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TWO STAGE FROTH FLOTATION FOR SEPARATION OF SPODUMENE FROM PEGMATIC ORE IN THE EAST KAZAKHSTAN

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Abstract

TWO STAGE FROTH FLOTATION FOR SEPARATION OF SPODUMENE FROM PEGMATIC ORE IN THE EAST KAZAKHSTAN

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There are two main sources of Lithium: The first is brine and the second is pegmatic ore. Decades ago ore mining was out of commercial concern. However due to increasing price of lithium, the exploration and development of pegmatic ores became economically feasible. Therefore, the aim of this study is to explore a efficient flotation method for spodumene separation. The samples were delivered from Kazakhstan, near Altay mountain region. Prior to batch flotation, crushing and grinding was carried out in the jaw crusher and ball mill grinder. Using rotap appropriate size range was selected. In order to identify and measure the presence and amounts of minerals and their species in the sample, the XRD analysis was performed while the XRF analysis was used to examine the chemical composition of the
sample. Furthermore, the FTIR analysis was conducted in order to determine chemical bond and functional groups by their characteristic absorption of infrared radiation in vibrational modes.

The interaction of main reagents such as Sodium Oleate, DTAC, TBAB were studied separately and in mixed states. A few other cationic and anionic potential reagents were experimented and proved to be less functional than DTAC and Sodium Oleate respectively. The optimum concentration of each additive was determined experimentally. Their performance was evaluated on grade and recovery curve. It has been concluded that DTAC can be not only sub collector for main anionic collectors in a mixed flotation, but also happen to be a main single reagent in reverse flotation where valuable minerals report to tail part while gangues report to froth part. Reverse flotation did separate gangue from valuable mineral where tail part’s grade was 2.39% and recovery 83% while lithium oxide’s grade in the froth part only 0.67% and recovery 17%. Main reason for successful reverse and direct flotation lies in the interaction between DTAC with negatively charged quartz and oleate with aluminum atoms on the spodumene surface, respectively.

Keywords: lithium, grinding, spodumene, flotation, collectors

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1. Introduction

1.1. Background

Lithium is the 25th most abundant element in the earth's crust. Lithium finds an application in rechargeable lithium ion batteries (LIBs) because of its very high energy density by weight and high electrochemical potential (3.045 V). With a present consumption level of ~22% of the total lithium produced in LIBs, it is expected to reach to ~40% by 2020 (Wang et al., 2012). It is also understood that the demand for lithium is increasing further due to its application in nuclear and strategic areas. Every deposit has a various mineralogical composition and thus requires the appropriate technology to process. Demand from the lithium battery market from all applications including mobile devices like cell phones and tablets, e-bikes, hybrid and electric vehicles and large scale energy storage is growing at an astounding rate. The rising demand for lithium for various applications thus calls for prospecting and processing all viable resources. Lithium extraction from ores/minerals utilizes roasting followed by leaching, while its extraction from brines includes evaporation, precipitation, adsorption and ion exchange (Garrett, 2004).
The froth flotation process utilizes the natural hydrophobicity of grinded or liberated minerals after making them hydrophobic in order to precisely separate them from one another. The process was patented by E.L Sulman, H.F.K Pickard and John Ballot in 1906. The froth flotation flotation could save the extremely fine particles in the bulk. The principles of froth flotation are sophisticated combination of the laws of surface chemistry, colloidal chemistry, crystallography and physics, which are not clearly understood since discovery of the mechanism.

There are three flotation stages: roughing, cleaning and scavenging. The main aim of roughing is to recover as much of the valuable minerals as possible, not accounting grade of the concentrate produced. Cleaning, is performed to produce as high a concentrate as possible. Last but not least, scavenging is a
continue of the rougher flotation of tail part of rougher. The purpose is to recover any of the valuable particles that were not recovered during the initial roughing stage.

While reverse flotation is an opposite of flotation where valuable mineral does not float to froth part but sink to tail part. There are various reasons that lead to reverse flotation such as hydrophobicity and hydrophilicity shift using depressants or due to electrostatic attraction between reagent and solid surface. Detailed research is going to be discussed later in this study.
1.3. Literature review

Spodumene flotation is a substantial and challenging step for lithium recovery. The increasing demand for lithium requires exploitation of lithium resources. Spodumene (LiAlSi$_2$O$_6$), a silicate mineral with a monoclinic structure, is an important economic mineral of lithium-rich pegmatite ores (Zhu et al., 2015). Spodumene exists on the whole in granite pegmatite deposits, which contain feldspar, quartz, muscovite as well as some tantalite and niobite.

As a typical anionic collector, oleic acid occurs naturally in vegetable oils and animal fats from which they are extracted by distillation and crystallization. Oleic acid consists of a long hydrocarbon chain and a carboxylate functional group. A regular fundamental study of spodumene flotation using oleic acid as collector was carried out by Moon and Fuerstenau (2003), who reported both the maximum recovery of spodumene and the amount of oleic acid adsorption on the mineral exhibited at pH 8. The longer the hydrocarbon chain length of a collector, the more powerful is the water-repulsion produced, but solubility decreases. The broad application of pure or single surfactant, however, is not economical from an industry perspective due to its high dosage (Tiwari and Saha, 2013). A new single collector with a long development cycle has too high a cost to be used. A collector blend is a good choice for this purpose. Recent researches demonstrated that the flotation separation efficiency of minerals could be enhanced using a mixture of anionic collectors with anionic, nonionic and cationic collectors,
respectively (Sis and Chander, 2003a; Vidyadhar and Hanumantha Rao, 2007; Von Rybinski and Schwuger, 1986; Von Rybinski et al., 1987; Xu et al., 2013). Recently several researchers reported that collector blends would be often more efficient than single collectors in flotation of sulfide and oxide ores (Lotter and Bradshaw, 2010). The adsorption behavior of mixed surfactants has been investigated using MD simulation by several research groups (Chun et al., 2015; Wang et al., 2014a).

