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Effect of Leaching Temperature on Lithium Recovery from Li-Montmorillonite (Bledug Kuwu’s Mud)

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Abstract. The availability of lithium resources in the world is important along with the development of electronic technology and electrical transportation, especially lithium batteries. In Indonesia, lithium is found in brine associated with hot spring, clay associated with a geothermal activity, and spodumene minerals. The Bledug Kuwu’s Mud contained a Li-Montmorillonite phase with lithium content that was 0.0029%. For lithium recovery, Li-Montmorillonite was leached with distilled water on the S/L ratio was 1:2 for 3 hours. In this experiment, the leaching temperature was varied as follows: 25°C, 30°C, 35°C, and 45°C. Experimental results showed that lithium recovery from Li-Montmorillonite was successfully carried out by water leaching at atmospheric pressure. This result was supported by XRD analysis on residues. It indicated that there was no Li-Montmorillonite phase at the residues. The highest percentage of lithium recoveries in this research obtained at leaching temperature of 25°C was 87.2% (15.78 ppm).

1. Introduction
Lithium is critical metal that can be used as the future energy in the world. According to physical and chemical properties. Lithium is a lightweight metal that provides higher cell voltage, high specific energy, high energy density, high durability, discharge characteristics excellent, long service life, and high environmental compatibility when compared with alloys of lead, copper, vanadium, and nickel-zinc, [3]. Therefore, lithium is very suitable to be applied in the development of lithium batteries. Currently, the existence of lithium is one of the main focuses for technological development in several developed countries.

In recent years, worldwide lithium market has a deficit value due to high lithium demand, especially for battery applications. When compared with other applications, lithium-ion battery has the largest consumption about 39% of total lithium. Globally, the major types of lithium resources could be obtained from minerals, brine water, and clay (an example of hectorite groups) with lithium reserves most often found in Argentina, Bolivia, and Chile. In minerals, commercial lithium is usually found in spodumene (Li₂O·Al₂O₃·4SiO₂), lepidolite (KLi₁.₅Al₁.₅[Si₃O₁₀][F,OH]₂), and petalite (LiO₂·Al₂O₃·8SiO₂) and others. Lithium is evenly distributed on hard rock (25%), Hectorite (7%),
Geothermal Brines (3%), Oilfield Brines (3%), Jaderite (3%). Indeed, the number of clays containing lithium is lower than hard rock, which is only about 7% of total production. However, it has a possibility for technological development of lithium recovery from clays source, especially Hectorite form [1].

The process of lithium recovering from clay resource has been studied since the discovery of hectorite and Li-Montmorillonite which contained 0.56% Li by Lithium Americas Corporation (Project of King Valley Lithium). Hectorite clays belong to the clay of smectite group that is as same as Montmorillonite because they have a typical 2:1 type of hydrous aluminosilicate. Their framework of layers are composed by one octahedral sheet (mainly AlO₆ and MgO₆) sandwiched between two tetrahedral sheets (mainly SiO₄ and AlO₄ units). On the geological formation of Montmorillonite, the isomorphous substitution (Al³⁺ by Mg²⁺ or Fe²⁺ on anocahedral sheet) causes a negatively charged layer. It needs to be neutralized by exchangeable cations in the layers. The presence of inorganic cation substitution (eg Li⁺, K⁺, Na⁺, Ca²⁺, and Mg²⁺) into interlayer structure causes different thermal layer characteristic [2]. Besides that, Li-Montmorillonite after heating at 250-300 °C caused the migration of the lithium cations from the interlayer space into the hexagonal and octahedral site with occupancies of 60 and 40%. According to this concept, the temperature is very influential in the migration of lithium to the interlayer position from Li-Montmorillonite when leaching is carried out by using water.

In Indonesia, smectite groups found in Mud Volcano such as Lapindo Sidoarjo Clay (with contain 5 ppm Li) and Bledug Kuwu Clay (with contain 1.6 ppm). Bledug Kuwu contained Li-Montmorillonite phase. Rohmah (2018) found that solid/liquid ratio between Bledug Kuwu clays and distilled water affected lithium recovery, but, leaching time has no significant effect in the water leaching process [3].

