Lithium carbonate sedimentation using flocculants with different ionic bases

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Abstract

Lithium has become a metal of enormous interest worldwide. The extensive use of rechargeable batteries for a range of applications has pushed for rapid growth in demand for lithium carbonate. This compound is produced by crystallization, by reaction with lithium chloride (in solution) and by adding sodium carbonate. Low sedimentation rates in the evaporation pools present a problem in the crystallization process. For this reason, in this work, mineral sedimentation tests were carried out with the use of two flocculant types with different ionic charges. The tests were carried out at a laboratory level using different dosages for each flocculant and measurements were performed to obtain the increase in the content of solids in the sediment. The anionic flocculant had better performance as compared to that of the cationic flocculant, increasing the sedimentation rate of lithium carbonate by up to 6.5. However, similar solids contents were obtained with the use of the cationic flocculant at 3.5 times lower dosage making it the flocculant of choice regarding the economic point of view.

Keywords: Brine treatment; anionic flocculation; cationic flocculation; nonferrous metals; mining.

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1. INTRODUCTION

The mining industry in Chile is constantly growing [1–4] . Historically, Chile has traded copper, this commodity being the main economic income contributing approximately 10 % of the gross domestic product (GDP) [5]. However, the quantity of high-grade Cu minerals on the surface is decreasing [6]. Therefore, in recent years, considerable investments have been made in utilization of other elements, lithium being the second most exploited commodity in the country [7].

Lithium is an electrochemically active metal and it has been recognized as one of the critical materials for the advancement of modern life due to its superior properties compared to other materials, such as redox potential and specific heat capacity [8]. This metal has a low atomic mass, low coefficient of thermal expansion, and high electrochemical reactivity, which has made it a particularly attractive material for use in rechargeable batteries for electronic devices, electric vehicles, and network storage systems [9,10].

The commercial value of lithium carbonate has increased over the years to reach US $ 13 per kg in 2017 [9]. In turn, the production of LCE (Lithium Carbonate Equivalent) also increased, so that during 2016 approximately 200,000 tons were produced, where Argentina is the largest producer (58.3%) followed by Chile (38.9 %) [11]. It is precisely in South America where the largest reserves are concentrated [12] (85 % of lithium reserves in brines) [13], accompanied also with other elements such as potassium, sodium, boron, sulfate and chlorides [14].

Prices doubled between 2013 and 2017 and lithium demand is expected to triple by 2025, with the World Bank estimating a 965 % increase in demand by 2050 [15]. By 2025, the investment banking group UBS forecasts that batteries

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will account for at least 80 % of the total lithium demand in the application industries [15]. Chilean lithium production comes from companies located in the Salar de Atacama (SQM and Albemarle).

Given the low concentration of this metal in nature reserves and other difficulties in its extraction from hard rocks, many industrially advanced countries have struggled to ensure an uninterrupted supply of lithium to meet their manufacturing demands. This fact has a significant impact on the price of lithium in the international market. For example, in 2017 in China, there was an increase in the price of lithium carbonate to US$ 6,400-12,000 per ton from US $ 2,000 per ton in 2005 [16]. Therefore, innovative processes to increase lithium production must be developed and evaluated to meet future demands.

Lithium is present in a wide range of minerals (spodumene, lepidolite, petalite, amblygonite, and eucriptite). At the same time, lithium is found in natural brines, brines associated with oil wells and geothermal fields. It is also present in various clays (hectorite being the most important) and even in seawater [17]. Although the sources of lithium can be diverse, at present only two production processes are economically feasible: using brines and minerals, with brine being mostly used [18], due to lower manufacturing costs in comparison with the costs of extraction and processing of lithium minerals [19].

The composition of commercial brines from which lithium is recovered varies considerably from brines with low lithium contents (0.02 %) to some with higher contents (close to 0.4 %), with the presence of other elements and salts such as potassium, sodium, calcium, magnesium, iron, boron, bromine, chlorine, nitrates, chlorides, sulfates and carbonates, which imposes a requirement that each brine is treated in a particular way, according to its composition.

