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Hydrometallurgical Recovery of Lithium Oxide from Mud Sidoarjo

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Abstract: One of the efforts to meet the limited supply of fossil fuels is to replace the use of electric vehicles. Lithium batteries are one of the energy storage media that are environmentally friendly and cost-effective. Lithium batteries contain lithium ions. There are two sources for obtaining lithium ions as raw materials for batteries: used and natural resources. A natural resource that can potentially contain lithium ions is Sidoarjo mud. This research aims to determine the percentage efficiency of lithium extraction by the hydrometallurgical method. Factors affecting the extraction process include temperature, extraction time, solid-liquid ratio, and concentration of lactic acid solvent. The best leaching conditions were obtained at a temperature of 90oC and a concentration of 0.9 M lactic acid of 69.82%..

Keywords: hydrometallurgical; recovery; lithium; mud Sidoarjo; sustainable

1. Prerequisites for the publication

The crisis of dominant primary energy sources such as fossil fuels, which are the basis of welfare¹⁾, economic movement and community development²⁻⁴⁾. In addition, the detrimental impacts that occur due to the use of fossil fuels are global climate change, health impacts, and soil and water contamination^{5,6)}. Development to produce comprehensive green energy and recycling continues to be a basis for sustainable development⁷⁻⁹⁾. One of the ways to overcome the demand for fuel and have environmentally friendly energy is by using lithium-ion batteries¹⁰⁻¹²⁾. A lithium battery, which is one of the media that functions as a potential energy store, is efficient, has a long service life, has high energy stability at high and low temperatures, and has superior performance^{12,13)}.

The development of lithium-ion batteries and demand continue to increase significantly¹⁴⁾. The electronics industry applies lithium-ion batteries to electronic devices, electric vehicles and large-scale energy storage^{15,16)}. Production of lithium-ion batteries is expected to reach 50 million units in 2025 and almost close to 140 million in 2030, with an average growth of nearly 30%^{17,18)}. However, with the increase in consumption comes an increase in lithium battery waste^{19,20)}. These used batteries can cause a waste of energy if they are still used and pose an environmental hazard if disposed of^{19,21)}. These used batteries contain many valuable metals, including Co, Ni,

Li, and C^{22,23)}. Therefore, these used lithium batteries must be processed to make them clean and efficient^{24,25)}.

Several methods are generally used to recycle metals in batteries and mineral rocks, namely pyrometallurgy²⁶⁾, hydrometallurgy²⁷⁾, and biometallurgy^{28,29)}. Generally, what is done chiefly to recover metals is hydrometallurgy³⁰⁾. However, these three methods have the advantages for hydrometallurgy of low energy use, high selectivity³¹⁾, and producing high-purity metals³²⁾. Pyrometallurgical methods can be used more profitably on an industrial scale (for large capacities)^{32,33)}. As for biometallurgy, only a little is used because the reaction rate is low, requires particular microorganisms, and is challenging to develop³⁴⁻³⁶⁾.

In the hydrometallurgical method, there are two types of solvents used, namely inorganic solvents such as HCl³⁷⁾, HNO₃³⁸⁾, H₂SO₄³⁹⁾, H₃PO₄⁴⁰⁾ and organic solvents such as citric acid⁴¹⁾, oxalate, and lactate⁴²⁾. Most of the metal extraction focus leads to environmentally friendly processes³¹⁾. Lactic acid is one of the organic solvents that can extract lithium.

Sidoarjo mud is a geothermal fluid containing high amounts of minerals^{43,44)}, especially lithium^{45,46)}. The results show that the lithium content in Sidoarjo mud reaches five ppm (mg/litre)^{47,48)}. So, the total lithium content in the Sidoarjo mud is estimated to be 10 tons annually^{49,50)}. This is an alternative to being used as a source of raw materials for lithium batteries and can

support the needs of lithium batteries^{24,51}).