The spodumene and gangue minerals, such as feldspar and mica, have similar surface properties, that is, the Al site acts as the active center, the flotation separation of spodumene and gangue minerals using traditional anionic collectors, such as sodium silicate, naphthenic soap and fatty acid, is inefficient (Moon and Fuerstenau, 2003; Rai et al., 2011). Outcomes of surface chemical examinations indicated that the chemisorption of anionic oleic acid on the surface aluminum sites of spodumene surface was responsible for the best selective flotation of spodumene (Moon, 1985; Menendez et al., 2004; Yi, 2011; Zhong et al., 2012). There is a consensus that conditioning in highly alkaline solutions prior to flotation improves spodumene separation effectiveness.

Quaternary ammonium salts (QAS) are extensively used as an efficient collector for clay-type aluminosilicate minerals such as kaolinite, pyrophyllite and illite in Chinese diasporic bauxite reverse flotation (Longhua et al., 2015; Xu et al., 2004). It is reasonable to assume that it would interact with pegmatitic aluminosilicate such as spodumene and feldspar.
The floatability of minerals such as albite and spodumene are closely related to the number and type of exposed surface atoms, and this, in turn, depends on the crystal structure of the mineral and the characteristics of the chemical bonds. The surface bond properties of minerals can be used to predict and verify the cleavage properties of minerals and to predict the chemical reactivity of the surface atoms.

Surface crystal chemistry studies show that the planes (110) and (010) are the most likely cleavage planes for spodumene and feldspar, respectively. Moreover, the surface Al sites on the (110) cleavage plane of spodumene are the most favorable sites for selective chemisorption of oleate. These active Al sites in feldspar are buried deep inside the crystallographic unit cells, which makes them unavailable to adsorb oleate (Moon and Fuerstenau, 2003).

The Si–O bond in spodumene crystallographic structure mainly possesses covalent component, the average length of which is 0.158 nm, while the bond of Li–O and Al–O has obvious ionic features. The average bond length of Li–O is 0.221 nm, and for Al–O the value is 0.192 nm (Chuanyao and Wanzhong, 2001). The bond strength of Si–O is bigger than that of Li–O and Al–O, therefore, the disaggregation of spodumene would occur mainly along the broken directions of Li–O bond and Al–O bond. The exposed cationic ions on the disaggregated surface of spodumene would mainly be Li, Al and a bit of Si, which makes spodumene have a fairly good flotability by using anionic collectors.

In the flotation of spodumene ore, it has been reported the use of Fe(III) ions as an activator for spodumene, albite, and quartz.
The results showed that Fe(III) ions interacted selectively with the spodumene surface through chemisorptions; this interaction could alter the zeta-potential of the minerals in solution, which were conducive to the adsorption of an anionic collector. Flotation test results and diagram of Ca ion species distribution vs pH show that the dominant hydrolyzed species are Ca(OH)$^+$ and Ca(OH)$_2$, which is in correspondence with the maximum flotation recovery of spodumene. (F.S. Yu, Y.H. Wang, J.M. Wang, Z.F. Xie, L. Zhang. 2013). It has been commonly recognized that the interactions between minerals and the cationic collectors are both affected by physical electrostatic effects and hydrogen bonds. (Xia, L., Zhong, H., Liu, G., Huang, Z. and Chang, Q., 2009)

The reverse flotation for silica removal was developed as an economical way to improve the A/S ratio (Hu et al., 2003; Jiang et al., 2001). The adsorption of cationic collectors on dioxide minerals is governed mainly by the electrostatic interaction and hydrophobic interaction (Cases and Villieras, 1992). Cationic surfactants are organic compounds with a positive species in the solution adsorbing as active ions on the negatively charged surfaces of minerals. There is a nitrogen group with unshared electrons as a common member in their chemical structures (Bulatovic and Srdjan, 2010; Fuerstenau and Pradip, 2005).
1.3. Objective

The objective of this study is to design a new two-stage flotation circuit. Experiments were carried out in two steps. In the first step, appropriate amount of cationic collector was used to obtain maximum desilication, whereas in the second step desilicated feed was conditioned with anionic collector thus selectivity of valuable mineral could be achieved. The studied operating conditions were the various collectors with different amounts, bulk pH and pretreated vs not pretreated feed. Finally, all experiments were analyzed throughout inductively coupled plasma.
2. Fundamentals

2.1 Flotation theory

The flotation is a physicochemical separation process that utilizes the difference in surface properties of valuable minerals and the unwanted gangue minerals. The theory of froth flotation is complex, involving three phases (solids, water and froth) with many sub-processes and interactions, and still not fully understood. The attachment of valuable minerals to air bubbles is the most important mechanism and accounts for the majority of particles that are recovered in the concentrate. Although true flotation is the dominant mechanism for the recovery of valuable minerals, the separation efficiency between valuable minerals and gangue also depends on the degree of entrainment and physical entrapment. Unlike true flotation, which is chemically selective based on mineral surface properties, both gangue and valuable minerals alike can be recovered through entrainment and entrapment. In industrial flotation plant practice, entrainment of unwanted gangue can be common, and a single flotation stage is therefore uncommon (Napier-Munn and Wills, 2006).

Most minerals are not water-repellent in their natural state, and flotation reagents must be added to the pulp. The most important reagents are the collectors, which adsorb onto minerals surfaces, which render them hydrophobic (or aerophilic) and facilitates bubble attachment. The frothers help maintain a reasonably stable foam. Regulators are used to control the flotation process; these reagents either activate or depress
mineral attachment to air bubbles and are used to control the pH of the system (Napier-Munn and Wills, 2006).

