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LITHIUM RECOVERY FROM LAPINDO SLUDGE WATER (BRINE) BY ADSORPTION METHOD USING MANGANESE DIOXIDE (MNO2) ADSORBENT

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ABSTRACT

This article discusses the recovery of lithium from Lapindo sludge water in Sidoarjo using the adsorption method with Manganese Dioxide (MnO2) adsorbents. This study aims to determine the optimal lithium recovery rate based on variations in stirrer speed and adsorbent concentration. The results of the analysis showed that the stirring speed of 150 rpm with an adsorbent concentration of 2.5 g/L gave the highest lithium recovery result, which was 53.2 ppm. In addition, the adsorption process follows the Freundlich model, showing that the adsorbent has a good capacity to bind lithium from the solution. This research provides new insights into the use of sludge water as an alternative source of lithium in Indonesia.

KEYWORDS *Lithium; Adsorption; Manganese Dioxide; Lapindo; Recovery; Freundlich.*

INTRODUCTION

Currently, Lithium is being mass-produced in many countries to be used as a raw material for battery manufacturing. Lithium batteries are commonly used as batteries for mobile phones and laptops, but are currently also being developed as raw materials for electric-powered cars. In Indonesia itself, lithium metal ore has not been discovered, but some regions such as Bangka Belitung, Aceh and Sumatra have the potential to have it, but it has not been explored further. In industrial development, Indonesia has established a Lithium battery material factory made from Nickel and Cobalt in Morowali district, Central Sulawesi in 2019. However, the production process is still in the assessment stage and will take a long time until we can enjoy the results of the production (Siregar, 2019). Therefore, an alternative way is needed to obtain Lithium so that Indonesia can also participate in developing the Lithium product industry.

Apart from mineral sources, Lithium can be obtained by carrying out a process *recovery* from alternative sources such as seawater, evaporation waste or

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salt field waste *(bittern)* and saltwater that comes out naturally or as a result of geothermal drilling (*brine)* (Manao, 2012). At present, the alternative source of Lithium raw materials from geothermal salt water *(brine)* is the most dominant because the production cost is cheaper. One of the water *brine* which is very abundant and has Lithium content in the Lapindo Porong Mud, Sidoarjo. This incident, which was once thought to be a disaster, can turn out to be a treasure for our country to be used as an alternative source of Lithium method *recovery*.

There are several methods *recovery* which can be used to recover Lithium from salt water *(brine)* Among them are selective extraction, deposition and adsorption. Among the many ways, Lithium separation through the adsorption method is the easiest and quite good technique to recover Lithium from *brine*. According to (Kim, 2020) the adsorption method has the advantage of being easy to apply in dilute solutions and has high selectivity. In addition, according to (Syauqiah, 2011) the speed of stirring affects the length of adsorption time, the faster the stirring, the shorter the adsorption time will be. Selective extraction has disadvantages, namely requiring expensive chelating materials, while the sedimentation method requires a longer time of about 1-2 years, low separation efficiency and requires a large amount of water (Kim, 2020). The adsorption carried out in recovering Lithium is carried out by the ion exchange method (Sumarno, 2012).

Recovery Lithium has been done by (Siregar, 2019) with materials *brine water* which comes from the geothermal spring source of Tirta Sanita Ciseeng, Bogor. This research was conducted using the application method. The first step is to find the initial level of Lithium at *brine water* through ICP-OES testing (*Inductively Coupled Plasma – Optical Emission Spectroscopy).* The results of the initial test on the sample using ICP-OES obtained a Lithium content of 17.27 ppm. This study uses fixed conditions, namely a temperature of 30°C and a stirring speed of 100 rpm. The variables of this study were the process time (0.5; 1; 2; 3; 4; and 24 hours) and the concentration of Hydrous Manganase Oxide adsorbents (5; 7.5; 10; 12.5; and 15 g/L). The results showed that the highest adsorption percentage was obtained at 7.28% at an adsorbent concentration of 7.5 g/L with an adsorption time of 1 hour. ICP-OES Testing (*Inductively Coupled Plasma – Optical Emission Spectroscopy)* It was carried out again after going through the adsorption process and obtained a result of 800-1000 ppm.

