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Evaluation of Water Softening with the Removal of Calcium Ion by Ion Flotation Approach

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Abstract – Ion flotation is an efficient method to remove metal ions from aqueous solution. In this work, ion flotation was applied to calcium removal from aqueous solution. The parameters used included sodium stearate (SS) and sodium dodecyl sulfate (SDS) as collectors, 1-butanol and 1-propanol as frothers, pH, and air-flow rate. An L16 orthogonal array was chosen according to the mentioned factors and levels, and experimental tests were conducted according to the Taguchi orthogonal array. The results showed that all of the factors except one had significant effect on the flotation performance. The percentage contribution of parameters showed that type of frother and type of collector made the greatest (43.14%) and the lowest (9.86%) contribution, respectively. In optimal conditions, the recovery of Ca (II) ion was 45.67%. Also, the results illustrated that the Taguchi method could predict calcium removal from aqueous solution by ion flotation with 2.63%. This study showed that the use of ion flotation was an effective method for Ca (II) ion removal from aqueous solution.

Key words: Water softening, Hard water, Calcium ion, Experimental design, Taguchi method

1. Introduction

Water plays an important role in all vital human, as well as in physical, chemical and biological, processes. Water quality protection is one of the most important pillars of sustainable development. Water quality can be studied from different perspectives, one of which is drinking water quality. One of the factors that determines the quality of water for drinking is the mineral compound content of the water: Minerals such as calcium, magnesium, strontium, iron, zinc, aluminum and manganese cations with anions carbonate, sulfate, chloride and silicate cause water hardness. However, calcium and magnesium are the main sources of water hardness [1]. Calcium is the most plentiful mineral that has a structural and functional role in the human body [2]. However, there is a significant relationship between the level of calcium in drinking water and some diseases such as colorectal, gastric and breast cancer [3-4]. The hardness of the water also causes problems in various industries, such as the formation of sediment on the boiler wall and hot water pipes. This causes the boiler wall to thicken, resulting in reduced heat transfer rates, increased fuel consumption, increased heating costs, increased maintenance costs, and reduced service life. So the best way to avoid the health and environmental hazards from water hardness is to reduce the amount of calcium in the water. For the removal of divalent ions (such as calcium) various methods have been widely applied for water softening including, electrochemical processes [5], enzyme catalyzed [6], nanofiltration [7], electro-dialysis [8], ultrasound [9], ultra-filtration [10], ion-exchange [11], membranes [12] and pulsed spark discharge [13]. Each of these methods has its advantages and disadvantages. The attention for developing new techniques to water softening containing calcium and magnesium ions has grown significantly in recent years. Therefore, ion flotation can be one of the alternative methods to remove water hardness. Our aim of this study is to present an ion flotation method to reduce water hardness for which, to our knowledge, there have not been any reports.

The basis of the ion flotation process, an approach mainly used in the mineral process industries, is to transport particles by air bubbles to the surface and remove them as foam. Ion flotation is one technique of recovering or removing metal ions from water and wastewater. This method has some advantages such as low energy consumption, high speed of process, small space requirement, the high flexibility in diverse ions removal at different scales, low sludge production and high efficiency [14-15].

There are several important factors such as pH, air flow rate, temperature and the chemical reagents added to the system such as collector and frother that affect the ion flotation process in reducing water hardness [16]. The classical method of studying one variable while keeping other variables constant may be effective in some processes but does not consider the combined effects of different factors. Also, this strategy needs a relatively great number of experiments and often difficult to predict optimal conditions. This essential defect is due to the inability of this approach to ignore the effects of possible interactions between the parameters. The shortage can be overcome by using more efficient, statistically based experimental design.

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Statistical experimental designs, such as the Taguchi orthogonal design, can collectively optimize all the affecting parameters to eliminate the limitations of a single-factor optimization process [17]. In this respect, the Taguchi approach is an important tool for determining optimal process conditions. The advantages of using the Taguchi method are as follows: simultaneous evaluation and optimization of several parameters, obtaining much quantitative information by only a few experiments. This method has been successfully and extensively applied for enhancing the performance of different processes, parameter optimization and process control in our experiments [17-19]. This approach as an effective statistical technique was used for process optimization. Thus, the aim of the current study was to investigate the water softening by ion flotation method and variables optimization using an orthogonal array of Taguchi methodology. Although the ion flotation potential for metal ion removal has been investigated for many heavy metal ions, so far this technique for water softening has not been reported.