2.1 Flotation illustrations

Firstly, ball mill was used for grinding, using conditions in Table 1, later using rotap sieve shaker - 0.150+0.038mm particle size range was obtained.

Table 1. Ball mill operation condition

<table>
<thead>
<tr>
<th>V</th>
<th>J</th>
<th>U</th>
<th>Apparent density</th>
<th>Balls weight, g</th>
<th>Feed, g</th>
</tr>
</thead>
<tbody>
<tr>
<td>5000</td>
<td>0.2</td>
<td>0.5</td>
<td>1.68</td>
<td>4722</td>
<td>317</td>
</tr>
</tbody>
</table>

In Figure 2 there is a schematic diagram of closed circuit flotation that is used in the laboratory scale to check collector efficiency.
By introducing a 50g of feed to a Denver Flotation Machine cell, the 5% bulk density was obtained. In the very beginning, MIBC used as a frother to maintain foam stability. Then sodium hydroxide and hydrochloric acid was used to reach desired pH of the bulk, pH conditioning time was 5 minutes. Then designated collectors added and conditioned for 5 minutes as well. Concentrate and tailings were filtered and dried in the oven under 100 Celsius degrees.

Figure 3 shows actual flotation scheme that used in the industry. Flotation was divided into two stages; first stage is reverse flotation where cationic collector was used to remove silica. In the second stage, direct oleate flotation has been applied to increase the grade. The froth part in the reverse cationic flotation contains much less Lithium oxide concentration.
comparing to tail part, however cationic flotation is not enough for high purity, thus a classical direct flotation in the second stage was applied. In the second stage, oleate flotation takes places only once in the froth part of the reverse flotation is because grade is already too low to apply second cycle. Whereas, the tail part of the reverse flotation has been floated twice. It should be noted that not a single activator or depressant was used during whole flotation cycle.
Figure 3. Industrial flotation scheme
Figure 4 shows the laboratory Denver flotation machine. Flotation experiments were carried out using 5% solids concentration. Maximum rotation per minute of flotation machine is 2200rpm, while experimental speed was constant at 1300rpm. The air valve was opened smoothly until the maximum during the actual flotation. It should be taken into account that if valve opened too quickly then bulk could spill over and mechanical flotation could take place which is experimentally wrong.

Figure 4. Denver flotation machine
Figure 5 demonstrates flotation illustration scheme of actual laboratory Denver flotation machine. Firstly, water and sample are poured to the flotation cell making up 5% solids, then by switching on the button shaft starts to rotate at the settled speed allowing reagents to condition in the bulk with solids. After appropriate conditioning air valve should slowly be turned on thus producing bubbles that will pick up hydrophobic particles up to the froth part.

Figure 5. Flotation illustration scheme
3. Materials and reagents

3.1 Ore sample

Samples were obtained from the Republic of Kazakhstan, in the East region bordering with Russia and China. The mine is located in Altay mountains area, approximately 100km from biggest city Oskemen. In the past times during Soviet Union, industries were interested in tantalum recovery only, while Lithium was out of commercial interest. Currently, mine is abandoned and has thousands tons of ore reserves estimated and much more to be estimated that will allow to profit on Lithium mining. Average grade of Lithium oxide is 1.5%.

The mineral sample used in the experiments was pegmatic ore which contained Quartz (SiO₂), Albite (NaAlSi₃O₈), Muscovite (KAl₂(AlSi₃O₁₀)(F,OH)₂), Spodumene (LiAlSi₂O₆).
According to XRD analysis, pegmatic ore contains 20.3% of spodumene which in terms of industry estimations, the mineral grade counts to be adequate and in most of the cases high.

Table 2. XRD content

<table>
<thead>
<tr>
<th>Pattern #</th>
<th>Compound Name</th>
<th>Formula</th>
<th>System</th>
</tr>
</thead>
<tbody>
<tr>
<td>PDF 01-074-3822</td>
<td>Lithium Cobalt Oxide</td>
<td>Li Co O$_2$</td>
<td>Orthorhombic</td>
</tr>
<tr>
<td>PDF 00-001-1098</td>
<td>Muscovite</td>
<td>H$_2$ K Al$_3$ (Si O$_4$)$_3$</td>
<td>Monoclinic</td>
</tr>
<tr>
<td>PDF 00-046-1045</td>
<td>Quartz, syn</td>
<td>Si O$_2$</td>
<td>Hexagonal</td>
</tr>
<tr>
<td>PDF 00-009-0466</td>
<td>Albite, ordered</td>
<td>Na Al Si$_3$ O$_8$</td>
<td>Triclinic</td>
</tr>
<tr>
<td>PDF 00-033-0786</td>
<td>Spodumene</td>
<td>Li Al Si$_2$ O$_6$</td>
<td>Monoclinic</td>
</tr>
</tbody>
</table>

Table 3 shows the elemental analysis of the mineral samples. This analysis was carried out by X-ray fluorescence (XRF).

Table 3. XRF of the pegmatic ore

<table>
<thead>
<tr>
<th>A(4.4894g)$_{oxide}$</th>
<th>Concentration(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO$_2$</td>
<td>59.32</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>16.5</td>
</tr>
<tr>
<td>CHO</td>
<td>15</td>
</tr>
<tr>
<td>Na$_2$O</td>
<td>4.29</td>
</tr>
<tr>
<td>K$_2$O</td>
<td>1.18</td>
</tr>
<tr>
<td>Fe$_2$O$_3$</td>
<td>1.17</td>
</tr>
<tr>
<td>P$_2$O$_5$</td>
<td>1.07</td>
</tr>
<tr>
<td>MgO</td>
<td>0.622</td>
</tr>
<tr>
<td>CaO</td>
<td>0.296</td>
</tr>
<tr>
<td>MnO</td>
<td>0.0755</td>
</tr>
<tr>
<td>Rb$_2$O</td>
<td>0.0448</td>
</tr>
<tr>
<td>Nb$_2$O$_5$</td>
<td>0.0057</td>
</tr>
</tbody>
</table>
An inductively coupled plasma optical emission spectrometer (ICP–OES, PerkinElmer Optima 8300) was used to determine the concentrations of Li ion.