The production of lithium carbonate from brines is carried out by precipitation [20]. This lithium compound is the most important compound commercially produced and represented 60 % of the market share of commercial products based on lithium in 2017 [21]. Lithium carbonate precipitation using sodium carbonate (Na_2CO_3) has been commonly used in industry as well as in research as the final step to produce crystals in a hydrometallurgical process [21].

After extraction of the brine (Sª) from a salt flat, it is pumped into huge pools for concentration of lithium, resulting in a decrease in the lithium content from an average value of 600 mg dm^{-3} in the Puna salt flats, to about 5,000 to 10,000 mg dm^{-3}. As a consequence of water evaporation, significant amounts of NaCl crystallize in the pools, accompanied with crystallization of KCl and, depending on the brine composition, other salts, such as $CaSO_4$ [22].

Prior to lithium precipitation, it is necessary to reduce calcium and magnesium contents, which is achieved by chemical precipitation (equations 1 and 2), with the use of calcium hydroxide ($Ca(OH)_2$), sodium carbonate (Na_2CO_3) or sodium sulfate (Na_2SO_4). In the case of boron, its removal is achieved by solvent extraction and/or ion exchange [22].

$$Ca(OH)_2 + Mg^{2+} \rightarrow Mg(OH)_2 + Ca^{2+} \quad (1)$$

$$Na_2(SO)_4 + Ca^{2+} \rightarrow Ca(SO)_4 + 2Na^+ \quad (2)$$

The lithium carbonate precipitation stage is carried out at a high temperature (90 ºC), by adding sodium carbonate to the concentrated and purified brine [22] as:

$$Na_2(CO)_3 + 2Li^+ \rightarrow Li_2(CO)_3 + 2Na^+ \quad (3)$$

Once the carbonate is obtained, particle sedimentation begins at rather low rates, which motivated this investigation. In order to reduce the sedimentation time, it is necessary to add certain reagents that help formation of flocs or particle binding, thus increasing the particle weight and, consequently, the sedimentation rate [23]. In the present work, we present the importance of the use of flocculants to increase production of lithium carbonate, by reducing the sedimentation time.

Flocculation of colloidal particles is typically performed by using high molecular weight polyacrylamides, which can be different with respect to the electrical charge (cationic, anionic, or non-ionic), depending on the use required. Typical chemical structures of anionic, neutral, and cationic polyacrylamides used as flocculants.

2. MATERIALS AND METHODS

2.1. Lithium carbonate sample

Lithium carbonate was acquired from Merck (N” CAS 554-13-2; Chile), having a density of 2.11 g cm^{-3}. To reproduce a brine, lithium carbonate was mixed with water up to a 7.5 wt.% solid content.

2.2. Flocculant preparation

As a cationic flocculant interfloc 5002 was used while SNF 60420 was used as the anionic flocculant. Small flocculant crystals were dissolved in distilled water at the concentration of 1g dm^{-3} while stirring on a magnetic stirrer for 5 h due to the high density of the reagent to obtain a homogeneous solution (without lumps).

2.3. Sedimentation tests

In the present work, the flocculant amounts for the sedimentation tests were 5, 15, 30, 50, 75, 100, 125, 150, 175, 200 and 275 g t^{-1} for each flocculant type. The chosen dosages are the typical values used in non-metallic mining industry.

Sedimentation tests, performed in duplicates, were conducted by pouring 270 cm^3 of the homogeneous pulp into a cylinder and performing interface height measurements every 20 min (due to the low sedimentation rate of lithium carbonate) at the lowest point of clear liquid. The test tubes were previously graduated at the mm precision.

For pulp characterization, the mineralogical and chemical features can be reflected in the content of solids [24]. The relative proportion in which the mineral and water are found is represented by the solid content (C_p), which corresponds to the content ratio of the mineral weight to the pulp weight (eq. 7). For this calculation, from the data obtained in the laboratory, the solid content by volume (C_v) is obtained by using eq. (6) and the volume of the thickened pulp (eq. 5) from the conditions in the developed test. The content of solid is determined at the moment at which the height of the interface stops changing, that is, it does not decrease further.