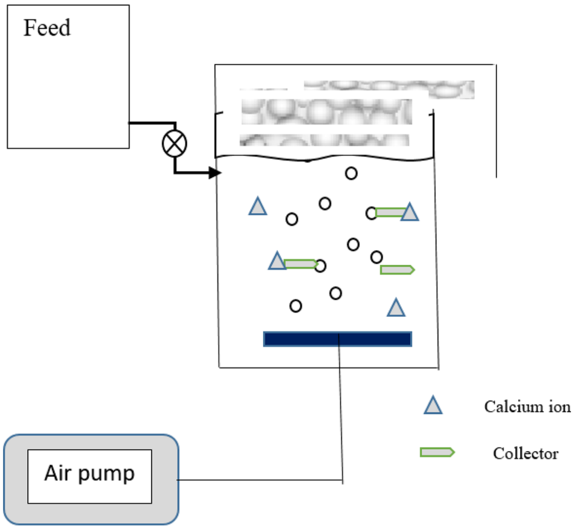

Fig. 1. Schematic diagram of experimental setup for ion flotation method.

2. Experimental

2-1. Chemicals

The chemicals, including sodium stearate, sodium dodecyl sulfate (as collector), 1-butanol, 1-Propanol (as frothers), and ethylenediaminetetraacetic acid (EDTA) (for titration of calcium) were supplied from Merck with minimum purity of 99.0%, 85.0%, 99.5%, 99.5%, and 98.0%, respectively. Also, Eriochrome Black T (as complexometric indicator), NaOH and HCl (for pH-adjustment) were obtained from Merck. All the chemicals used in this study were of analytical grade. The stock solution of calcium ion (200 ppm) was prepared by dissolving 0.735 g $CaCl_2{\cdot}2H_2O$ in distilled water and then diluting to 1L by distilled water. Double distilled deionized water was used to prepare the samples.

2-2. Experimental setup

A schematic diagram of the experimental setup is shown in Fig. 1. In this work, a glass cylindrical vessel with 12 cm internal diameter and 20 cm height (with a working volume of 2L) was used as the flotation tank. A porous ceramic sparger was installed as a gas distributor at the bottom of the vessel. A peristaltic pump was utilized to supply the air. The gas flow rate can be controlled by a peristaltic pump and a rotameter.

The experimental apparatus was run in batch mode. The synthetic sample used in this work was prepared from the stock solution of calcium with collector and frother in the desired pH (Table 1). The ion flotation was carried out for 4 hours and the foam was separated from the surface of the vessel. Each experiment was performed in duplicate to understand the reproducibility. Finally, the average value was presented for each set of values.

Table 1. Experimental L16 orthogonal array and results the calcium ion removal efficiency (%)

Exp. No.	Factor levels				Removal efficiency (%)
	type & con. of collector (ppm)	type & con. of frother (%V/V)	pH	air flow rate (ml, min)	
1	SDS (100)	1-But (1.0)	5	5	12.18
2	SDS (100)	1-But (0.5)	7	20	24.80
3	SDS (100)	1-Pro. (1.0)	9	35	11.68
4	SDS (100)	1-Pro. (0.5)	11	50	1.38
5	SDS (10)	1-But (1.0)	7	35	8.40
6	SDS (10)	1-But (0.5)	5	50	3.09
7	SDS (10)	1-Pro. (1.0)	11	5	19.56
8	SDS (10)	1-Pro. (0.5)	9	20	4.87
9	SS (100)	1-But (1.0)	9	50	8.39
10	SS (100)	1-But (0.5)	11	35	39.01
11	SS (100)	1-Pro. (1.0)	5	20	0.78
12	SS (100)	1-Pro. (0.5)	7	5	13.83
13	SS (10)	1-But (1.0)	11	20	22.96
14	SS (10)	1-But (0.5)	9	5	41.83
15	SS (10)	1-Pro. (1.0)	7	50	11.45
16	SS (10)	1-Pro. (0.5)	5	35	1.49

