

Article

Using Waste Brine from Desalination Plant as a Source of Industrial Water in Copper Mining Industry

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Abstract: One of the main challenges of seawater desalination is a large volume of waste brine production that is commonly discharged into the sea and may threaten the marine ecosystem. This is critical in regions where conventional water resources are scarce and desalinated seawater is an alternative to meet water demand. Especially in regions where the mining industry is a key player in the economic development. The novelty of this research consists in the determination of the potential use of waste brine, discharged from the reverse osmosis process, as a source of industrial water in copper mining industry. To enable the waste brine applicability, there should be reduced calcium and magnesium ions concentration for improving copper recovery in the froth flotation process. The flotation tests were conducted in a batch cell with synthetic minerals composed of chalcopyrite, kaolinite, and quartz using different water qualities. The results showed that treated waste brine significantly improved copper recovery compared to untreated waste brine and seawater. Similar copper recovery was achieved when flotation test was performed with tap water and treated waste brine. Therefore, treated waste brine could provide a suitable water quality required in the froth flotation process as an alternative non-conventional water resource.

Keywords: seawater desalination; waste brine; froth flotation process; copper sulfide minerals; industrial water

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1. Introduction

Mining and metallurgical plants cannot operate without safe and continuous access to water, which is mainly used in mineral processing and dust suppression [1,2]. Therefore, mining industries located in arid or semi-arid regions have the challenge of maintaining their plants in continuous operation with the limited availability of conventional water sources in those regions [3]. Copper mining is an example of such a situation, as many major copper mining companies operate in water-stressed regions such as Chile, Peru, and Australia [4]. Among these countries, Chile is the largest copper producer globally, supplying approximately one-third of the world's copper production, accounting for 5.7 million metric tons in 2020 [5]. The majority of the current mining operations in Chile are processing copper sulfide minerals, where copper concentrates are obtained by the froth flotation process. This process requires considerable water input to obtain copper concentrate, and the water quality is a relevant factor in achieving high enrichment of valuable minerals and high process performance [6].

Currently, Chilean copper mining companies are facing critical problems with declining copper ore grades and, consequently, with the additional processing of gangue

associated with complex minerals such as clays. Most of the Chilean copper mining companies operate in the Atacama Desert, where the climate is arid or semi-arid with a low level of precipitation, accounting for 87 mm/year [7]. In this context, many copper mining companies have obtained water for their operations from fossil aquifers for decades, as conventional surface and groundwater sources are almost nonexistent [8–10]. To face this problem, various copper mining companies use seawater in their operations, either through a seawater desalination plant or by using it directly without desalting [6,11]. The decision to use desalinated or direct seawater depends on each mining operation, considering the type of ore and minerals to be processed, the distance of their processing units from the coast, and the technologies considered for mineral processing [12,13].

The froth flotation process of copper sulfide minerals using seawater has attracted much attention as it leads to different challenges in the copper mining industry due to the high concentration of ions. As a result, several reviews have been published on this topic in the last ten years [14–17], highlighting that among the ions present in seawater, calcium and magnesium ions are detrimental to the flotation of copper sulfide minerals. As mentioned above, another option for processing copper sulfide minerals is the use of desalinated seawater obtained from reverse osmosis (RO) plants in Chile. Desalinated seawater consumption increased considerably from 0.13 to 2.22 m³/s between 2010–2019. It is expected to increase further because several new desalination projects in the portfolio of mining companies will be materialized in the coming years [18,19]. However, the RO process creates large volumes of waste brine commonly discharged into the ocean [20,21]. Waste brine is mainly characterized by a high salinity concentration that is approximately 1.5–2 times higher than the seawater concentration, depending on the intake water salinity and the recovery rate of the RO process [22,23]. Despite the high salinity, it may also contain chemical reagents commonly used in the pre-treatment, membrane, and filter cleaning process that is discharged into the waste brine. These include antiscalants, antifouling agents, antifoaming agents, coagulants, biocides, cleaning solutions, pH adjusters, and hardness adjusters [21,24,25]. This situation may negatively impact marine ecosystems if waste brine is not correctly managed [22]. For instance, antiscalants may induce local eutrophication by adding phosphorus (e.g., polyphosphates), while coagulants may enhance water turbidity and coloration [26]. Other potential environmental damage includes a reduction of the abundance and diversity of benthic communities, pH fluctuations, and a heavy metal concentration increase where waste brine is disposed [22,24,26,27].