![Image of ICP-OES instrument](image)

Figure 7. The inductively coupled plasma

<table>
<thead>
<tr>
<th>Element</th>
<th>Li 670.784</th>
<th>Si 251.611</th>
<th>Al 396.153</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.759</td>
<td>25.25</td>
<td>8.02</td>
</tr>
<tr>
<td></td>
<td>0.768</td>
<td>26.5</td>
<td>8.195</td>
</tr>
<tr>
<td></td>
<td>0.721</td>
<td>26.13</td>
<td>6.802</td>
</tr>
</tbody>
</table>

Table 4. ICP data

Using ICP Li concentrations can be found. According to Table 4 there are three experiments with 0.759, 0.768 and 0.721 Li concentrations. Moreover, other main elements such as Si and Al analyzed too.
3.2 Reagents and chemicals

DTAC was purchased from Samchun Pure Chemical Co.Ltd. MIBC (98%) and sodium oleate (82%) was purchased from Sigma Aldrich. DTAC and sodium oleate were used as collectors, and MIBC was used as a frother. Hydrochloric acid and sodium hydroxide were used to regulate pH.
4. Results and discussion

4.1 One stage flotation

4.1.1 Single and mixed collectors

Oleic acid is a widely used anionic collector in the flotation of Spodumene. Sodium Oleate is a salt of Oleic Acid, thus it can be equally treated in the functional interpretation. The maximum flotation recovery and adsorption amount of collector were obtained under slightly alkaline pH conditions. Oleate speciation analysis illustrates that oleic acid hydrolyzes to form ions (RCOO\(^-\)) at strong alkaline pH and molecules (RCOOH) at acid and neutral pH: in the slightly alkaline pH region, both ions and molecules exist in oleic acid solution, which associate by van der Waals force to form ionic–molecular complexes. (Fushun Yu, Yuhua Wang, Lei Zhang, Guangli Zhu, 2014).

The longer the hydrocarbon chain length of a collector, the more powerful is the water–repulsion produced, but solubility decreases. The surface activity of oleic acid is due to the non-polar hydrocarbon chain. The polar carboxylate functional group in oleic acid promotes the dissolution of the surfactant in aqueous solution. These properties make oleic acid used widely in the flotation of spodumene (Amarante et al., 1999).
Figure 8. Species distribution diagram of oleic acid as a function of pH for $6 \times 10^{-4} \text{M}$ aqueous solution. (Fushun Yu, Yuhua Wang, Lei Zhang, Guangli Zhu, 2014)

Figure 9. Longhua Xu, Yuehua Hu, Houqin Wu, Jia Tian, Jing Liu, Zhiyong Gao, Li Wang, 2016
According to Figure 8 carboxylate complex appears at slightly alkaline medium, thus all experiments involving sodium oleate were conducted at pH around 8.5 which suits the Figure 8 showing RCOO⁻ complex. Due to sample limitation, oleate flotations other than pH 8-8.5 were not performed.

To investigate theory of oleate speciation, the FTIR measurements were conducted with pure sodium oleate, spodumene with 95% purity, spodumene conditioned in a bulk with a sodium oleate concentration and spodumene conditioned in a bulk with a DTAC. Fourier-transform infrared spectroscopy (FTIR) is a method used to gain an infrared spectrum of absorption or emission of a solid, liquid or gas. An FTIR spectrometer contemporaneously gather high-spectral-resolution data over a wide spectral range.

![Figure 10. The FTIR of pure and mixed states.](image-url)
Figure 10 presents the FTIR run by Nicolete 6700 in the wavenumber range cm\(^{-1}\) 4000-400. The samples were spodumene, sodium oleate and the separate mix of spodumene with DTAC with oleate. For the IR spectra of sodium oleate, two peaks at 2926.3 and 2854.7 cm\(^{-1}\) are attributed to \(-\text{CH}_2\) asymmetric and symmetric stretching frequencies. The characteristic sharp band at 1710.8 cm\(^{-1}\) is due to the asymmetric stretching frequencies of \(-\text{C}=\text{O}\) in the carboxyl of sodium oleate. After treated with sodium oleate, the FTIR spectra of spodumene display new bands at 2920.22 and 2854.43 cm\(^{-1}\), which are assigned to the \(-\text{CH}_2\) asymmetric and symmetric stretching frequencies respectively. The new band at 1559.46 cm\(^{-1}\) is attributed to the asymmetric stretching frequencies of \(\text{COO}^-\), which is combined with Al atom on the surface of the mineral. This indicates a chemical adsorption of oleic acid ion on the aluminum sites of the spodumene surface that is in consistence with a study by Moon and Fuerstenau, 2003.
Figure 10 subject does not show differentiable new peaks belonging to carboxylate complexes; therefore, detailed graph in Figure 11 clearly demonstrates the new peaks of spodumene with sodium oleate including numerical descriptions.

Figure 11. The FTIR of the mix of the spodumene and sodium oleate.
According to the Figure 12 when spodumene mineral was mixed with DTAC, a main characteristic adsorption peak was observed at 2984.25 cm\(^{-1}\). Because no band shift was noticed, it can be deduced that the adsorption of DTAC on the spodumene surface is depends on electrostatic attraction and possibly some tiny part of hydrogen bonding.