Lithium extraction from Sidoarjo mud needs to be done because, in addition to being an alternative source^{52,53}) of raw material for lithium batteries, it can also increase the economic value of the Sidoarjo mud. The processing and use of environmentally friendly solvents are chosen to save costs and be efficient. This paper will review lithium leaching with the effects of operating variables such as leaching temperature, leaching time, solid-liquid ratio, lactic acid concentration, and stirring speed to obtain the best leaching process operating conditions for the highest percentage leaching efficiency.

2. Method

Material

The Sidoarjo mud used is shown in Fig. 1 with the ordinate point 7°31'53,472"5 on March 5, 2023, at 09.51. The mud is dried to $\pm 10\%$ and then reduced in size to 100 mesh. Mud with 100 mesh is sampled using the coning quartering method, which aims to homogenize the mineral metals in the mud. This representative sample was pre-analyzed with an Inductively Coupled Plasma Mass Spectrometer (ICP-MS)⁵⁴). In addition to mineralogical analysis, samples were subjected to powder X-ray diffraction (XRD)⁵⁵) and SEM-EDX analysis⁵⁶). Lactic acid, which functions as a solvent with an analytical class, is diluted using distilled water.

Leaching Experiments

The sample used for the extraction process was 10 grams in 100 mL of solvent. The mixture of sludge and acid solution is put into a closed beaker to avoid water loss caused by high temperatures and placed on a hot plate with a magnetic stirrer for stirring. The leaching solution was stirred at 300 rpm for 2 hours. The operating variables discussed are as follows: lactic acid concentration (0.1 – 0.9 M), solid/liquid ratio (S/L), temperature (30 – 90 °C) and reaction time (hours). The extraction results obtained were separated by filter paper. ICP-MS analyzed the resulting filtrate to determine the metal concentration after the extraction process⁵⁷). The research steps are shown in Fig. 2 while calculating the extraction efficiency using equation (1)⁵⁸):

$$\eta = \frac{CVD}{C_0m} \times 100\% \quad (1)$$

Where C is the Li concentration in the leachate (mg/L), V is the volume of the leachate (L), D is the dilution factor, C_0 is the Li content in the initial sample (mg/kg), and m is the mass of the lake sediment sample (kg).

3. Results and Discussion

Characteristics of Sidoarjo Mud

An analysis was carried out using ICP-MS to determine the initial content of Sidoarjo mud. The initial lithium

content in the Sidoarjo mud was 99.3 ppm. The second characteristic is XRD, which is shown in Fig. 3. XRD analysis was carried out to show the crystallographic properties of the Sidoarjo mud. Based on the results of a simple quantitative analysis using Match 3 calculations⁵⁹), it shows that the quartz SiO_2 (silicate) phase dominates, namely 81%, then magnesium ferrite 25%, Albit 3%, and Fluorohectorite 1%^{60,61}). The XRD pattern shows that the sample has high crystallinity as indicated by the peak $2\theta = 24.97^\circ$ for SiO_2 , which dominates according to JCPDS 00-021-127. At $20^\circ - 30^\circ$, there is a maximum distribution peak, which means the SiO_2 is in an amorphous state. The second largest content is hematite (Fe_2O_3), with JCPDS No.00-036-1296 occurring at a peak of 20° .