The volume of ore is calculated as:

$$V_\mathrm{m} = \frac{m}{\rho_\mathrm{s}} \tag{4}$$

The volume of the thickened pulp is calculated as:

Thikened volume = $\pi r^2 h_\mathrm{n}$ (5)

The solid content by volume is calculated as:

$$C_\mathrm{v} = \frac{V_\mathrm{s}}{V_\mathrm{p}} 100 \tag{6}$$

The solid content by weight is calculated:

$$C_\mathrm{p} = \frac{\rho_s C_\mathrm{v}}{(1-C_\mathrm{v})\rho_\mathrm{l} + \rho_\mathrm{s} C_\mathrm{v}} 100 \tag{7}$$

V_m - Mineral volume
V_p - Pulp volume
r - Specimen inner radius
C_v - Solid content by volume
ρ_s - Mineral density
C_p - Solid content by weight
m - Mineral mass
V_s - Solid volume
h_n - Height at each measurement

2. 4. Sedimentation rate

Once the tests in duplicates have been completed, the average height values at each measured time were graphically presented (Figure 1.) and the sedimentation rate is obtained by using the Coe and Clevenger method as the slope of the initial tangent line. In the present investigation first three points were used for construction of the tangent, with the aim to minimize error.

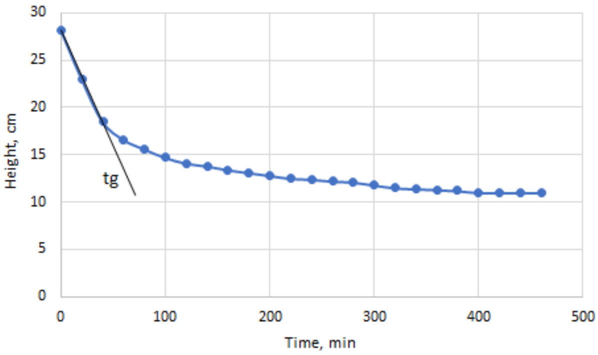

Figure 1. Determination of the sedimentation rate as a slope of the tangent *(tg) at initial values of* the *thickened pulp height vs. time*

3. RESULTS AND DISCUSSION

Table 1. presents the content of lithium carbonate in the sediment after 460 min based on utilization of both flocculant types (anionic and cationic).

Table 1. Content of solids in the sediment obtained in flocculation tests after 460 min.

Dosage, g t^{-1}	Cationic flocculant		Anionic flocculant	
	Content of solids, wt.%	Standard deviation, %	Content of solids, wt.%	Standard deviation, %
0	11.17	± 1.07	11.17	± 1.07
5	15.96	± 2.90	14.88	± 2.36
15	16.08	± 2.58	15.29	± 2.47
30	16.44	± 2.91	15.51	± 2.62
50	17.21	± 2.86	16.20	± 2.23
75	16.95	± 2.76	16.44	± 2.65
100	16.69	± 2.55	16.20	± 2.11
125	16.56	± 2.51	15.62	± 2.42
150	16.69	± 2.48	15.51	± 2.06
175	15.40	± 2.04	18.68	± 2.38
200	15.73	± 2.29	17.77	± 3.01
275	15.19	± 1.90	15.40	± 2.52

The highest solid concentrations in the sediments were ~17 % for the cationic and ~19 % for the anionic flocculant. To reach these concentrations, dosages of 50 and 175 g t^{-1} for the cationic and anionic flocculant, respectively, were used, which indicates an exceptionally large difference between these two flocculant types. The cationic flocculant is used for sedimentation of non-metallic and organic particles, whereas the anionic one is more used for metallic materials [25].

Accordingly, higher contents of solids were achieved for the cationic flocculant in most of the cases, but it should be noted that above the dosage of 175 g t^{-1} the opposite is observed that is higher solid concentrations for the anionic flocculant. The cause of this phenomenon is high adsorption of the flocculant on the particles, reducing the particle surface area to interact with the polymer chain attached to another particle [26], which causes a large part of the solids to remain in suspension.

Figure 2. presents the sediment height over time for the two tests producing the best results.