To the best of our knowledge, there are no records in the literature on the use of waste brine in the froth flotation process of copper sulfide minerals, considering that it is a water-intensive process in copper mining industry. In this context, waste brine discharged from RO desalination plants installed along the coast of the Atacama Desert in Chile presents a potential non-conventional water source for the froth flotation of copper sulfide minerals. The novelty of this research is assessing the potential use of waste brine discharged from RO desalination plants as a source of industrial water in the copper mining industry. To ensure the applicability of this waste stream, the concentration of calcium and magnesium ions must be reduced to improve copper recovery in the froth flotation process. This work proposes to treat waste brine with sodium hydroxide (NaOH) solution as an alkali source to promote the removal of calcium and magnesium ions. In this context, the research questions addressed are: (1) Is it possible to reduce the calcium and magnesium concentration of the waste brine discharged from RO desalination to improve copper recovery copper in froth flotation process? (2) Can treated waste brine increase copper recovery from chalcopyrite with a high presence of clay minerals in froth flotation process?

2. Materials and Methods

The experimental methodology of this research was divided into two stages. The first stage consisted of the removal of calcium and magnesium ions from waste brine samples using a NaOH 10 [M] solution. The second stage consisted of carrying out froth flotation tests to evaluate the copper recovery, copper separation efficiency, and enrichment ratio

using water with different qualities: (i) Seawater, (ii) waste brine, (iii) treated waste brine, and (iv) tap water. It is worth mentioning that the tap water obtained from the RO plant and the seawater were the base cases to contrast the other experimental tests, as they comprised the current situation regarding copper mining operations for the froth flotation process to address the water scarcity problem, as was previously mentioned

2.1. First Stage: Treatment of Waste Brine from RO Desalination

2.1.1. Waste Brine Samples and Reagents

Waste brine samples collected from a desalination plant located in the Atacama Desert were used to perform the experimental tests, and their chemical composition is shown in Table 1. This desalination plant uses an RO process to remove the salts contained in seawater to produce drinkable water for human use. In addition, a NaOH solution [10 M] prepared with NaOH pellets supplied by Merck Millipore was used as the alkali source to react with waste brine, aimed at precipitating calcium and magnesium compounds. Deionized water was used to prepare the NaOH solution [10 M] and to wash all of the materials and experimental units. The experimental tests were carried out in a glass reactor with a capacity of 5 L (Winglass), located above a magnetic stirrer with a respective stirring bar of 4 cm to keep the solution homogeneous throughout the experimental test. A micropipette of 500–5000 μ L was used to add the alkalizing reagent to the waste brine sample. The solution pH was always controlled using a portable pH meter (Hanna portable pH/ORP meter, model HI 98190, Woonsocket, RI, USA). To separate the calcium and magnesium precipitates from the treated waste brine, a glass filtration system of 47 mm disc filters (Glassco) using a 0.45 μ m membrane filter (Merck Millipore, Jaffrey, NH, USA) connected to a vacuum pump was utilized.

Table 1. Chemical composition of waste brine from an RO at pH 758 plant in Atacama Desert, Chile.

Ions	Value (mg/L)	Ions	Value (mg/L)
Chloride	24,231	Potassium	499
Sodium	14,174	Bicarbonate	133
Sulphate	3486	Nitrate	3.94
Magnesium	1693	Fluoride	0.98
Calcium	553	Nitrite	<0.1

2.1.2. Procedure for Waste Brine Treatment

As shown in Figure 1, experimental tests were performed by placing 4 L of waste brine sample in the reactor at room temperature (25 °C), mixed at a constant speed of 300 rpm using a magnetic stirrer for 5 min to homogenize the solution. The reactor had a portable pH meter introduced without touching the bottom of the vessel to monitor and record the pH of the reaction in the solution throughout the experimentation process. Once the waste brine was conditioned, 65 mL of NaOH 10 [M] was added into the reactor using a micropipette to reach pH 11 to precipitate the calcium and magnesium compounds. The pH solution was monitored for one hour, and the experimental test was terminated after this time. The stirring stopped to settle the calcium and magnesium precipitated for one additional hour. These precipitates separate from the treated waste brine using a vacuum filtration system. Atomic absorption spectrometry (AAS) was used to analyze the concentrations of calcium and magnesium ions in the filtered solution. In addition, the treatment of waste brine was carried out in triplicate to ensure good reproducibility of the results.