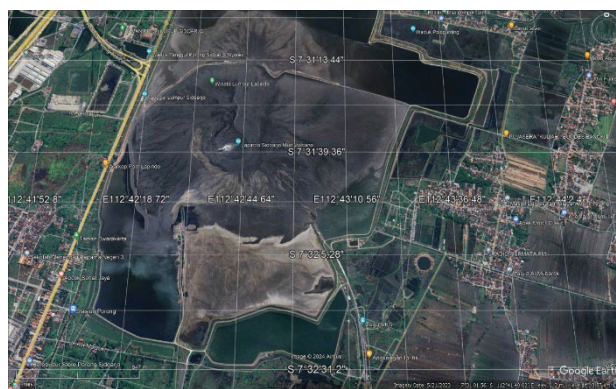


Fig. 1: The coordinate point for taking the Sidoarjo mud

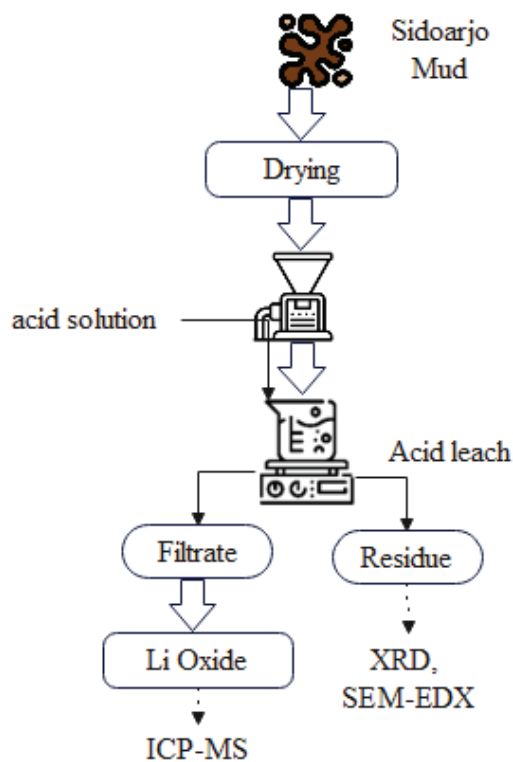
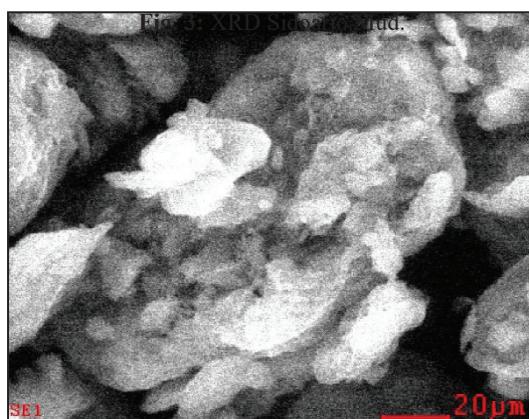
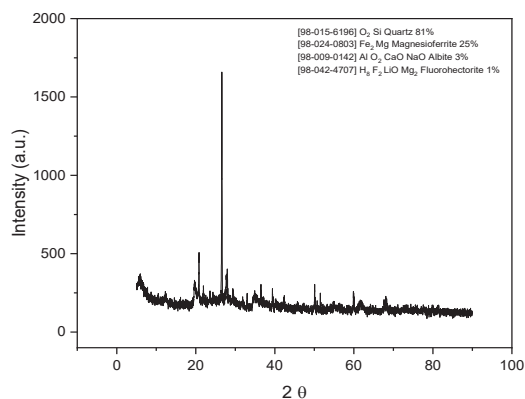


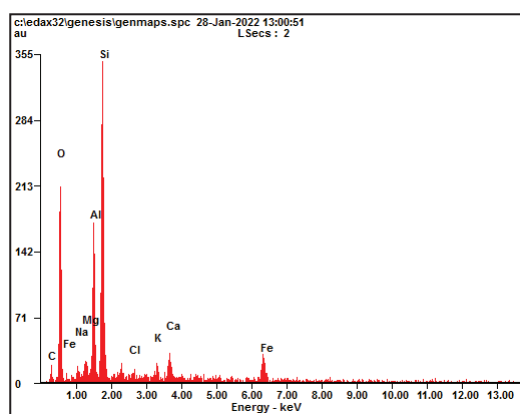
Fig. 2: Lithium leaching steps

Figure 4. a shows the results of the initial SEM-EDX analysis of the Sidoarjo mud at 5000 x magnification, which shows that the morphology is lumpy like clay^{62,63}).