Then *recovery* Lithium has also been done by (Sumarno, 2012) with materials *brine water* which comes from the mud of Bledug Kuwu, Grobogan, Central Java. In addition to these materials, this study also uses synthetic salt water. This research was conducted using the precipitation method. The reagents used for this method are LiCl, NaCl, NaOH, HF, $Al2(SO4)_3$ and Aluminum Potassium Sulphate. This study uses fixed conditions, namely a sample volume of 250 ml and a pH of 8. The variables of this study were the type of material (Bledug Kuwu mud water and synthetic salt water), temperature (30°C and 40°C) and process time (1 and 2 hours). Test results obtained Lithium from water *brine* Bledug Kuwu sludge is 400 ppm while synthetic salt water is 220 ppm. The results of the study showed that the percent *recovery* by 90.60%.

Moreover *recovery* Lithium has also been done by (Sapputra, 2014) with water materials *brine* who came from Lapindo Mud, Porong, Sidoarjo. The method used is adsorption and desorption with Lithium Manganese Oxide Spinel

(LiMn2O4) as adsorbent. This study uses a fixed condition, namely a temperature of 200°C while the variable is the process time (24, 48, and 72 hours). The researcher also conducted an ICP-AES test to determine the lithium level that was successfully recovered with the results of 9,150 – 9,310 ppm. The results showed that the highest percentage of adsorption was obtained at 42.76% with a process time of 72 hours.

Based on some of the research above, we update the research *recovery* Lithium in water *brine* from Lapindo Mud, Porong, Sidoarjo using the adsorption method with Manganese Dioxide (MnO2) adsorbents. The fixed conditions we use are temperature (± 30°C) and time (1 hour). Meanwhile, the variables we use are the stirring speed of the magnetic stirrer (100 ; 150 ; 200 ; 250 and 300 rpm) and the adsorbent concentration (2.5 ; 7.5 ; 12.5 ; 17.5 and 22.5 g/L). The Lapindo mud that we will use as a material has been tested using the ICP-MS tool *(Inductively Coupled Plasma – Mass Spectrometry)* with a Lithium content of 0.241 ppm.

Based on research from (Kim, 2020) *recovery* Lithium using MnO2 adsorbents succeeded in increasing the concentration of Lithium ions up to 37 mM. Therefore, we chose MnO2 adsorbent which is one of the adsorbents that is selective for Lithium.

Purpose

To find the recovered Lithium levels from Lapindo mud water *(Brine)* by the adsorption method. Also to find out the rotational speed of the magnetic stirrer and the best concentration of MnO2 adsorbent to recover Lithium by adsorption method.

Benefit

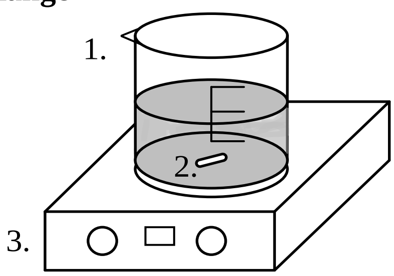
1. In order to be able to utilize Lapindo mud water which has been discarded as an alternative source of Lithium by adsorption method
2. In order to be able to streamline the adsorption process by knowing the best rotation speed (rpm) to recover Lithium.

RESEARCH METHOD

Material

The material used in this study is 500 mL of Lapindo mud water as the main material for the Lithium source that will be used in the *recovery*. In addition, an adsorbent in the form of MnO2 is used which is a specific adsorbent for Lithium with a concentration (g/L) of 2.5; 7,5 ; 12,5 ; 17.5 and 22.5.