Figure 12. The FTIR of the mix of the spodumene and DTAC
First experiment was done to find out approximate amount of collector to be used in the bulk of 5% solids ratio and study efficiency of single collector. Moreover, very important limitation, is the improper size of the rotation shaft of the Denver machine with respect to the flotation cell. As all the flotation results did not suit ideal laboratory conditions, thereby some deviations may have occurred.

![Graph showing effect of sodium oleate collector amount](image)

Figure 13. Effect of sodium oleate collector amount.

According to the Figure 13, sodium oleate collector amount of $3 \times 10^{-4}$ M showed grade of 3.6% and recovery 47% while double concentration decreased grade to 2.72% and recovery 50%. This can be explained by the monolayer and multilayer adsorption theories. Too high concentration starts to select not only valuable minerals but gangues as well, thus selectivity on spodumene decreases.
Single cycle flotation showed that sodium oleate can selectively float spodumene, but not able to keep high recovery. According to other studies, recovery is being higher than 47% while grade not much decreased. This can be explained by the usage activators and depressants in the flotation that enhance recovery of spodumene.

A most famous mechanism of flotation is using mixed anionic and cationic collectors. Recalling that every mine is different in terms of mineral composition and grade distribution, thus practical examination is required. The most famous cationic collector is dodecyl trimethyl ammonium chloride (DTAC). Firstly, the most appropriate oleate and DTAC ratio was investigated, then total concentration of mixed collectors necessary for efficient flotation were examined and lastly DTAC was substituted with other cationic collectors to check if a mixed cationic flotation of the same reagent would give different outcome comparing with single cationic flotation.
According to the Figure 14 there are three different rations where major concentration belongs to oleate and minor to DTAC. Ration 9:1 beyond dispute is a best ration in terms of grade which is 3.24%, and slightly lower in the recovery perspective 71%.

Figure 14. Mix of oleate and DTAC at different rations.
Definitely, more is not always good. Figure 15 shows two concentrations of the mix of collectors oleate and DTAC at the ratio of 9:1. The grade of $3 \times 10^{-4}$ M is 3.24% and recovery 71% while $6 \times 10^{-4}$ M have grade of 2.67% and recovery is higher just for 4% that is 75%. Thus, $3 \times 10^{-4}$M amount was used in all experiments.

Figure 15. Mix of oleate and DTAC at different concentrations
Last but not least is the comparison of another cationic collector such as tetrabutyl ammonium chloride when mixed with oleate collectors. As it can be seen from the Figure 16 grade and recovery of the mix of oleate and TBAC is 3.43% and 44% respectively while Oleate with DTAC is 3.49% and 76%.

![Figure 16. Comparison with other cationic collector](image)

The low recovery of TBAB can be referred to molecular shape whereas DTAC has chain structure. In order to obtain the detailed information about the adsorption mechanism between sample and the collectors, the changes in the polarity parameter I3/I1 of pyrene in the feed suspension should be examined using fluorescence spectroscopy. Therefore, the adsorption behaviors and mechanisms deserve further investigations.
According to the structures in the Figure 17, DTAC has a straight single 12 hydrocarbon chain while TBAB has 4 branches each having 4 hydrocarbon chain which can be the reason of the low recovery.

Figure 17. DTAC and TBAB structures
4.1.2 Watermelon seed experiment

The Kazakhstan no doubt can be categorized to the list of agricultural countries, and watermelon cultivation in South Kazakhstan is immense. One of the main anionic collector of spodumene mineral is carboxylic acid with hydrocarbon chain. In this experiment watermelon seed oil was attempted to take role of noted sodium oleate. Watermelon seeds were bought from online shopping. Seeds were dry and professionally selected, thus there was no question about the quality. Table 5 below is the property of the seed that has been submitted for the analysis using gas chromatography machine.

Table 5. Fatty acids concentrations in the watermelon seed

<table>
<thead>
<tr>
<th>Name</th>
<th>Ratio %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pentadecylic acid (C15:0)</td>
<td>9.3</td>
</tr>
<tr>
<td>Palmitic acid (C16:0)</td>
<td>9.9</td>
</tr>
<tr>
<td>Stearic acid (C18:0)</td>
<td>7.4</td>
</tr>
<tr>
<td>Oleic acid (C18:1)</td>
<td>14.6</td>
</tr>
<tr>
<td>Linoleic acid (C18:2)</td>
<td>57.9</td>
</tr>
</tbody>
</table>

According to the Table 5 main fatty acids are Oleic acid of 14.6% and Linoleic acid 57.9% while Stearic acid 7.4%, Palmitic acid 9.9% and Pentadecylic acid 9.3% are being fatty acids however were not able to dissolve in the bulk. Both of them have extremely low solubility in water, but better in ethanol.
Figure 18. Flotation comparison using watermelon seed oil

Figure 19. Flotation comparison of Linoleic and Oleic acids
Watermelon seed composition was reproduced by mixing exact amount of separate reagents shown in the Table 5. Watermelon seed alone demonstrated grade of 3.18% and recovery of 63%, but with DTAC increased to 76% while grade slightly decreased. Mix of DTAC proves to be good at recovery.

According to Figure 20 both fatty acids have carboxylate complex which are responsible with Al sites interaction on the spodumene surface. Even though Linoleic and Oleic acids both have same number of C18, the Linoleic acid is still less efficient than classical oleic acid. The reason might be lie in the difference in the fatty acid distinction. Linoleic acid is polysaturated which misses two hydrogen pair on their chain while oleic acid monosaturated misses one pair.

![Figure 20. Structures of Oleic acid and Linoleic acid](image-url)
4.3 Two stage flotation

4.3.1 Reverse flotation

The experiments were conducted into two stages where each stage going to be studied in details using various analyzing instruments. According to XRF data on mineral composition, it is found that near 60% of composition belongs to silicon oxide. The purpose of first stage is to decrease amount of silicon oxide, as much as it is possible. Quaternary ammonium salts (also called quaternary ammonium compounds, QACs) are generally halide salts with four alkyl groups attached to a central cationic nitrogen atom. Quaternary ammonium salts have strong surface activities and are extensively used as surfactants.

