt.%]	Norm.C [wt.%]	Atom.C [at.%]	Error [&]
O	8	K-series	55,74	66,04	78,60	7,0
Si	14	K-series	12,70	15,05	10,20	0,6
Fe	26	K-series	6,71	7,95	2,71	0,5
Na	11	K-series	5,20	6,16	5,30	0,3
Al	13	K-series	4,05	4,80	3,39	0,2
		Total :	84,40	100,00	100,0	

Figure 9. Results of EDS (Energy Dispersive X-Ray) Zeolite-Y

In Figure 10, a mapping analysis is presented, which is a mapping or distribution of the elements contained in zeolite Y. From the picture, it shows evidence that the distribution of Fe elements is red, Na elements are green, Si elements are blue, and Al elements are yellow and purple element O.

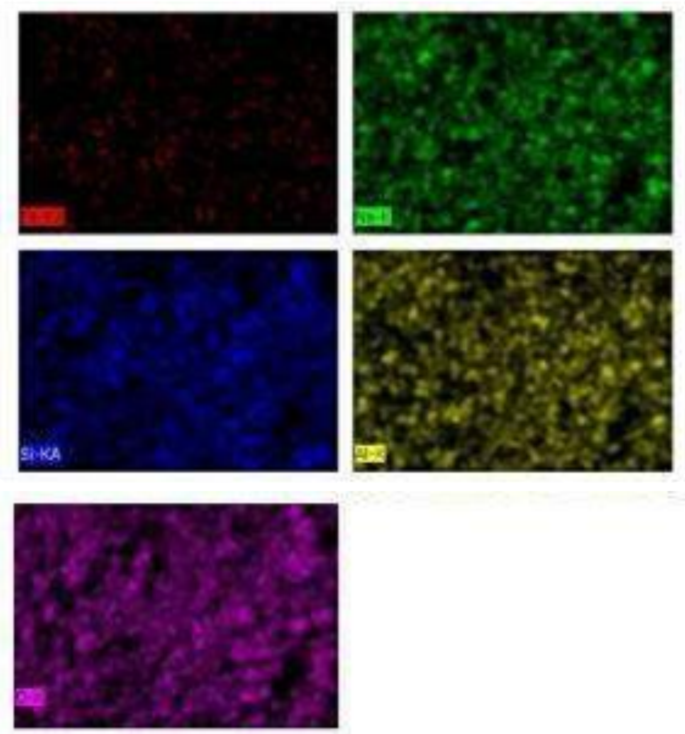


Figure 10. Results of Mapping Zeolite Y

b) Zeolit Adsorption Cu 600 ppm

Figure 11 the results of EDS samples of zeolite adsorption Cu 600 ppm. The content contained is dominated by Si at 30.65%, Al at 7.56%, Ca at 0.10%, Fe at 6.39%, and Cu at 8.04%. This shows that the zeolite has succeeded in absorbing the Cu content.

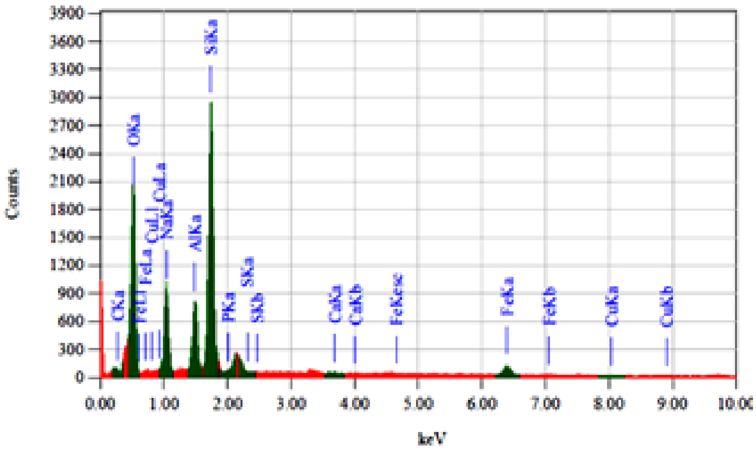


Figure 11. Results of EDS (Energy Dispersive X-Ray) Zeolite adsorption Cu 600 ppm