On the previous research, lithium recovery increased with increasing temperature from 180°C until 250°C on the Egyptian Montmorillonite processing with H₂SO₄ leaching. So that, lithium recovery reached 90% at 250°C after 90 minutes [4]. So, in this research will aim to effect temperature on the lithium recovery for water leaching process.

2. Materials and Method
Raw material was the Montmorillonite-Containing Clay from Bledug Kuwu’s Mud, Grobogan, Central Java. The Li-Montmorillonite phase of the clay determined by X-Ray Diffraction (XRD) and identified by Software of High Score Plus (HSP). The XRD result is shown in Figure 1. Bledug Kuwu’s Mud is dominated by quartz compound and Li-Montmorillonite (40.1% semiquantitative with software Higscore). Based on Inductively coupled plasma - optical emission spectrometry (ICP-OES) test, Bledug Kuwu’s Mud contains 0.0041% Li at sludge condition and 0.0029% at slurry condition [3].

![Figure 1. XRD pattern of Bledug Kuwu’s mud [3]](image-url)
Bledug Kuwu’s Mud has done water leaching process with a variation of temperatures of 25°C, 30°C, 35°C, and 45°C at the solid/liquid ratio between clay and distilled water was ½ for 3 hours. A total of 250 grams of wet sludge was dissolved into 500 ml of distilled water, then stirred by using Mixer Settler MX machine brand EYELA. After the stirring process, the solution was filtered to separate the filtrate and mud solid for 3 days. The filtrate was tested by ICP-OES analysis to determine the chemical composition of filtrate so that could be calculated the percentage of lithium recovery. Meanwhile, the leaching residue was dried at 100°C for 2 hours. The dried mud residue was tested by XRD and SEM-EDS analysis to confirm the change of compound and morphology in the Bledug Kuwu’s Mud.

3. Results and Discussion

3.1 Effect of temperatures on the chemical composition of filtrates

The water leaching experiment was investigated to determine the optimum condition of lithium recovery from Li-Montmorillonite at different temperatures. Temperature affected significantly on the dissolution of lithium and associated impurity from Li-Montmorillonite, as shown in Table 1.

| Element (ppm) | Temperature (°C) |
|---------------|------------------|
|               | 25               | 30 | 35 | 45 |
| Aluminum (Al) | 0.004            | 0.042 | 0.033 | 0.037 |
| Ferrous (Fe)  | 0.001            | 0.006 | 0.003 | 0.002 |
| Magnesium (Mg)| 22.45            | 29.76 | 8.69 | 12.49 |
| Calcium (Ca)  | 62.72            | 72.01 | 30.78 | 38.92 |
| Sodium (Na)   | 1360.26          | 1419.82 | 1726.07 | 1311.35 |
| Potassium (K) | 135.62           | 179.91 | 183.63 | 155.16 |
| Lithium (Li)  | 15.78            | 15.11 | 4.36 | 9.29 |

Based on Table 1, the lithium recovery tends to decrease with increasing the leaching temperature. The highest acquisition of lithium is obtained at a leaching temperature of 25°C which is 15.75 ppm Li. As viewed in Table 1, the acquisition of Li at leaching temperatures of 25°C and 30°C is not significant or tends to be constant at 15 ppm Li. The recovery of lithium is seen to decrease at a temperature of 35°C that the lowest lithium content is approximately 4.36 ppm. At 45°C leaching temperature looks a recovery of lithium in the range of 9.29 ppm.

The decrease of lithium recovery is due to the desorption competition of Na and K ions which tends to increase solubility with increasing the leaching temperatures. The acquisition of K and Na in filtrate reaches a maximum point at 35°C. This condition causes the exchangeable rate of Li with hydroxyl ions on the active side of clay would be disrupted. The Na and K ions might be in 2 side of the active surface (side and side edge) due to electrostatic repulsion so that the solubility of Na and K of Li-Montmorillonite in water is increased (more than 1700 ppm Na and more than 180 ppm K) [5,6]. In addition, the simulation results of HSC Chemistry software proved that lithium solubility in water decreased as temperature increased. It was showed from data of log K values at 25°C, 30°C, 35°C, and 45°C that were 70.07; 69.59; 68.44; 67.36; and 66.31, respectively. It can be explained that the lithium acquisition decreases at warm temperature.