Figure 21 presents speciation of DTAC. The RNH$_3^+$ and (RNH$_3^+$)$_2$ as main species at acidic pHs are not able to adsorb electrostatically on the minerals surfaces with positive charges. The maximum flotation recovery of quartz at the pH range of 7–10 can be attributed to the presence of the RNH$_2$RNH$_3^+$ as ion molecular species, which are more surface-active species to interact electrostatically with the minerals with negative surface charges.
Figure 21. DTAC speciation. (Shima Rahimi, Mehdi Irannajad*, Akbar Mehdilo, 2017)
The concentration of 0.02mM and 0.05mM of DTAC was used in the bulk of 5% solids under pH 8.5. As the task of whole flotation is to get higher grade and recovery at the same time it is recommended to find out best trade-off. The Figure 22 demonstrates that using 0.02mM of DTAC concentration brings 73% of recovery and 3.07% of grade which is suitable to continue on to second stage.

Figure 22. Flotation comparison of 0.02mM and 0.05mM of DTAC at pH 8.5
The zeta potential measurement is an efficient method to interpret the trend of the flotation efficiency and the modification performance caused by the presence of reagents. Figure 23 shows Zetasizer nano series used to determine zeta potentials.
Figure 24 shows zeta potentials of silicon dioxide and spodumene minerals under natural state. As many research studies have proved, the spodumene zeta potential is positive until around pH 3 state, when over pH 3 it continuously decreases to negative direction. In case of quartz, starting from pH 2 zeta potential steadily decreases.

Figure 24. Spodumene and quartz at natural states
Figure 25 demonstrates quartz behavior in the natural state and in the presence of cationic collector DTAC. Data of pure quartz zeta potential coincides with experimental data of this research.

The M302 and M301 are both cationic collectors, where other study were aimed to compare both. Either way, quartz in the presence of DTAC is constantly positive which means that electrostatic force is the main mechanism of the flotation of quartz to the froth part. This can be explained by the adsorption of DTA+ ions, which neutralizes the negative charges of quartz surface and shifts the zeta potential towards the positive direction. Empirical data is provided using XRF data in the further pages.

![Figure 25](image.png)

Figure 25. Quartz zeta potential with and without cationic collector named M302 which stands for DTAC. (Xiaoqing Weng1,2*, Anh V. Nguyen2, Guangjun Mei1*, Yongfu Yu1, 2014)
Experiment under name BF75 was conducted. After flotation, froth and tail parts were filtered, dried and sent to XRF analyzer to check for desilication percentage. Silicon oxide is 14% higher in froth part which apparently means that desilication took place in the cationic reverse flotation. The important part is that aluminum oxide in tail part happens to be 30% higher than in froth part. This can be explained that the bond strength of Si–O is bigger than that of Li–O and Al–O, therefore, the disaggregation of spodumene would occur mainly alone the broken directions of Li–O bond and Al–O bond. Spodumene mineral contains aluminum atom, thus it is in high priority to not let aluminum oxide move to froth part in the first stage of flotation which is going to be discussed in details in the next section.

Table 6. XRF analysis of the experiment under name BF75
4.3.2 Second stage – Direct flotation

Firstly, in order to check the ideal flotation state, the sink and float experiments were carried out using tetrabromoethane as a heavy media. Centrifuge machine made by Hanil Science Industrial MF550 was used to sink down all particles that have density higher than 2.97 and float up particles that possess density lower than 2.97. The density of spodumene is 3.2 and density of other minerals are around 2.7. Centrifuge was set on 1500rpm for 10 minutes, plastic falcon tube was filled with 40mL of heavy media and 10g of feed. Then, samples were filtered, dried and concentration was analyzed using inductively coupled plasma.

Figure 26. Centrifuge machine
Table 7. The Sink&Float analysis

<table>
<thead>
<tr>
<th>40 min Ball Mill</th>
<th>10 g + 40mL of tetrabromoethane</th>
</tr>
</thead>
<tbody>
<tr>
<td>100-400 Feed</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Float</th>
<th>Sink</th>
<th>Loss</th>
<th>%</th>
<th>Float</th>
<th>Sink</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield, g</td>
<td>8.62</td>
<td>1.13</td>
<td>0.25</td>
<td>Grade, Li₂O</td>
<td>0.51</td>
</tr>
<tr>
<td>Ratio, %</td>
<td>86.2%</td>
<td>11.3%</td>
<td>2.5%</td>
<td>Recovery</td>
<td>0.34</td>
</tr>
</tbody>
</table>

As it can be seen from Table 7 the grade of Lithium oxide in the sink part is 7.66% which is close to maximum possible separation, however tetrabromoethane is toxic and expensive as well, thus it is not allowed to use in the industrial scale.

As Al sites play significant role in spodumene flotation, it is of great interest to increase Al sites on the spodumene surface. There exist an assumption that conditioning in highly alkaline solutions prior to flotation improves spodumene separation effectiveness. Pretreatment results in alkaline medium at pH 12 with the minerals for 30 minutes at 20% bulk density was studied by X-ray photoelectron spectroscopy (XPS). X-ray photoelectron spectroscopy (XPS), also known as electron spectroscopy for chemical analysis (ESCA), is a technique for analyzing the surface chemistry of a material. XPS can measure the elemental composition, empirical formula, chemical state and electronic state of the elements within a material.
Table 8 shows 1. before and 2. after; which indicate the feed that was not pretreated and pretreated in alkaline medium respectively. After pretreatment aluminum atom mass concentration increased by 16.4% while other main atom silica did not show chemical shift. Pretreatment was done in the 20% solids ratio, NaOH concentration was 2250mg/L and conditioning time was 30minutes.