Table 4. Results of EDS (Energy Dispersive X-Ray) Zeolite adsorption Cu 600 ppm

Element		(keV)	Mass % ND	Counts	Sigma	Mol% ND	Compound	Mass % ND	Cation	K
C K			ND			ND		ND		
O			46,52					ND		
Na K^+		1,041	8,10	5982,54	0,21	11,47	Na_2O	10,92	2,91	0,9481
Al K		1,486	7,56	5430,59	0,31	9,12	Al_2O_3	14,29	2,31	0,9748
Si K^+	(Ref.)	1,739	30,65	21459,71	0,71	71,04	SiO_2	65,57	9,01	1,000
P K			ND			ND		ND		
S K			ND			ND		ND		
Ca K		3,690	0,10	42,56	0,08	0,17	CaO	0,14	0,02	1,6953
Fe K^+		6,398	6,398	1242,86	0,36	7,95	FeO	8,78	1,01	3,8436
Cu K^+		8,040	8,040	24,35	0,18	0,25	CuO	0,30	0,03	6,9469
Total			100,00			100,00		100,00	15,29	

The mapping analysis of Figure 12 on the 600 ppm Cu adsorption zeolite sample shows the distribution of the elements contained in the sample, namely Cu, Fe, Al, P, C, and O.

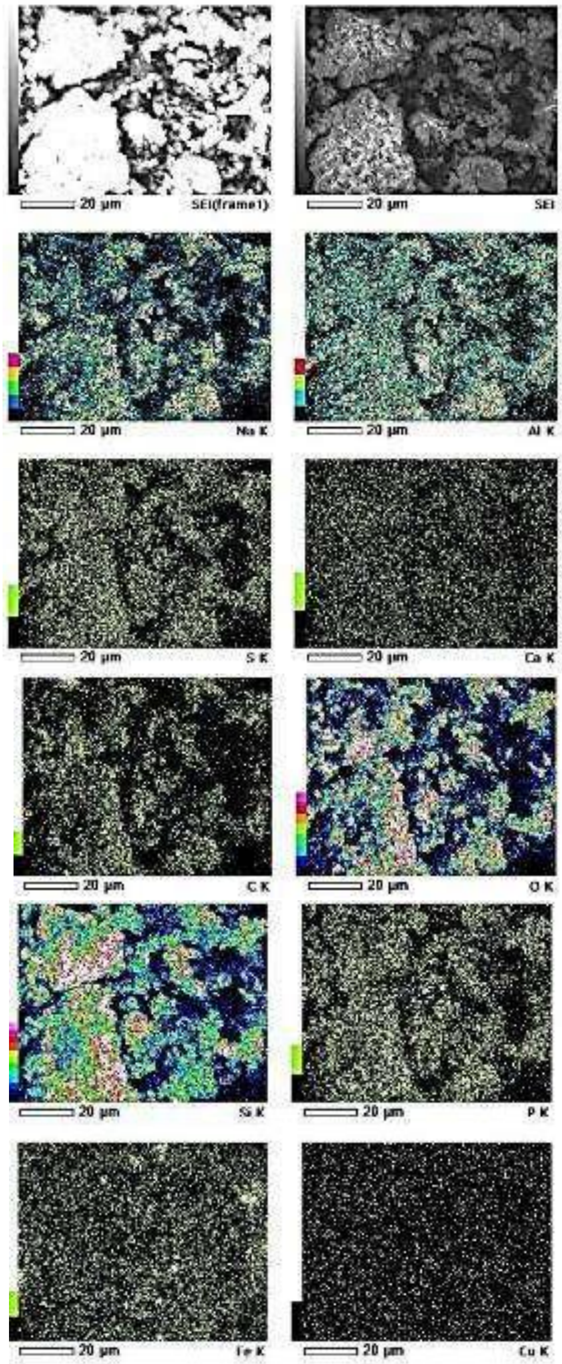


Figure 12. Results of mapping Zeolite adsorption Cu 600 ppm

c) *Zeolit Adsorption Pb 600 ppm*

Based on the results of the EDS in Figure 13, it can be seen that the metal adsorption zeolite content of Pb 600 ppm consists of elements of Na of 30.06%, Al of 6.85%, Si of 16.90%, Ca of 0.05%, Fe of 7, 86, and Pb of 0.20%. Pb content in the sample indicates that the zeolite can adsorb Pb metal well.

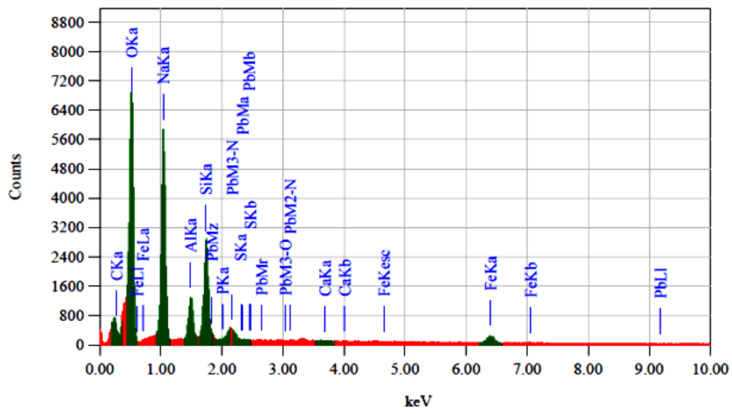


Figure 13. Results of EDS (Energy Dispersive X-Ray) Zeolite adsorption Pb 600 ppm