The trend of lithium acquisition almost is as same as Ca and Mg acquisition. However, it is different from K and Na acquisition. The warm temperature increases the adsorption level of the hydroxyl ion and monovalent element in the Li-Montmorillonite structure. It is caused by the bonds on the Li-Montmorillonite interlayer that will be very weak with the existence of competitive adsorption processes of Na and K ions[7].

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To investigate the effectiveness of the effect of temperatures on the optimum lithium recovery from the water leaching of Bledug Kuwu's Mud for 3 hours, it was necessary to calculate the selectivity ratio of impurities to lithium in the filtrates as shown in Figure 2. From Figure 2 can be seen that the optimum leaching conditions are obtained at a temperature of 25°C. This has the lowest Na/Li ratio of 86.2; K/Li of 8.59; Ca/Li of 3.97; and Mg/Li of 1.42. The following is the sequence of obtaining impurity levels as follows: Na/Li > K/Li > Ca/Li > Mg/Li > Al/Li = Fe/Li at water leaching of Bledug Kuwu’s Mud for 3 hour. The lowest selectivity ratio of impurities to lithium shows that the filtrate produced by the water leaching process of Bledug Kuwu’s mud at 25°C is rich in lithium content.

3.2 Effect of temperatures on the leaching residues
The effect of temperature on the residues from leaching Bledug Kuwu’s Mud for 3 hours was investigated with the characterization of residues by using XRD analysis as shown in Figure 3.

According to Figure 3, there is phase changes between the initial raw material of Bledug Kuwu’s mud and residues from water leaching processes. In initially Bledug Kuwu’s mud appears the peaks with the intensity of phases of Li-Montmorillonite, SiO₂, feldspar and Potassium Borondisilicate that transforms phases into peaks with intensities of SiO₂, H₂Al₃KO₁₂Si₃, Al₂O₃, and Al₃B₂K₂O₇. The
change of phase is indicated by the dissociation reactions between Li-Montmorillonite, feldspar, and Potassium Borondisilicate with water solvents at various temperatures of 25°C, 30°C, 35°C, and 45°C.

The leaching process of Li-Montmorillonite was very dependent on the interaction of the structure of clay with water or during the molecular hydration process. The initial cations in the Montmorillonite interlayer would react with water molecules so that the interlayer distance was wider. Ions with high hydration numbers tended to form outer sphere complexes that only attached to the surface of clay, such as Li and Na ions. While ions of K, Mg, Ca tended to form the inner sphere complex [8]. The Li-Montmorillonite leaching reaction in the Bledug Kuwu’s mud using water medium was likely to be predicted as follows:

\[
H_2Al_{1.31}Fe_{0.63}Li_{0.65}Mg_{0.54}O_4Si_{7.8} + 6Na^+(aq) + 19H^+(aq) \rightarrow 3.21Al(OH)_4^{4-} + 0.45Fe^{3+} + 0.62Li^+ + 0.54Mg^{2+} + 7.8HSiO_3^{3-} + 14OH^{-}(aq)
\]  

(1)

Reaction (1) referred to the results of previous research by Takase et al (2004) [9] which explained the Montmorillonite reaction in a variety of neutral, acidic, and alkaline media. In other conditions, Hellman (2009) described that the dissolution of feldspar was influenced by pH, temperature, and solution composition [10]. Feldspar can be modified in distilled water media so that the possibility of a reaction that occurred as in Reaction (2). The leaching of feldspar also affected the content of compounds in the leaching residues. Therefore, the leaching residues tended to form Al-Si-K compounds.

\[
Na_xK_yCa_{a-x}Al_{1.31}Si_{7.5}O_{15}(s) + 8H_2O(l) \rightarrow xNa^{+}(aq) + yK^{+}(aq) + zCa^{2+}(aq) + (1+2)[Al(OH)_4]^{2-}(aq) + (3-z)H_2SiO_3(s)
\]  

(2)