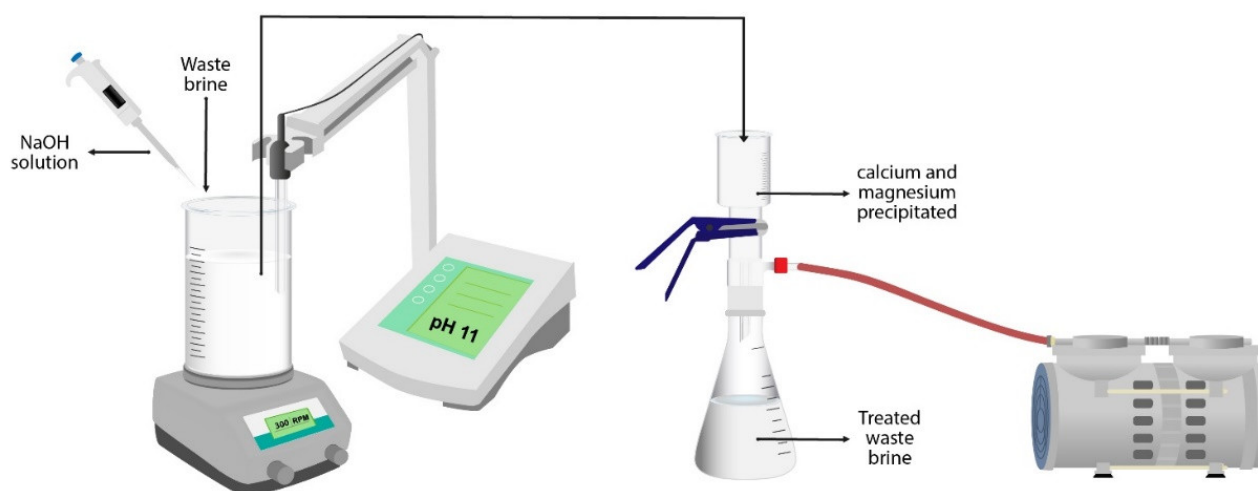


Figure 1. Experimental unit used in the waste brine treatment.

2.2. Second Stage: Froth Flotation Tests of Chalcopyrite with Clay Content

The synthetic ore samples were prepared with three minerals; kaolinite particles, fine granular quartz acquired from Merck, and chalcopyrite provided by Ward's Science S.A. The synthetic ore sample contained 2.9% chalcopyrite, 24.3% kaolinite, and 72.8% quartz. Quantitative X-ray powder diffraction analysis indicated that the kaolinite contained 84% kaolinite and 16% illite. In addition, the kaolinite had a density of 2.6 g/cm³ and an average size of 12 µm, while the average size of the chalcopyrite and quartz was 1680 µm. The experimental tests were carried out in a stainless-steel laboratory ball mill to ground the synthetic ore sample, which was floated using a laboratory-scale Denver flotation machine with a 1 L flotation cell. The pH of the mineral pulp was adjusted using a NaOH solution. In addition, TC-123 as a primary collector (12 g/t) and Matfroth-202 as a frother (10 g/t) were the reagents used in the flotation tests.

Copper Sulfide Ore Samples and Reagents

For evaluating the flotation process of copper from synthetic ore samples using different water qualities, separation efficiency, enrichment ratio, and recovery were considered. As shown in Figure 2, ore samples of 595 kg were ground in a stainless-steel laboratory ball mill with 0.5 L of water and the collector for 14 min to arrive at a particle size distribution with P80 of 105 µm. The mineral pulp is transferred to the flotation cell, and water is added until reaching the volume of the cell (1 L). A solid/liquid ratio of 36% was maintained. The collector, frother, and NaOH solution were added to the mineral pulp. The NaOH solution adjusts the pH of the pulp to 11. The mineral pulp was stirred at 1200 rpm, and the reagents were conditioned for 3 min. Then the mineral pulp was stirred at 1000 rpm, and airflow was introduced into the cell as a flotation gas at a flow rate of 5 L/min. The froth was scraped every 5 s for 12 min (flotation time), and the pH of the mineral pulp was constantly monitored using a portable pH meter. In addition, the AAS method was used to determine the composition of the flotation concentrates. Triplicate tests were performed to ensure good reproducibility of the results.