Table 5. Results of EDS (Energy Dispersive X-Ray) Zeolite adsorption Pb 600 ppm

Element		(keV)	Mass % ND	Counts	Sigma	Mol% ND	Compound	Mass % ND	Cation	K
C K			ND			ND		ND		
O			38,09					ND		
Na K+		1,041	30,06	37604,71	0,30	42,87	Na_2O	40,53	13,18	1,000
Al K		1,486	6,85	8327,67	0,23	8,32	Al_2O_3	12,93	2,56	1,0281
Si K+	(Ref.)	1,739	16,90	20039,93	0,41	39,45	SiO_2	36,15	6,07	1,0547
P K			ND			ND		ND		
S K			ND			ND		ND		
Ca K		3,690	0,05	33,52	0,08	0,08	CaO	0,07	0,01	1,7880
Fe K+		6,398	7,86	2425,11	0,30	9,23	FeO	1,42	1,42	4,0539
Pb K+		2,342	0,20	48,58	0,22	0,06	CuO	0,01	0,01	5,0354
Total			100,00			100,00		100,00	23,25	

From Figure 14, it can be seen that the results of the mapping analysis of the 600 ppm Pb adsorption zeolite sample are shown in the distribution of the elements contained in the sample, namely Pb, Fe, Al, P, C, and O elements.

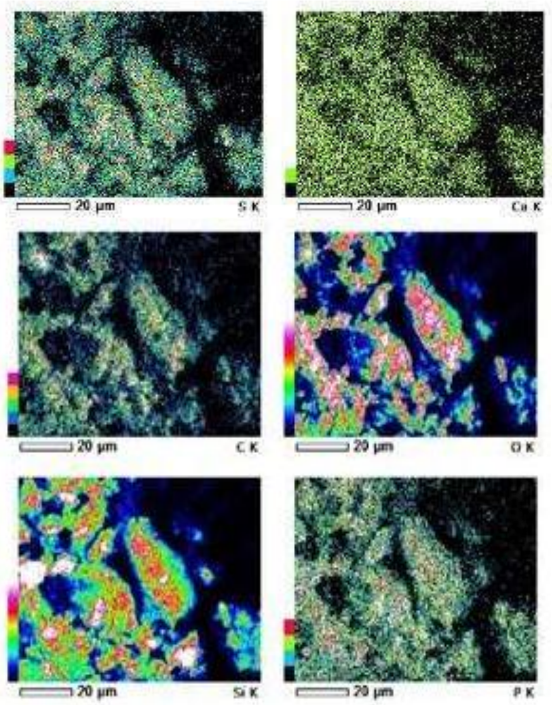


Figure 14. Results of mapping Zeolite adsorption Pb 600 ppm

Based on the results of the EDS-mapping of Cu and Pb zeolite samples, it can be seen that the content contained in each 600 ppm sample is very little Cu and Pb in the zeolite, so during AAS testing, it can be seen that only a small amount of 600 ppm Cu is present. Detected is 57.43 ppm, and Pb 600 ppm is 488.91 ppm. This is because, at the time of the mixing process, the Cu and Pb powders are not perfect, so the particles are not completely dissolved, resulting in only a small amount of Cu and Pb content contained at the time of mixing the zeolite.

CONCLUSION

Based on the results of the study, it was found that zeolite-Y was able to adsorb Cu and Pb metals well, as evidenced by the presence of Pb and Cu in the mapping and EDS results as well as in the SEM images shown in the morphological image of each Cu and Pb metal sample, there were pores indicating the absorption of Cu and Pb metals in the zeolite. And the AAS results show that the greater the levels of Cu and Pb produced, the greater the level of absorbent absorbed. Namely, each 200 ppm Cu sample with 47.56 ppm Cu content can be absorbed by the adsorption power of 0.0830. Samples of Cu 400 ppm with a concentration of Cu with levels of 38.17 ppm can be absorbed by the adsorption power of 0.0213. and Samples of Cu 600 ppm with Cu content of 57.43 ppm can be absorbed by the adsorption power of 0.0840. Pb sample of 200 ppm with Cu content of 1491.76 ppm can be absorbed by the adsorption power of 0.0113. Samples of Pb 400 ppm with Pb levels of 842.65 ppm can be absorbed by the adsorption power of 0.0057, and Pb samples of 600 ppm with Pb levels of 488.91 ppm can be absorbed by the adsorption power of 0.0047.

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