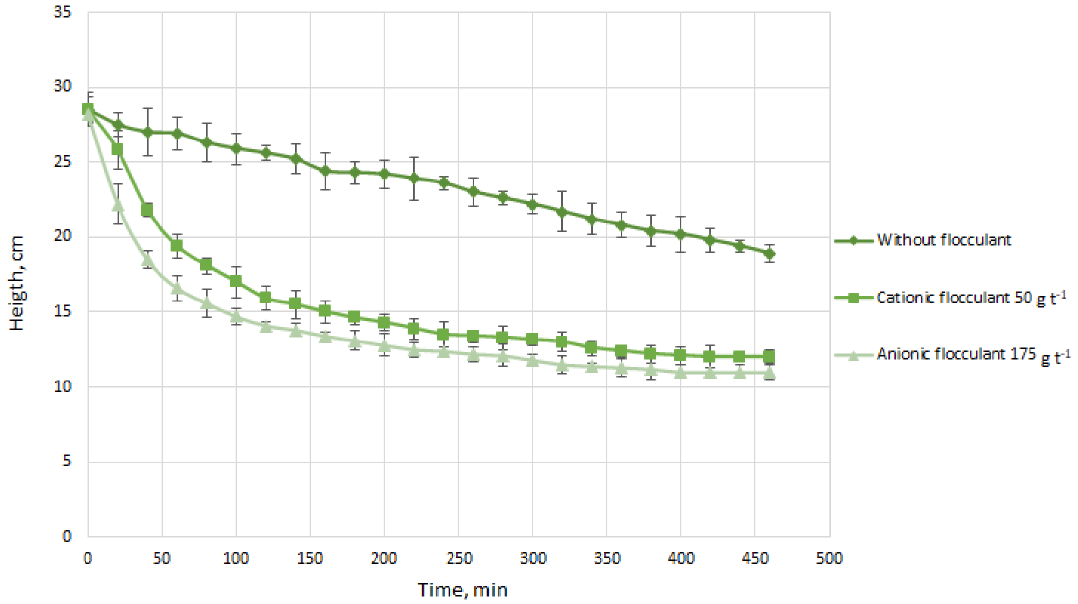

Figure 2: Interface height over time for the control sedimentation test (without flocculant) and the tests with the use of 50 g t^{-1} cationic flocculant and 175 g t^{-1} anionic flocculant

A noticeable difference is seen with regard to the final interface heights obtained in the sedimentation tests, decreasing from 28.5 cm to 12 cm for the cationic flocculant and to 11 cm for the anionic flocculant. Thus, there is a greater compaction of the sediment and consequently a higher content of solids is obtained, possibly due to lower entrapment of water in the test with the anionic flocculant in the compaction zone.

Figure 3. and Figure 4. show the solids concentrations in the sediment zone over time for the cationic and anionic flocculant, respectively, at the dosages studied. It can be seen that the increase in the solids content is not very large (it increases from 7.5 % to approximately 18 %), possibly due to the inverse solubility of lithium carbonate [27]. In specific, at low temperatures, sedimentation is minor because part of the lithium carbonate is dissolved in the solution, thus reducing the number of particles.