2-3. Experimental design and statistical analysis

The design of Experiments (DOE) is used to optimize the process parameters and to investigate the effect of parameters on response [20]. The Taguchi method has been used to develop an appropriate strategy to perform experiments as well as for quality control under optimal conditions [18-19]. This method is a combination of mathematical and statistical techniques used in an empirical study [21]. It is especially used to evaluate several process parameters at a time with the least number of experiments based on a table, known as the orthogonal array. In the present study, the factors--type and concentration of collector, type and concentration of frother, pH solution and air flow rate--were considered at two and four levels by the L16 symbolic array. The array and levels of the experimental factors are given in Table 1. The dependent parameter for each experiment in the experimental design was the efficiency of calcium ion removal from aqueous solution. According to Table 1, the total number of required trials was only 16 runs as opposed to 1024 for a full factorial experiment. Analysis of variance (ANOVA) was used to manage the data analysis and to determine the relative importance of each parameter. Also, it was applied to examine the significance of the influence and reliability of processing parameters on performance. The p-values were used as a means of examining the significance of each variable, which also indicates the power of interaction between each independent variable [18]. The significance of all terms was statistically determined by calculation of the p-value <0.05. All calculations and optimization process were carried out using Minitab statistical software version 18.

2-4. Analysis of samples

The amount of calcium ion present in the each sample (before and after a run) was analyzed by titration. The calcium ion concentration in each sample was determined by titration with EDTA solution of 0.05 M. Eriochrome Black T solution was used as an indicator for the titration. Initially, a calibration curve was prepared using a standard calcium ion solution (200, 150, 100, 50 and 10 ppm).

The calcium ion removal efficiency (Y%) was calculated as the removal percentage of calcium ion from aqueous solution in accordance with the following equation:

$$\text{Calcium ion removal efficiency (Y\%)} = \{(C_i - C_f) / C_i\}*100 \quad (1)$$

where C_i and C_f are the initial and final calcium ion concentrations in aqueous solution.

3. Results and Discussion

3-1. Effects of independent variables on responses

Increasing demand for fresh water due to the world's population growth and development of industrial applications is making the water supply an imperative issue. The presence of some minerals, such as calcium in the water, makes it hard water. Hard water can pose critical problems in industrial fields, including breakdowns in boilers, cooling towers, and other equipment that handles water. Where water hardness is a concern, water softening is usually used to reduce the side effects of hard water. Various methods are commonly used for this purpose. Ion flotation technique may be an alternative method to water softening. The process of ion flotation is based on imparting the ionic metal species in water by use of surface active agents and subsequent removal of these species by air bubbles. The ion flotation approach is affected by several environmental parameters such as pH, air flow, temperature and the chemical reagents added to the system such as collector, and frother. [22]. So it is known that each chemical process needs specific operating conditions [18]. In this case, the influence of four factors was studied: type and concentration of collector, type and concentration of frother, pH solution and air flow rate in water softening process for calcium ion removal from aqueous solution using Taguchi experimental design in 16 runs. The main effects of each parameter are presented in Fig. 2, which acts as a scale to study individual variables' contributions on the calcium ion removal yield. This was calculated based on the averages of evaluation made at the level of each parameter.

One of the important factors in the ion flotation process is collector type. In this study, two types of anionic surfactant as collector (sodium dodecyl sulfate and sodium stearate) with different concentration (10 and 100 ppm) were investigated for calcium ion removal and water softening. The results showed that sodium stearate had more effect on the removal of calcium ion from water than sodium dodecyl sulfate (Fig. 2A). The reason for this may be due to the longer hydrocarbon chain of sodium stearate than sodium dodecyl sulfate. There is a direct relationship between the hydrocarbon chain length of the collector and the ion adsorption capacity in aqueous solution [16]. Therefore, sodium stearate with 18-carbon chain has higher calcium ion absorption capacity than sodium dodecyl sulfate with 12-carbon chain.