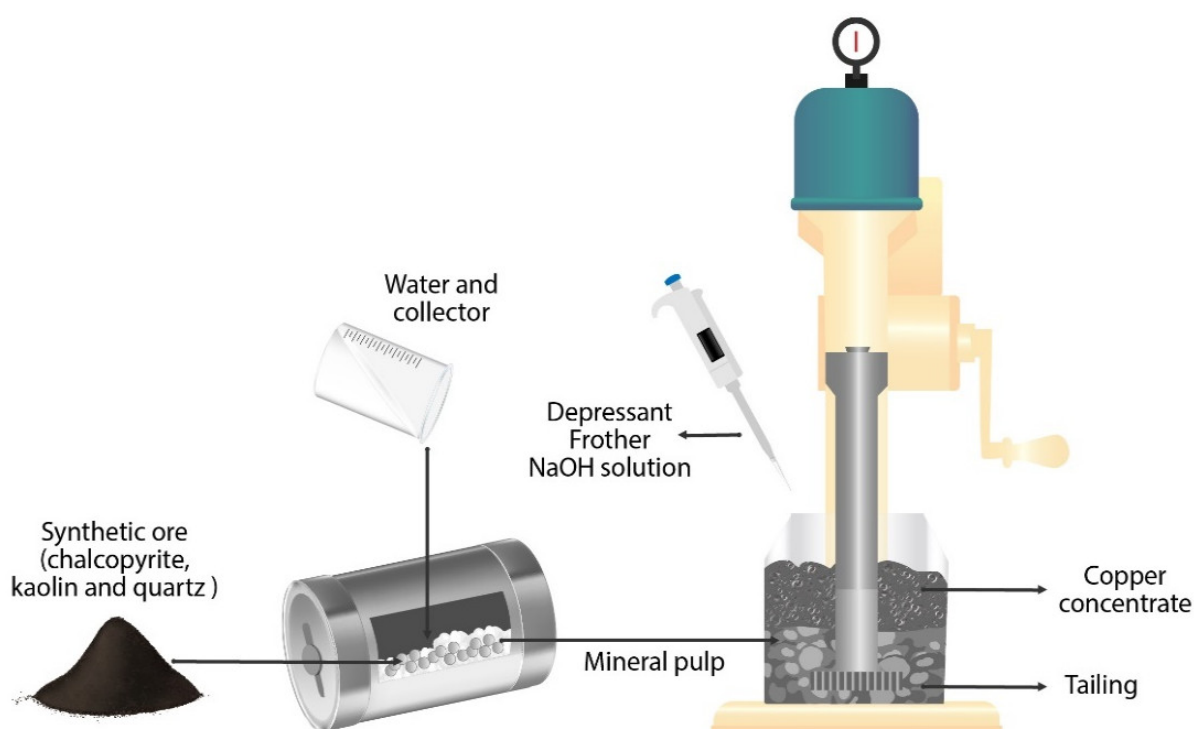


Figure 2. Experimental unit used in the froth flotation tests.

3. Results and Discussions

3.1. Treated Waste Brine

Waste brine treatment aims to reduce the concentrations of calcium and magnesium ions by using treated waste brine as a source of industrial water in the froth flotation process of copper sulfide ores. A NaOH solution [10 M] was used as an alkali source to adjust the pH and control the solution's pH during the waste brine treatment process. Table 2 shows the calcium and magnesium ion concentrations of untreated waste brine and the final concentrations of these ions after treatment. In addition, Figures 3 and 4 show the removal rates of calcium and magnesium for each treated waste brine sample, respectively. From Figure 3, it can be observed that the maximum magnesium removal rate obtained was between 97% and 99% at a pH level over 11 using the NaOH solution, where the maximum magnesium removal rate was achieved at pH 11.90 by reducing the magnesium concentration from 1693 to 0.15 mg/L (see Table 2). Regarding the calcium removal rate, Figure 4 shows that the maximum removal was 23.90% at pH 11.90, reducing the magnesium concentration from 553 to 420.80 mg/L (see Table 2). Meanwhile, for pH 11.15, the calcium removal rate was lower than in the previous case, reaching a value of 14.56% and 10.04% for treated waste brine samples 2 and 3, respectively.