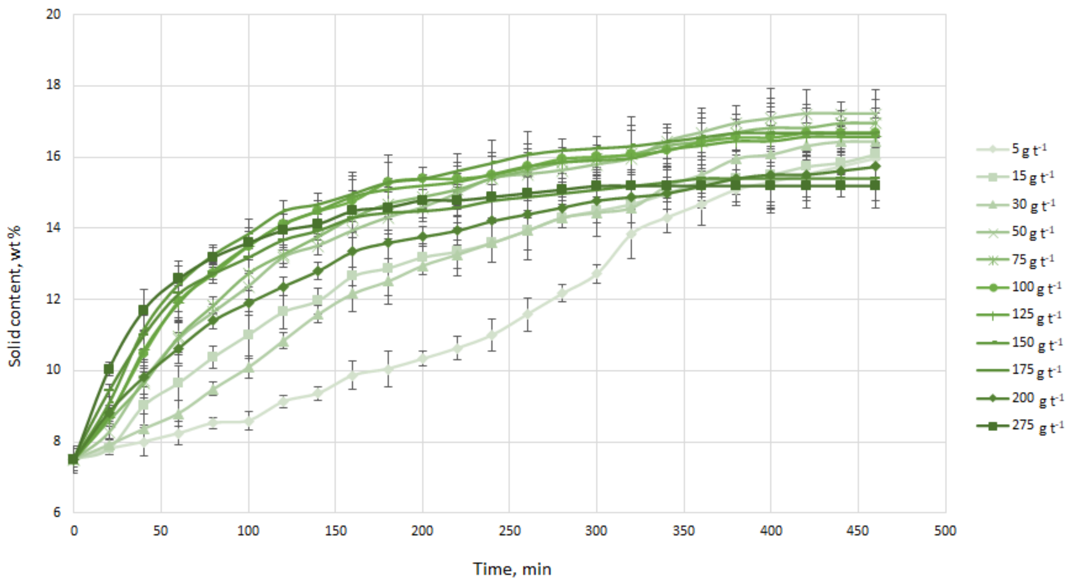

Figure 3: Solids content over time at different dosages of the cationic flocculant

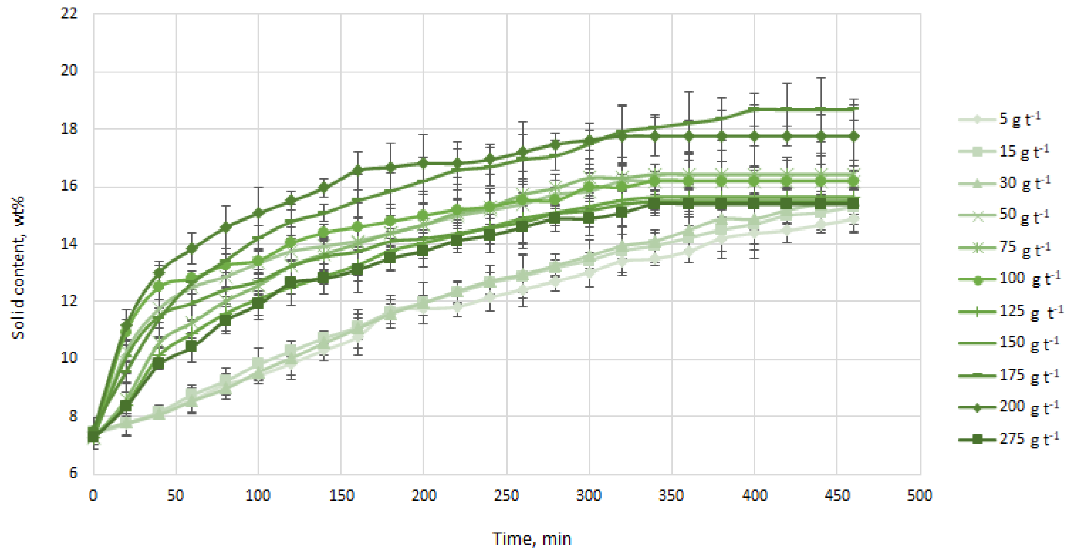

Figure 4. Solids content over time at different dosages of the anionic flocculant

It can be seen that, at high dosages, the solids content stagnates at around 17 % for both flocculant types. It is possible that there is an increase in the viscosity of the sediment due to the large amount of flocculant [28] causing trapping of water and decreasing the content of solids.

It can be seen that even at low dosages of the cationic flocculant except at 5 g t^{-1}, the content of solids exceeds 12 % at the time of 160 min (Figure 3.), whereas for the anionic flocculant (Figure 4.) this result occurs at 220 min, which indicates that the sedimentation rate at low dosages is higher for the cationic flocculant as compared to that for the anionic flocculant. It can be also observed that at the initial times and at high flocculant dosages the solids content increases faster for the anionic flocculant. This is possibly due for to the action mechanisms of each type of the flocculant as mentioned by Concha [29] and Diaz [25]. In the case of the anionic flocculant, covalent bonds and/or hydrogen bridges are formed between the particles and these flocculants are mainly used for concentrates and tailings of copper, lead, zinc, *etc.* On the other hand, cationic flocculants act in a very similar way as coagulants, which predominantly comprises neutralization of particle surface charges, rather than formation of bridges [30].