Table 8. Comparison data of the pretreated and non pretreated samples

<table>
<thead>
<tr>
<th>1. Before</th>
<th>2. After</th>
</tr>
</thead>
<tbody>
<tr>
<td>Name</td>
<td>Position BE (eV)</td>
</tr>
<tr>
<td>O 1s</td>
<td>530</td>
</tr>
<tr>
<td>C 1s</td>
<td>283</td>
</tr>
<tr>
<td>Na 1s</td>
<td>1070</td>
</tr>
<tr>
<td>Si 2p</td>
<td>101</td>
</tr>
<tr>
<td>Al 2p</td>
<td>72</td>
</tr>
<tr>
<td>O 1s</td>
<td>530</td>
</tr>
<tr>
<td>C 1s</td>
<td>283</td>
</tr>
<tr>
<td>Na 1s</td>
<td>1070</td>
</tr>
<tr>
<td>Si 2p</td>
<td>101</td>
</tr>
<tr>
<td>Al 2p</td>
<td>73</td>
</tr>
</tbody>
</table>
Figure 27 has many elements with their respective position according to binding energy. The Al atom has binding energy of 73eV, which corresponds to the marked arrows. There is a slight increase can be observed in the form of two additional blue circles in the “after treatment” part which proves Al sites increase due to alkaline conditioning.

Figure 27. XPS graph of the pretreated and not pretreated samples
Figure 28 demonstrates two experiments with all same conditions except one is pretreated in alkaline medium, another one is not pretreated. Sodium oleate was used as a collector, pH of 8 was kept in both cases. As it can be seen, pretreated one has grade of 3.6% while not pretreated of 2.75%, which leads to 23.6% difference, thus it is highly recommended to this actions before flotation.

![Graph showing recovery and grade comparison between pretreated and non-pretreated samples.](image)

Figure 28. Flotation of pretreated and non-pretreated samples
Figure 29. First flotation cycle

THESIS SCHEME
Experiment BF85

Reverse Flotation

Direct Flotation

HCL treated

HCL treated

DTAC

FROTH

GRADE: 2.39%
Recovery: 83%

FROTH

OLETE

Grade: 0.67%
Recovery: 17%

OLETE

Grade: 0.52%
Recovery: 53%

OLETE

Grade: 1.18%
Recovery: 47%

FROTH

FROTH

5.77g 0.032g

7.46g 0.133g

Grade: 2.96%
Recovery: 60%

Grade: 3.81%
Recovery: 40%

15.56g 0.133g

Grade: 1.83%
Recovery: 65%

15.56g 0.133g

Grade: 3.54%
Recovery: 35%

Grade: 0.071g

Grade: 0.201g

Grade: 2.96%
Recovery: 60%

Grade: 3.81%
Recovery: 40%

Grade: 1.83%
Recovery: 65%
Figure 29 is the data of actual experiment where stage one and stage two flotation has been conducted. From the stage one it can be observed that cationic reverse flotation does separate gangues from valuable minerals. As called reverse flotation, valuable mineral reported in the tail part where grade is 2.39% and recovery 83% while lithium oxide’s grade in the froth part just 0.67% and recovery 17%. As froth part contains tiny amount of lithium oxide, only one time oleate flotation was enough to get maximum of it, thus grade of 0.52% and recovery 53% has been disposed.

Whereas, direct flotation of tail part from the reverse flotation has two oleate flotation steps where first tail part’s grade is 2.06% and recovery 60%, which is irrational to ignore thus second step oleate flotation was conducted finally reporting grade of 1.83% and recovery 65% that added to a continuous flotation, back to the beginning. Before sending to the beginning of the flotation process, the last tail part including froth of the reverse part were treated in 1M of hydrochloric acid in order to remove any chemi-physisorbing interaction, thus new cationic interaction can have a fresh start.

The total grade is 3.74% and recovery 51.8% of the whole first cycle that include reverse and direct flotation.
Below illustrated two boxes that were copied and pasted from Figure 29 in order to show grade and recovery calculation methodology.

Recovery is how effectively the separator has extracted the valuable contained in the input stream having formula as \(\frac{\text{mass of valuable in product stream}}{\text{mass of valuable in input stream}}\times100\). Whereas, grade is quality of any stream which defined as \(\frac{\text{mass of valuable in stream}}{\text{total mass of stream}}\times100\)

In order to calculate grade, it is required to get ICP data on the target sample. After obtaining Li concentration from ICP analysis, concentration converted to lithium oxide form, which easily found by dividing ICP result by atomic mass of \(\text{Li}_2\) from \(\text{Li}_2\text{O}\). After that, yield of 7.46g and 3.16g multiplied by lithium oxide grade of 0.0381 and 0.0364 respectively that lead to 3.75% grade. The recovery calculated by dividing sum of 0.133g and 0.071g of Li by the total 0.380g of Li in the introduced feed.