In an effort to investigate in detail the content of the compounds contained in the leaching residues of the Bledug Kuwu’s mud with water, the results of XRD analysis needed to be further calculated using the High Score Plus (HSP) software. The software could interpret quantitative of each phase of leaching residues as shown in Table 2

| Table 2. Quantitative Analysis of phase of leaching residues in variation temperatures |
|-----------------------------------------------|-----------------------------------------------|-----------------------------------------------|-----------------------------------------------|
| Temperature | SiO₂ (ICDD no 98-002-9210) | H₂Al₃KO₂Si₃ (ICDD no 98-002-7170) | Al₂B₂K₂O₇ (ICDD no 98-040-9420) | Al₂O₃ (ICDD no 98-016-5594) |
| 25°C | 25% | 39% | 31% | 5% |
| 30°C | 23% | 33% | 30% | 14% |
| 35°C | 41% | 29% | 18% | 12% |
| 45°C | 18% | 47% | 21% | 17% |

In Table 2 could be seen that leaching residues are dominated by SiO₂ and H₂Al₃KO₂Si₃ phases according to the XRD pattern in Figure 3. They have the highest peak intensity. Whereas the peaks showing the Al₂B₂K₂O₇ and Al₂O₃ phases also appear in the XRD pattern in Figure 3, but have a lower relative intensity. Therefore, to prove the existence of this phase, it is necessary to know the content of the chemical elements in the residues. The chemical composition of the residues with semi-quantitative can be further confirmed by EDS-SEM analysis.

3.3 Morphology of leaching residues

Figure 4 shows the morphological changes of the particles in the leaching residues. It looks that water leaching process at 25°C is capable of breaking the particle grains into the smaller size (4a). However, increasing temperature up to 45°C cause the particles to re-agglomeration to become larger (4b-d). The change in particle size indicates an increasing of adsorption level in the monovalent cation. Related to Mustopa (2013), morphological changes in sludge has not affected by alkaline or acidic conditions but more affected by temperature changes [11]. Al-Ani et al (2008) showed that
temperature effected the ability of Li-Montmorillonite in the water adsorption and expansion of adsorption surface. The higher the temperature, the greater the capacity of water adsorption, but tended to decrease after the temperature reached up to 50°C and depended on the composition of the exchangeable cation in the interlayer of Li-Montmorillonite [7].

Figure 4. The morphology of leaching residues in variation temperatures of : (a) 25°C (b) 30°C (c) 35°C (d) 45°C (Magnification 500X)

Figure 5. Elemental mapping of leaching residue at a temperature of 30°C(Magnification 500X)
In this discussion is only explained the elemental mapping of leaching residues at temperatures of 30°C (Table 5) and temperatures of 45°C (Table 6). It is clear that the particles of the leaching residue at room temperature at 30°C are relatively smaller compared to warm temperatures at 45°C. At room temperature of 30°C, the Bledug Kuwu’s mud is likely to be dissolved in water so much that the particles break into smaller sizes. This indicates that Li-Montmorillonite, which is a constituent of Bledug Kuwu’s mud, also dissolved in water. The larger solubility of Li-Montmorillonite in water at a temperature of 30°C causes lithium-rich filtrate compared to filtrate at a temperature of 45°C, as shown in Table 1. The same conditions may also occur in the leaching process of Bledug Kuwu’s mud at a temperature of 25°C where the morphology in Fig. 4a shows particles of smaller residue compared to particles of residue at 30°C. If observed more clearly in Figure 5 and Figure 6, it appears that the dominant constituent elements present in the leaching residues are Al, Si, and O. These elements are indicated as SiO₂ and H₂Al₃KO₁₂Si₃ which are constituents of leach residues. These compounds are difficult to dissolve at low temperatures, as shown in Table 2.

Table 3. The Chemical composition of residue (EDX-SEM Test)

| Element (%) | 25°C | 30°C | 35°C | 45°C |
|-------------|------|------|------|------|
| O           | 52.23| 44.35| 46.03| 43.64|
| Na          | 1.35 | 1.36 | 1.07 | 1.34 |
| Mg          | 1.31 | 1.42 | 1.29 | 1.37 |
| Al          | 8.85 | 9.30 | 9.79 | 9.10 |
| Si          | 20.28| 21.72| 22.06| 21.51|
| K           | 1.30 | 1.39 | 1.45 | 1.69 |
| Ca          | 10.01| 12.19| 12.07| 13.15|
| Fe          | 4.67 | 7.27 | 6.25 | 7.29 |
According to Table 3, the alteration of chemical composition based on the EDX-SEM analysis occurs on Na, Mg, Al, and Si elements. The content of Na and Mg decreases with temperature rose from 25°C to 35°C. However, the levels of these two elements increase at 45°C. The decrease of Na content to 1.07% and Mg to 1.29% proves that the rising temperature can accelerate the Na and Mg adsorption level so that both of these elements would be dissolved larger in water as shown in Table 1. That was supported the acquisition of decreased Li content due to competitive adsorption between Li, K, and Na to hydroxyl ions[6].