The thickening process provides concentration of a dilute pulp into a thickened pulp as solid particles settle under the influence of the force of gravity. In the sedimentation tests, the addition of flocculants directly affects the suspension properties, which can be identified by means of the sedimentation rate. Measurement proceeds from the time zero that represents the pulp sample after complete mixing and the beginning of the test until the pulp reaches the maximum compression point (critical point), at which the interface of the water-suspension meets with the interface of the sediment-suspension at a critical height so that the sedimentation ends. The sedimentation rates obtained from Figure 2. and initial tangent slopes are presented in Table 2.

Table 2. Sedimentation rates for the tests in which the highest solid content was achieved

Test	Rate, cm min^{-1}	σ, cm min^{-1}
No flocculant	0.040	0.964
Cationic Flocculant (50 g t^{-1})	0.143	0.991
Anionic flocculant (175 g t^{-1})	0.250	0.978

The sedimentation rate increased 3.5- fold with the flocculant addition to the pulp for the cationic flocculant at a dosage of 50 g t^{-1}, and 6.5-fold for the anionic flocculant at 175 g t^{-1}. The obtained results may be since the cationic flocculant destabilizes the charge of colloidal particles present in the pulp causing particle coagulation and consequently better agglomeration, while another possible cause is that the bonds formed by the anionic flocculant are not strong enough to allow preservation of the flocs over time.

The flocculant dosage difference for the tests resulting the highest solids contents is very high (125 g t^{-1} difference), which could be a determining point in the choice of the reagent, since the expenses that could be caused by the use of the anionic flocculant could be very high as compared to the those of the cationic flocculant.

4. CONCLUSIONS

Sedimentation of lithium carbonate was studied with the use of two flocculant types. For the maximum time studied (460 min), the highest obtained of solids was ∼19 % at the use of the anionic flocculant (175 g t^{-1}) and ∼17 % for the cationic flocculant (50 g t^{-1}). Therefore, it could be concluded that the best results are obtained with use of the cationic flocculant, because similar contents of final solids are obtained for the two flocculants but at hugely different dosages.

When working at 25 °C, the highest increase in the content of solids was ∼11 %, obtained with the anionic flocculant (175 g t^{-1}). Also, the sedimentation rate for this flocculant was higher than that for the cationic flocculant (0.250 and 0.143 mm min^{-1}, respectively), with the disadvantage that the flocculant amount is 3.5-fold higher. From the economic point of view, it is more convenient to use the flocculant with a positive ionic base since it achieves results remarkably similar to the anionic one, but at a noticeably lower dosage.

Overall, it is shown that the use of flocculants is a great alternative to accelerate the processes of obtaining lithium carbonate by thickening in ponds. Still, it is necessary to investigate the behavior of other chemical compounds present in brines during this process.

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SAŽETAK

Taloženje litijum karbonata korišćenjem flokulanata sa različitim jonskim bazama

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(Stručni rad)

Litijum je postao metal od ogromnog interesa u celom svetu. Široka upotreba punjivih baterija za čitav niz primena podstakla je brzi rast potražnje za litijum karbonatom. Ovo jedinjenje se dobija kristalizacijom, reakcijom sa litijum hloridom (u rastvoru) uz dodavanjem natrijum karbonata. Niske stope taloženja u bazenima za isparavanje predstavljaju problem u procesu kristalizacije. Iz tog razloga, u ovom radu su izvršena ispitivanja sedimentacije minerala upotrebom dva tipa flokulanta sa različitim jonskim naelektrisanjem. Ispitivanja su izvedena na laboratorijskom nivou koristeći različite doze za svaki flokulant, a cilj istraživawa je bio postizanje povećanja asadržaja čvrstih materija u sedimentu. Anjonski flokulant je imao bolje performanse u poređenju sa kationnim flokulantom, povećavajući brzinu taloženja litijum karbonata do 6,5 puta. Međutim, slični sadržaji čvrstih materija dobijeni su upotrebom katjonskog flokulanta u dozi nižoj 3,5 puta, što ga čini flokulantom izbora sa ekonomskog stanovišta.

Ključne reči: tretman rastvora; anjonska flokulacija; katjonska flokulacija; obojeni metali; rudarstvo.