Table 2. Calcium and magnesium ion concentrations before and after waste brine treatment.

Type of Brine	PH	Magnesium (mg/L)	Calcium (mg/L)
Waste brine	8.00	1693	553
Treated waste brine 1	11.90	0.15	420.80
Treated waste brine 2	11.15	45.75	472.50
Treated waste brine 3	11.15	19.80	497.50

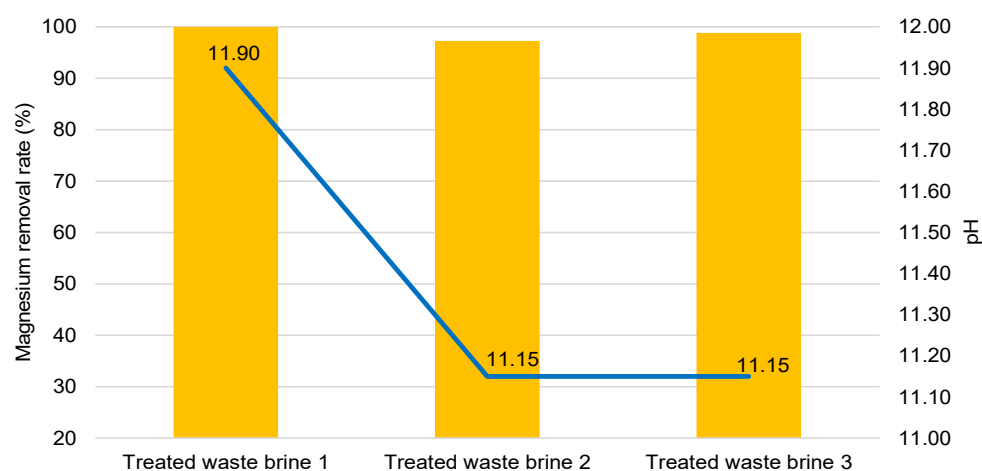


Figure 3. Removal rate of magnesium ions from waste brine at a pH above 11.

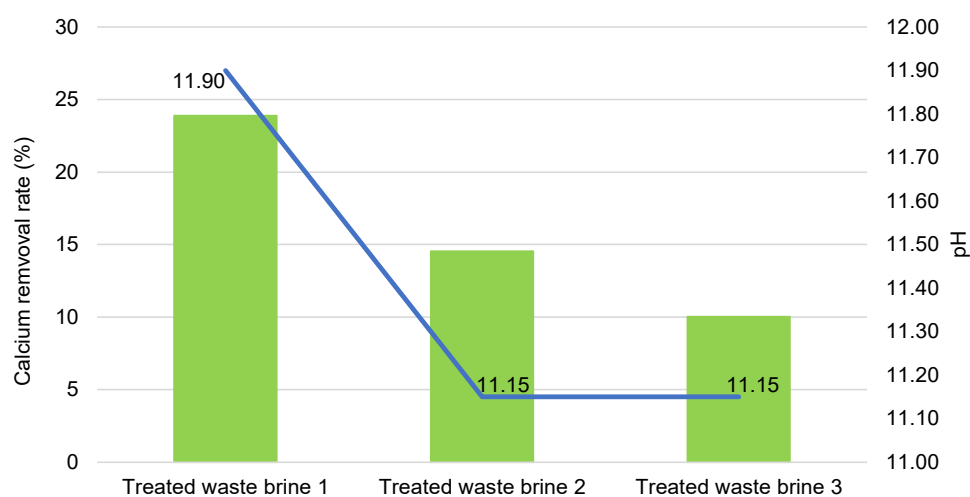
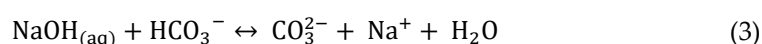


Figure 4. Removal rate of calcium ions from waste brine at a pH above 11.