Also, the results showed that increasing sodium dodecyl sulfate concentration increased calcium ion removal efficiency, whereas the increased concentration of sodium stearate decreased the efficiency (Fig. 2B). This behavior may be related to the critical micelle concentration (CMC) of the two surfactants studied. Since micelles are formed at the higher concentration than the CMC and micelle formation reduces the efficiency of the surfactant as collector for the ion flotation process. Therefore, since the CMC of sodium dodecyl sulfate is greater than that of sodium stearate (2364 ppm and 92 ppm, respectively). Our observations show that the effect of surfactant concentration in the ion flotation process depends on the surfactant CMC. Increasing the surfactant concentration to the CMC increases the efficiency of the process, but increasing the surfactant concentration to a higher concentration than the CMC decreases the efficiency due to micelle formation. So increasing the sodium stearate concentration to 100 ppm decreased the efficiency calcium ion removal.

Frothers form a stable foam layer by reducing the surface tension and size of the bubbles. So that if the bubbles do not have the required stability and are destroyed before they leave the system, the loaded ions by bubbles will return to the solution [23]. Hence, one of the

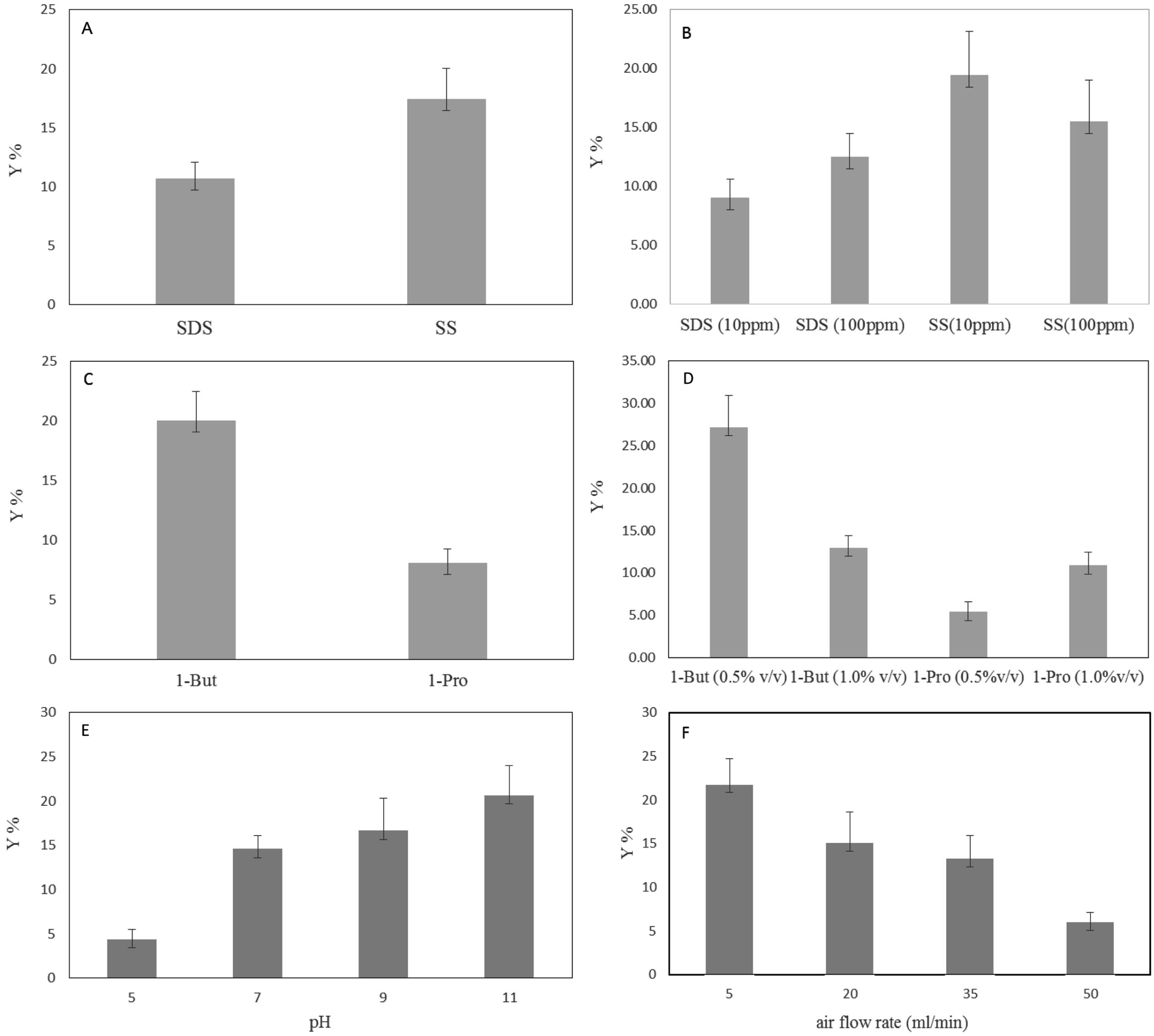

Fig. 2. Main effects of the variable for the calcium ion removal efficiency based on the Taguchi experimental design results as function of: (A) type collector; (B) collector concentration; (C) type frother; (D) frother concentration; (E) pH; (F) air flow rate.

effective parameters in the ion flotation approach is the choice of type and concentration of the frother. In this study two types of alcohols, 1-butanol and 1-propanol, were used as frothers with different concentrations of 1.0 and 0.5 (%v/v). The results showed that 1-butanol was more effective than 1-propanol in the ion flotation (Fig. 2C). This effect may be due to the higher molecular weight of 1-butanol than 1-propanol [23].

The results also showed that increasing the concentration of alcohol as frother decreased the efficiency (Fig. 2D). Wang *et al.* [24] showed that the addition of chemical compounds into pure water produces a strong effect on dampening bubble deformation. Perhaps the excessive concentration of alcohol in the solution reduces the removal efficiency by deforming the air bubbles. Also, the interaction between collector and frother affects the ion flotation process [14]. The extra alcohol can form hydrogen bonds with collector oxygen atoms. Hydrogen in the frother structure of alcohol and oxygen atom in the SDS collector form hydrogen bonds and provide a situation that may influence the recovery of ions. Mahmouda and Lazaridis [23] also reported that increasing the ethanol concentration to more than 0.6 (%v/v) reduced the removal efficiency of nickel (II) and chromium (VI).