According to the Figure 30 froth and tail from the first cycle that sent back to origin in order to balance steady state of the whole flotation. The tail part has grade of 2.38% and recovery of 70%, which approximated with the first cycle in the Figure 30 where tail part was 2.39% and recovery 83%
Figure 30. Second flotation cycle

THESIS SCHEME
Experiment BF85
The second cycle

Reverse Flotation
- 0.497g Li in 66.66g of feed
- 31.93g 0.355g
  - Grade: 2.38%
  - Recovery: 70%

Direct Flotation
- 17.92g
  - Grade: 1.83%
  - Recovery: 65%

- 7.54g
  - Grade: 3.81%
  - Recovery: 40%

- 3.90g
  - Grade: 3.64%
  - Recovery: 35%

OLEATE
- 23.21g
  - Grade: 2.06%
  - Recovery: 60%

FROTH
- TAIL
  - Grade: 0.94%
  - Recovery: 30%
Last but not least, according to study of spodumene activation, good floatability of spodumene was obtained with Ca(II) and Mg(II) ions at a pH of 12.5 and 10.0, respectively. In strongly alkaline solutions, Ca(II) and Mg(II) ions readily form hydroxy complexes (CaOH(I) and MgOH(I)) and precipitates (Ca(OH)$_2$ and Mg(OH)$_2$). FT-IR and XPS analyses revealed that the hydroxy complexes and the precipitates adsorbed onto the spodumene surface. These species contributed to the formation of oleate complexes and thus increased the amount of collector adsorption and the floatability of spodumene. (Weijun Liu, Shiqiu Zhang, Weiqing Wang, Jie Zhang, Wu Yan, Jie Deng, Qiming Feng, Yang Huang, 2015)

This research was dominantly concentrating on the reverse flotation using cationic flotation and direct flotation to get maximum purity as possible using prior conditions, thus activators and depressants in the second stage was ignored however recommended to include in further studies.
5. Conclusion

The experimental results obtained from this investigation showed that indeed the science of flotation is tremendous and various mechanisms can be designed depending on the ore chemistry and market demands. It was shown that sodium oleate without questioning is a superior in the recovery of the spodumene mineral and DTAC can be either sub-collector for main oleate or primary collector in the reverse flotation. In particular, reagent-mineral amount with their structures and pH level were considered. Moreover, it was shown that alkaline pretreatment significantly influences the effectiveness of the oleate functionality in the bulk, therefore samples should always be conditioned using sodium hydroxide reagent. Flotation utilizes the difference in surface properties of the valuable and gangue minerals where attachment of valuable mineral to the float part is the most important mechanism. Overall, three types of experiments were conducted.

First, single anionic collector namely sodium oleate was used. Grade of 3.6% and recovery of 47% reported, which proves that sodium oleate indeed has high selectivity function.

Second, mixed anionic and cationic collects namely sodium oleate and DTAC were used. Grade of 3.24% while recovery sharply increased up to 71%. Previous studies determined role of DTAC as a sub-collector whose atoms were packing between unfilled sites of oleate and mineral surface, thus increasing recovery. However, this research proved that DTAC could be a main collector too.

Third, due to importance of gangue presence with the valuable mineral, it is of great interest to eliminate the most simple
and abundant mineral quartz before introducing sodium oleate. Therefore, cationic DTAC reverse flotation was implemented to separate quartz from spodumene in the first stage flotation, which resulted in grade of 2.39% and recovery of 83%. Then tail part of reverse flotation was refloated using oleate collector that increased valuable mineral to froth part up to 3.81% and recovery 40%, while tail part still had grade of 2.06% and recovery 60%. Thus additional direct flotation of this tail part was required that finally lead froth grade to 3.64% and recovery 35%. In addition, froth part of reverse flotation was floated only once using oleate collector obtaining grade of 1.18% and recovery 47%. Combining two froth part of two direct flotation that originally coming from a reverse tail part was upgraded up to 3.74% and recovery 51.8%.

The results from zeta potential analysis implied that electrostatic interactions play a primary role in the flotation separation of silica when DTAC used as the collector and carboxylate complexes when using sodium oleate in the direct flotation.

Reverse flotation using cationic collector on spodumene recovery has never been published; therefore this research is a first that suggested desilication of pegmatic ore is efficient. Moreover, many studies proved spodumene Ca activators to be efficient for enhancing sodium oleate interaction, but it was out of scope of this research.
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요약(국문초록)

리튬은 주로 해수(海水)와 페그마타이트 광에 존재한다. 광물 형태의 리튬은 채산성 부족으로 거의 이용되지 않았지만 리튬의 가격이 크게 상승한 오늘날엔 경제적 가치가 높게 평가되어 탐사와 개발이 활발히 이루어지고 있다. 본 연구는 카자흐스탄에서 존재하는 페그마타이트 광에서 리튬이 포함된 스포듀민을 부선과 역부선의 혼합 공정을 통해 효과적으로 선별해내는 방법을 도출하였다. 부선 과정에 앞서 조크러셔와 볼밀을 이용해 시료를 분쇄하고 로맵 세이커로 부선에 적절한 사이즈를 선별하였다. 광물의 조성 파악을 위한 XRD 분석과 화학 조성 파악을 위한 XRF 분석을 실시하였으며 화학적 결합과 작용기들은 FTIR 분석을 통해 확인하였다.

본 연구에서는 Sodium Oleate, DTAC, TBAB 를 단독 또는 복합 조건으로 적용하여 최적의 조건을 도출하였다. 이들 외에도 다수의 전통적인 포수제와 잠재적인 신규 시약들이 실험되었으나 DTAC 에 비해 효과가 떨어지는 것으로 나타났다. 첨가제들의 최적 농도는 생산물의 회수 곡선과 풍위 변화를 분석하여 실험적으로 도출되었다. 실험 결과 DTAC 는 혼합 부선에서 주요 음이온 포수제에 대한 보조 역할을 할 뿐 아니라 역부선에 단독으로 사용되었을 때 스포듀민 표면에 있는 알루미늄과 전기적으로 결합하여 우수한 결과를 내는 것으로 나타났다. DTAC 를 활용한 역부선은 원광 내 존재하는 1.5% 의 리튬 산화물을 2.39% 로 농축하면서 전체 리튬 산화물의 83% 를 회수할 수 있었다. 부선된 부분의 리튬 농도는 0.67% 를 농축하여 DTAC 를 활용한 역부선이 광물내 리튬 회수를 위한 효과적인 수단임을 보였다.