Tool Range



Information:

Beaker glass

Magnetic stirrer

Hot plate

Fixed Conditions

1. The type of adsorbent used is MnO2
2. The type of material used is Lapindo *mud water (brine)* 500 mL

3. Temperature used ± 30°C
4. The duration of the adsorption used is 1 hour

Variable Conditions

1. Adsorbent concentration (g/L): 2.5 ; 7,5 ; 12,5 ; 17.5 and 22.5
2. Magnetic stirrer stirring speed (rpm): 100 ; 150 ; 200 ; 250 and 300

Trial Procedure

1. Prepare the tools and materials to be used.
2. Put 500 mL of Lapindo mud water into the glass beaker.
3. Place the *beaker glass* on the *hot plate* and insert *the magnetic stirrer*.
4. Put adsorbent concentrations (g/L) of 2.5 ; 7.5 ; 12.5 ; 17.5 and 22.5 into the *glass beaker.*
5. Carry out the adsorption process of Lapindo Mud *(brine)* water with predetermined conditions, namely a temperature of ± 30°C, a time of 1 hour and with a variation in the stirring speed of the magnetic stirrer (rpm) of 100 ; 150 ; 200 ; 250 and 300.
6. After the adsorption process is complete, filtration is carried out which will produce residues and filtrates. The residue was analyzed for elemental levels by *Inductively Coupled Plasma – Mass Spectrometry* (ICP-MS) to determine the Lithium content. Later, after knowing the concentration, it can be used to calculate the freundlich and langmuir models.

Lithium Analysis Using ICP-MS

1. The residual sample in the form of a mixture of adsorbent and adsorbate is aspirated and flowed through a capillary tube to the *nebulizer*
2. Inside the *nebulizer*, the sample is converted into an aerosol form
3. The aerosol will be carried by argon gas to the plasma
4. In plasma, aerosol particles are desolvated and sample molecules are dissociated into gaseous atoms, which are then excited and ionized
5. The ions go into the mass quadrupole and are separated by *the mass analyzer* based on their masses
6. Ions with the appropriate mass ratio emit their radiation. The radiation is detected and converted into an electronic signal that is converted into concentration information for quantitative analysis.

RESULT AND DISCUSSION

Results of Initial Lithium Rate Analysis in Lapindo Mud

Lapindo mud is first tested using the ICP-MS tool to determine whether there is Lithium content in the mud. After testing, the initial levels of Lithium in Lapindo Mud were obtained as follows:

Table .1 Results of Analysis of Initial Lithium Levels in Lapindo Mud

Test Sample	Test Parameters	Result	Unit
Lapindo Mud	Lithium	0,241	Ppm

(P.T. Angler BioChemLab)

Research Results

After preparing the materials and tools used, 500 ml of lapindo mud is put into the glass beaker. Then an adsorbent of Manganese Dioxide (MnO2) with a variation (g/L) of 2.5 is added; 7,5 ; 12,5 ; 17.5 and 22.5 into beaker glass. After that, the adsorption process is carried out using a magnetic stirrer with a stirring speed variation (rpm) of 100; 150 ; 200 ; 250 and 300 with a temperature setting of ± 30°C and within 1 hour. Next, a filtration process is carried out to separate the residue and filtrate. Residues were used to test the content of Lithium that had been recovered from the adsorption process using ICP-MS.

Results of Lithium Level Analysis

Based on the analysis test using ICP-MS, the lithium recovered from residues was obtained as follows:

Table 2. Results of Analysis of Recovered Lithium Levels

Adsorbent Concentration (g/L)	eStirring Speed (rpm)	Results of Lithium Terecovery (ppm) Content Analysis
2,5	100	19,40
	150	53,2
	200	3,6
	250	5,67
	300	11,3
7,5	100	14,5
	150	35,6
	200	15,6
	250	23
	300	17,9
12,5	100	17,4
	150	2,95
	200	5,78
	250	11,9
	300	8,35
17,5	100	7,13
	150	30,1
	200	8,03
	250	38,5
	300	13,2
22,5	100	6,49
	150	5,15
	200	3,78
	250	4,02
	300	7,44

(P.T. Angler BioChemLab)