The precipitation of magnesium and calcium compounds from the waste brine was due to the use of a NaOH solution [10 M], which provides OH^- to reach a suitable pH level to form precipitates. The magnesium precipitation was because the formation of $\text{Mg}(\text{OH})_2$ precipitated through the reaction between magnesium Mg^{2+} in the waste brine and OH^- provided by the NaOH solution. During this process, it was essential to raise the pH level equal to or greater than 11 in the solution to form the $\text{Mg}(\text{OH})_2$ precipitate since the solubility of magnesium starts to decrease at a pH value higher than 10.5. This means that to precipitate $\text{Mg}(\text{OH})_2$ optimally, the pH value must be above 10.5 [28]. The reaction pathways observed during this process are shown in Equations (1) and (2). The addition of a NaOH solution also allowed the conversion of HCO_3^- present in the waste brine to CO_3^{2-} . This situation led to a reaction of the CO_3^{2-} with Ca^{2+} and resulted in the precipitation of CaCO_3 . These two reaction pathways are shown in Equations (3) and (4). However, from Figures 3 and 4, it clearly observed that the magnesium compound precipitation was higher than the precipitation of calcium compounds at a pH level above 11. Raising the pH at 11 led to the CaCO_3 precipitate dissolving, causing a shift (in

Equation (4)) toward the left-hand side. This resulted in the generation of additional CO_3^{2-} , since the CaCO_3 was practically dissolved when the pH was raised to 11. In fact, for the CaCO_3 to precipitate optimally, the pH value should be maintained close to 9.5 since the solubility of calcium begins to decrease at this value, and it should not be raised to pH 11 because otherwise, CaCO_3 is dissolved in the solution [29].



3.2. Flotation of Chalcopyrite Using the Treated Waste Brine

Chalcopyrite flotation is evaluated by copper's recovery, separation efficiency, and enrichment ratio. The flotation test used synthetic mineral samples using different qualities of water (seawater, tap water, waste brine, and treated waste brine). On average, the treated waste brine removed approximately 16.20% and 98.70% of the calcium and magnesium concentrations, respectively, as presented in Table 3. It is worth mentioning that the mineral pulp was adjusted at pH 11 for each water quality in the froth flotation process. This operational condition was utilized because alkaline pH is usually applied to depress non-valuable sulfide minerals in the froth flotation process.

Table 3. Evaluation of the copper flotation process using different water qualities.

Water Quality	Cu (%)	Separation Efficiency (%)	Enrichment Ratio
Waste brine	80.80	19.00	1.3
Seawater	92.40	21.40	1.3
Treated waste brine	94.70	48.70	2.1
Tap water	97.70	45.80	1.9

From Table 3, it is clear that chalcopyrite flotation with clay content using untreated waste brine and seawater was strongly affected since the copper recovery reached 80.80% and 92.40%, respectively. Similarly, separation efficiency and enrichment ratio values present a low performance using both water qualities compared to treated waste brine and tap water. The low flotation performance of seawater and waste brine is because of the generation of hydroxy complexes of calcium and magnesium species (CaOH^+ , MgOH^+ , and $\text{Mg}(\text{OH})_2$) that under alkaline conditions cause heterocoagulation between the kaolinite and chalcopyrite. The heterocoagulation phenomenon causes a hydrophilic coating formed by the clay, which adhered to the surface of the chalcopyrite, preventing their interaction with bubbles and/or collector, resulting in the depression of chalcopyrite [30,31]. Figure 5 shows the interaction mechanism of kaolinite with chalcopyrite. The heterocoagulation phenomenon has been widely studied in the literature, as stated by Yepsen et al. [32] and Hu et al. [33].