The shape of the agglomerates in Sidoarjo mud is irregular and has a rough surface. Rough surfaces can absorb alkaline solutions and cause reduction. The main phase in the sample is quartz, where lithium can exist as physisorbed ions in this clay mud. This is also confirmed by Fig. 4. b with Si, Al and O peaks. The highest composition is the same as shown by the quantitative XRD analysis, namely Si (silicate).



(a)



(b)

Fig. 4: SEM-EDX Sidoarjo Mud (a), morphology (b), composition.

Effect of Concentration of Lactic Acid on Leaching

The effect of lactic acid concentration on leaching efficiency is shown in Fig. 5. Lactic acid concentration was varied from 0.3 M to 0.9 M, solid/liquid ratio 1:10

(gr/mL), stirring speed 300 rpm, reaction temperature from 30°C to 90°C, and reaction time for 2 hours. When the lactic acid concentration increased from 0.1 M to 0.9 M, the leaching efficiency increased, although not significantly. The lowest leaching efficiency was 48.77% at a temperature of 30°C with a concentration of 0.3 M lactic acid, while the highest occurred at a temperature of 90°C with a concentration of 0.9 M containing 69.82%. When the concentration of lactic acid is high, H^+ ions increase and can bind with Li^+ to form stable complex compounds^{64,65}. In the leaching process, hydrogen ions attack the metal and dissolve it into a solution. Therefore, optimal leaching efficiency occurs at a concentration of 0.9 M. Lactic acid is included in organic acids, which can act as a leaching agent in the leaching process. In addition, organic acids have the advantage that they are more thermally stable compared to inorganic acids and play an essential role as complexing agents^{66,67}.

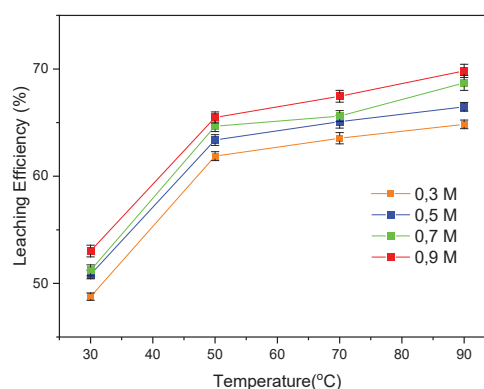


Fig. 5: Leaching Efficiency Lithium.

Effect of the Temperature on Leaching

The effect of temperature on leaching efficiency in the range of 30°C – 90°C is presented in Fig. 5. It is clear that when the leaching temperature increases, the leaching efficiency curve also increases. It can be explained that increasing the reaction temperature can accelerate the leaching reaction rate^{68,69}. So, a higher leaching reaction rate can increase the leaching efficiency^{70,71}. The best conditions for lithium leaching efficiency were used at 90°C, which was 69.82%. High temperatures can melt mud particles so that the lithium metal contained in the mud is easily extracted^{43,72}. Besides that, this is because the Li metal leaching process is an endothermic reaction^{73,74}. Temperature also produces activation energy, impacting the chemical reaction between Li ions and hydrogen ions. So, the existence of high temperatures can provide benefits in leaching efficiency. However, temperature has positive and negative influences, determined by another parameter, time⁷⁷. In the leaching process, the maximum temperature is limited to less than 100°C, this is related to preventing the evaporation of water in the leaching solution.

4. Conclusion

This work proposes an environmentally friendly hydrometallurgical process to extract lithium from the Sidoarjo mud, which can impact economic value. This leaching process is much more straightforward by direct contact of lactic acid with Sidoarjo mud without pretreatment, such as soaking or calcining. The results show that lactic acid concentration and temperature are essential in increasing leaching efficiency. Leaching efficiency reached 69.82% under 0.9 M lactic acid conditions at 90°C temperature. The proposed process can realize lithium recovery in Sidoarjo mud, which is clean, efficient and inexpensive, so it has broad industrial prospects. However, it is necessary to study further the role of stirring speed, solid/liquid ratio, and reaction time to obtain a higher leaching efficiency value.

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