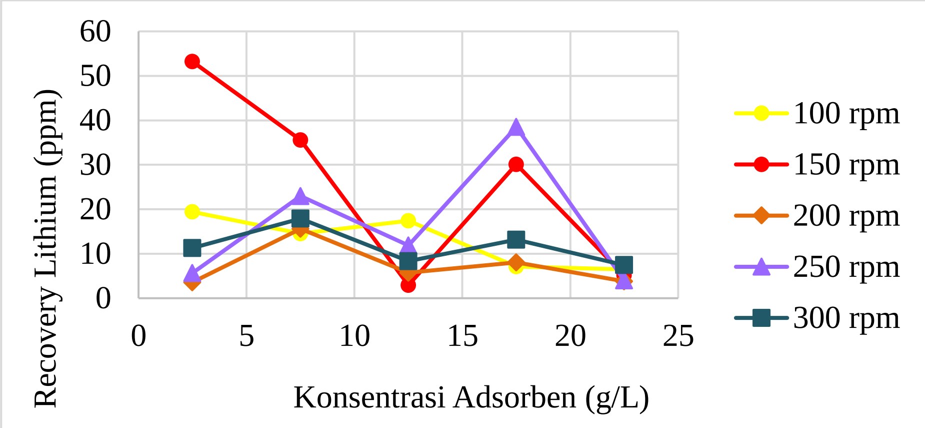


Figure 1. Relationship between Adsorbent Concentration (g/L) and Lithium Recovery Result at Stirring Speed Variation (rpm)

Discussion

Based on table IV.2 and figure IV.1, it can be seen that the rotation speed of 150 rpm has the best Lithium recovery result, which can be seen from the large lithium recovery result at the Adsorbent concentration of 2.5; 7.5 and 17.5 g/L which yielded 53.2 ; 35.6 and 30.1 ppm recovery Lithium. A rotation speed of 150 rpm with a variation in adsorbent concentrations of 2.5 and 7.5 g/L resulted in the highest recovery result among other rotational speeds with the same concentration, while with an adsorption concentration of 17.5 g/L the speed of 150 rpm ranked second after the speed of 250 rpm among other rotational speeds and with the same adsorbent concentration.

This can be due to the fact that the MnO2 adsorbent is likely to work most optimally to absorb Lithium adsorbates from Lapindo Mud water at a rotational speed of 150 rpm. If using a rotation speed below 150 rpm, the MnO2 adsorbent has not worked optimally to absorb its selective adsorbent, so the speed needs to be increased. However, if you use a rotation speed above 150 rpm, it is likely that the MnO2 adsorbent has experienced a decrease in absorption as a result of rotation that is felt to be too strong. Meanwhile, the speed of 150 rpm produces the highest overall recovery result at an adsorbent concentration of 2.5 g/L. This can be caused because this concentration is the best concentration for MnO2 adsorbents so that at that concentration a large amount of Lithium adsorbate is absorbed into the adsorbent surface. Increasing the adsorbent concentration above 2.5 g/L is likely to cause interference (disturbance) which can be caused by the interaction between the places of active adsorbent absorption so that there is a decrease in recovery results (Iftekhar, 2018).

From figure IV.1, it can also be seen that the increasing concentration of adsorbents, the recovery results of Lithium obtained increase and decrease (fluctuate). This can be caused by various factors including the type of adsorbate, namely Lithium in Lapindo Mud water. The mud we used was taken from a location quite far from the center of the eruption, which was about 700 meters from the center of the eruption and with a depth of 10 cm from the ground level.

Based on (Suprapto, 2012) the distribution of metal elements has a higher content value in the area of the center of the burst and is getting smaller in the area far from the center of the burst, and the content of the element Lapindo mud from time to time is not constant so that the value can fluctuate. Then based on the guide from Lapindo Mud, the area where we took the sample was mixed with rainwater, which could result in the value of the Lithium content in the sample we took getting smaller. The next factor is the temperature where during the adsorption process there is a possibility of an increase in temperature in the old magnetic stirrer so that according to (Syauqiah, 2011) a temperature that is too high can result in damage to the adsorbent so that the absorption capacity decreases.