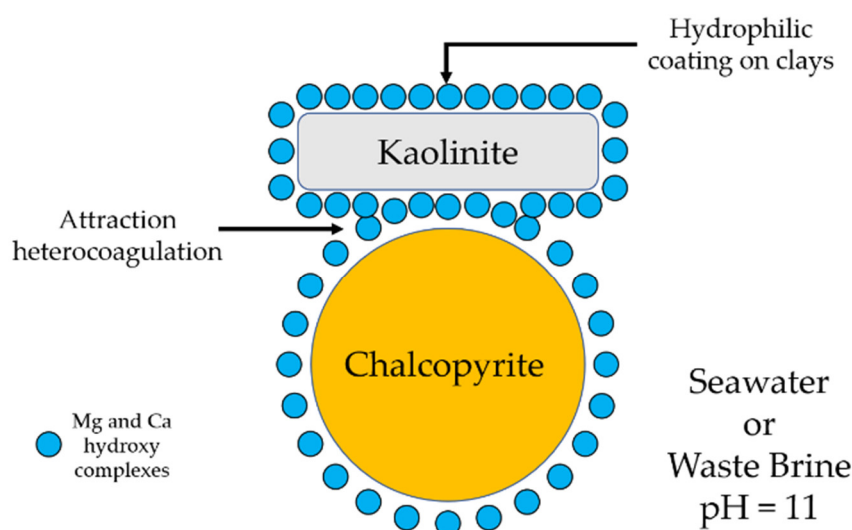


Figure 5. Schematic illustration of the interaction mechanism between kaolinite and chalcopyrite using seawater or waste brine at pH 11. Adapted from [32].

Interestingly, the copper recovery using waste brine was lower than seawater when the mineral pulp was adjusted to pH 11. This situation might be explained by the fact that waste brine from the RO process has a high salinity between 1.5 to 2 times higher than seawater, which depends on the intake water salinity and the recovery rate of the RO process. In this context, the high concentration of divalent ions in waste brine may generate more hydroxy complex species at pH 11, which increases the heterocoagulation between the kaolinite and chalcopyrite, reducing the floatability of copper.

In contrast, when flotation of chalcopyrite was carried out using treated waste brine, copper recovery reached 94.70%, representing an increase of approximately 14% and 2% when waste brine and seawater were used, respectively. The treated waste brine separation efficiency was 2.6 and 2.3 times higher than waste brine and seawater, respectively. In addition, the enrichment ratio using treated waste brine reached the highest value among other water qualities. Similar behavior was observed when chalcopyrite flotation with clay content was carried out with tap water at pH 11, reaching 97.70% copper recovery. The high flotation performance using the treated waste brine was because the concentrations of calcium and magnesium ions decreased, on average, by approximately 16.20% and 98.70%, respectively, compared to the waste brine. Using the treated waste brine reduced the generation of calcium and magnesium hydroxy complex species, whose speciation depends on each element's initial concentration and the solution's pH. Consequently, the heterocoagulation between chalcopyrite and kaolinite was reduced, reaching a higher copper recovery in the froth flotation process. Another factor that may have favored the high copper recovery using treated waste brine was the high electrolyte concentrations of sodium and chloride, which can reduce the bubble size, preventing bubble coalescence in the froth flotation process. In this context, treated waste brine could be a promising alternative to address the challenges generated by clays. It may be an adequate water quality for copper mining companies currently using seawater in the froth flotation process. In addition, the use of treated waste brine allows it to turn into a useful product as industrial water for the froth flotation process, reducing waste brine's disposal into marine ecosystems.

4. Conclusions

The paper's novelty consists in evaluating the potential use of waste brine discharged from RO plants as a source of industrial water in copper mining industry. On average, the treated waste brine removed approximately 16.20% and 98.70% of the calcium and magnesium concentrations at a pH level above 11, respectively. These low calcium and

magnesium ions concentrations increased copper recovery to 94.70% compared to untreated waste brine and seawater, which were 80.80% and 92.40% of copper recovery, respectively. In addition, similar copper recovery was achieved when chalcopyrite flotation was performed with treated waste brine and tap water, obtaining 94.70% and 97.70% copper recovery, respectively. The treated waste brine reached the highest separation efficiency and enrichment ratio among the other water qualities used. It is worth mentioning that seawater and tap water are the streams currently used in copper mining operations for the froth flotation process to address the water scarcity problem in Chile. The effectiveness of treated waste brine is because it prevents the formation of complex precipitates of calcium and magnesium, which are detrimental to froth flotation. Therefore, treated waste brine could become an alternative water source for the flotation of chalcopyrite with high clay content, providing an opportunity to use it as a source of industrial water in the froth flotation process and, at the same time, reducing the large volume of waste brine discharge into sea.

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