The trend of the alteration composition in Al and Si are inversely related to the trend of alteration in Na and Mg. The composition of Al and Si on the residue tends to increase to a maximum at 35°C and then decreases at 45°C. The alteration of these two elements (Al and Si) explains that the temperature can accelerate the absorption of hydroxyl ions in the sludge resulting in significant structural changes in the octahedral (Al³⁺) and tetrahedral (Si⁴⁺) layers of Li-Montmorillonite which are aluminosilicate compounds [7].

3.4 Lithium Recovery
The lithium recovery is calculated by using equation (3). The recovery of Bledug Kuwu’s mud leaching in distilled water medium as shown in Figure 7.

\[
\% \text{ Recovery Li} = \frac{\text{dissolved mass of Li in aquadest}}{\text{Initial Mass of Li}} \times 100\% \tag{3}
\]

Where dissolved mass of Li-ion (mg) which is the product of the concentration of Li (ppm) in the filtrate with the total volume of filtrate (ml). While the initial mass of Li (mg) is the product of the amount of Li (%) in the initial mass of the Bledug Kuwu’s mud sludge (gram).

![Figure 7. Effect of temperature on lithium recovery](image)

Figure 6 shows that the temperature has a relation with the percentage of lithium obtained from the Bledug Kuwu’s mud. The percentage of lithium recovery decreases with increasing temperature. Increasing temperature affects the adsorption level of H₂O in the clay structure. Bieski et al (2013) explained that higher temperatures could lead to higher surface area and modified the pore form of Li-Montmorillonite from crevice type to spheroidal, so that reaction rate was higher for Fe removal in acid treatment. Leaching with temperatures below 30°C was not capable of dissolving Fe. Meanwhile, the temperature leaching above 30°C (i.e at 35°C) tend to substitute higher Ca²⁺, Mg²⁺ and Na⁺ ions with oxonium ions [11]. Previous experiments conducted by Bieski et al (2013)[11] were different in conditions with this experiment where the increase in temperature caused a decrease in lithium recoveries from the water leaching of Bledug Kuwu’s mud for 3 hours. The percentage of maximum lithium recovery obtained in this experiment was 87.2% at a temperature of 25°C. This is due to the
temperature of 25°C, the solubility of Na and K ions is still relatively small in the water leaching of Bledug Kuwu’s mud so that the adsorption rate of Li with hydroxyl ions becomes higher.

4. Conclusion
From this experiment were obtained some conclusions as follows: recovery of lithium from Li-Montmorillonite was strongly influenced by leaching temperature. Lithium levels from filtrate tended to decrease with increasing temperature. The increase in leaching temperature above room temperature caused the higher water adsorption in the Li-Montmorillonite. This caused the interlayer bond of Li-Montmorillonite that tend to weaken so that the exchangeable rate of Li with hydroxyl ions on the active side of clay would be disrupted with increasing solubility of Na and K ions from Li-Montmorillonite. The water leaching at a temperature of 25°C gave a high Li solubility of 15.78 ppm, while at a temperature of 45°C gave a Li solubility of 9.29 ppm. From the XRD analysis on the residue, it was seen that the Li-Montmorillonite phase did not appear in the residue at 25°C. This indicated that lithium from Li-Montmorillonite was almost perfectly extracted. The peak intensity of SiO₂ and H₂Al₂K₀.₃Si₃ dominated the residue with increasing temperature. The morphology of the residue appeared irregular particles and occurred agglomeration with rising temperature. This experiment succeeded in recovering lithium from Li-Montmorillonite (Bledug Kuwu's Mud) by water leaching at atmospheric pressure where the percentage of lithium recovery was 87.2% at 25°C, 81.27% at 30°C and 46.75% at 45°C.

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