Determination of the Type of Adsorption of Langmuir or Freundlich

The Freundlich and Langmuir equations function to estimate the surface area of adsorbents that can be used to bind adsorbates (Handayani, 2012). The higher the R2 value, the more the area of the binding field. Here is a table of the overall results of Freundlich and Langmuir's calculations:

Table 3. Results of Freundlich and Langmuir's calculations

No.	RPM	Konsentrasi Adsorben (g/L)	Recovery Lithium (ppm)	X	M	C	X/M	Freundlich		Langmuir	
								Log X/M	Log C	1/(X/M)	1/C
1		2,5	19,4	19,4	1,25	0,29041	15,52000	1,19089	-0,53699	0,06443	3,44341
2		7,5	14,5	15	3,75	0,33941	3,86667	0,58734	-0,46928	0,25862	2,94629
3	100	12,5	17,4	17	6,25	0,31041	2,78400	0,44467	-0,50806	0,35920	3,22155
4		17,5	7,13	7,1	8,75	0,41311	0,81486	-0,08892	-0,38393	1,22721	2,42066
5		22,5	6,49	6,5	11,3	0,41951	0,57689	-0,23891	-0,37726	1,73344	2,38373
6		2,5	53,2	53	1,25	0,04759	42,56000	1,62900	-1,32248	0,02350	21,0128
7		7,5	35,6	36	3,75	0,12841	9,49333	0,97742	-0,89140	0,10534	7,78756
8	150	12,5	2,95	3	6,25	0,45491	0,47200	-0,32606	-0,34207	2,11864	2,19824
9		17,5	30,1	30	8,75	0,18341	3,44000	0,53656	-0,73658	0,29070	5,45227
10		22,5	5,15	5,2	11,3	0,43291	0,45778	-0,33935	-0,36360	2,18447	2,30995
11		2,5	3,6	3,6	1,25	0,44841	2,88000	0,45939	-0,34832	0,34722	2,23010
12		7,5	15,6	16	3,75	0,32841	4,16000	0,61909	-0,48358	0,24038	3,04497
13	200	12,5	5,78	5,8	6,25	0,42661	0,92480	-0,03395	-0,36997	1,08131	2,34406
14		17,5	8,03	8	8,75	0,40411	0,91771	-0,03729	-0,39350	1,08966	2,47457
15		22,5	3,78	3,8	11,3	0,44661	0,33600	-0,47366	-0,35007	2,97619	2,23909
16		2,5	5,67	5,7	1,25	0,42771	4,53600	0,65667	-0,36885	0,22046	2,33803
17		7,5	23	23	3,75	0,25441	6,13333	0,78770	-0,59447	0,16304	3,93066
18	250	12,5	11,9	12	6,25	0,36541	1,90400	0,27967	-0,43722	0,52521	2,73665
19		17,5	38,5	39	8,75	0,09941	4,40000	0,64345	-1,00257	0,22727	10,05935
20		22,5	4,02	4	11,3	0,44421	0,35733	-0,44693	-0,35241	2,79851	2,25119
21		2,5	11,3	11	1,25	0,37141	9,04000	0,95617	-0,43015	0,11062	2,69244
22		7,5	17,9	18	3,75	0,30541	4,77333	0,67882	-0,51512	0,20950	3,27429
23	300	12,5	8,35	8,4	6,25	0,40091	1,33600	0,12581	-0,39695	0,74850	2,49433
24		17,5	13,2	13	8,75	0,35241	1,50857	0,17857	-0,45295	0,66288	2,83760
25		22,5	7,44	7,4	11,3	0,41001	0,66133	-0,17958	-0,38721	1,51210	2,43896

After the calculation of the recovered Lithium content, the determination of Langmuir and Freundlich adsorption can be carried out through the following graph plots:

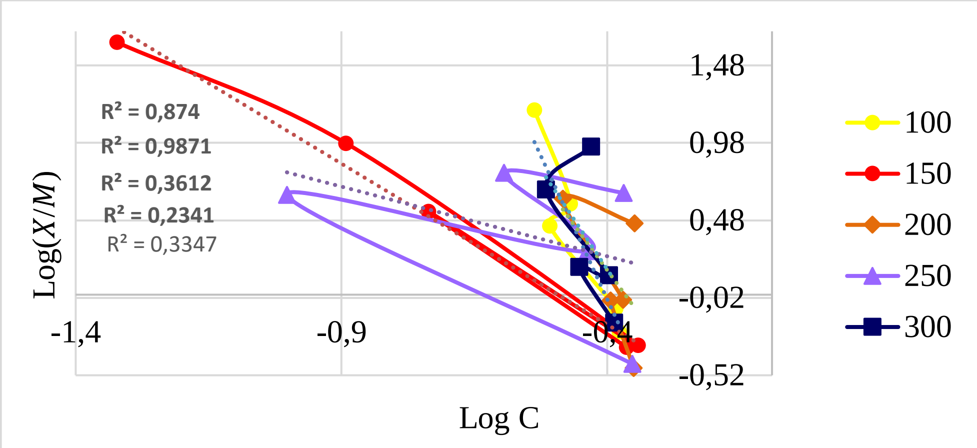


Figure 2. Determination of Freundlich Adsorption Type at Stirring Speed Variation

Based on the plotting of Freundlich's graph presented in Figure IV.2, it can be seen that the speed of 150 rpm has the highest R2 value compared to other stirring speeds, which is 0.9871.

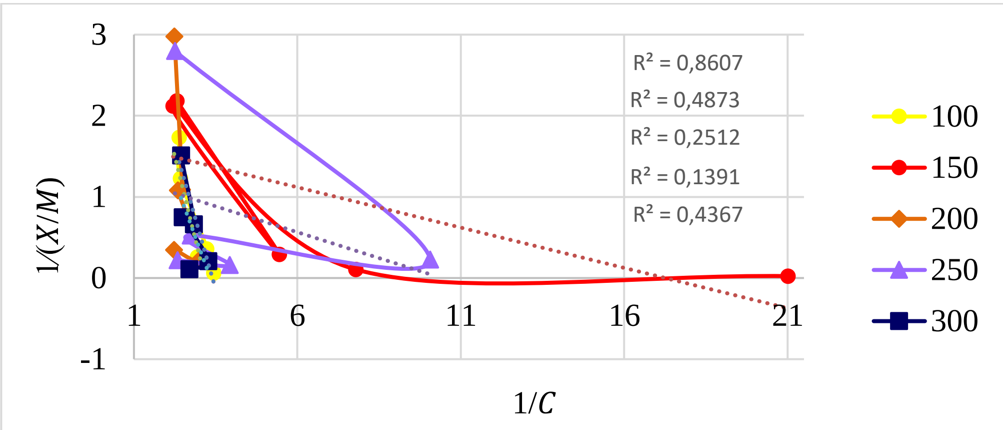


Figure 3. Determination of Langmuir Adsorption Type at Stirring Speed Variation

Meanwhile, in the graph of the Langmuir equation presented in Figure IV.3, the speed of 150 rpm has an R2 value of 0.4873. Therefore, it can be concluded that the adsorption experiment we conducted followed the Freundlich adsorption type with the equation y = -2.0763x – 1.0227 because overall the value of R2 using the Freundlich adsorption equation was higher compared to the Langmuir adsorption equation. It is also supported by the highest R2 result accuracy held by a stirring speed of 150 rpm, in accordance with the previous Lithium recovery results which have the best results. The experiment of one of the previous researchers (Siregar, 2019) also followed the Freundlich adsorption equation because the R2 value in the Freundlich adsorption graph was 0.899 while the R2 in the Langmuir adsorption graph was 0.866.

CONCLUSION

Based on the research that has been conducted, it can be concluded that the best rotation speed is 150 rpm with an adsorbent concentration of 2.5 g/L and the Lithium adsorption process from Lapindo Mud Water follows the Freundlich type of Adsorption with the equation y = -2.0763x – 1.0227.

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