The effect of medium pH on calcium flotation recovery was studied at different pH values of the solution. Since, the collectors, especially SDS, are effective in both acidic and alkaline solutions. The pH was adjusted within the range of 5-11. The results showed that calcium ion removal is strongly dependent on the pH of the solution (Fig. 2E). In acidic medium calcium ion removal rate was negligible but in alkaline medium the removal percentage increased. An almost similar kind of trend for nickel (II) cations removal by ion flotation process was observed by Mahmouda and Lazaridi [23]. They also reported that a sharp increase in the removal percentage was found at $pH > 9$ and maximum removal of about 97% was achieved a pH = 12.4. This may be due to the competition between the hydronium ion and the calcium ion for binding to the collector in acidic solution. Also, sodium stearate in acidic medium is mostly in the form of stearic acid, which results in a decreased binding affinity of the collector to the calcium ion, which results in a lower removal efficiency. However, increasing the pH does not always lead to a higher removal percentage. At high pH value and the formation of calcium hydroxide, its solubility drastically decreases.

In general, air flow rate is an essential step in the ion flotation process. Bubbles play the role of transporting the desired ions to the surface. Although increasing air flow rate increases the number of bubbles and consequently the number of efficient collisions, it can be seen that higher air flow rate causes larger diameter bubbles, which can decrease the efficiency of the flotation process [25].Therefore, the effects of air flow rate within the range from 5 to 50 ml/min on the removal efficiency were evaluated. The results in Fig. 2F show that the continuous decrease in the removal efficiency was understandably contributed with increasing air flow rate observed and 5 ml/min was the best air flow rate for the efficiency calcium ion removal.

An increase in air flow rate causes the bubbles to form together and form larger bubbles, thereby reducing the percentage of calcium ion removal. The smaller bubbles provide a bigger surface area, which results in the higher kinetics of the separation process. Hoseinian *et al*. [25] studied the effect of bubble size on the percentage of nickel ion removal. They showed that smaller bubbles performed better than larger bubbles.

3-2. Analysis of the experimental design

The yield of calcium ion removal from aqueous solution was found to range from 0.78 to 41.83% in response to the variation in the experimental conditions (Table 1). The results from the analysis of the experimental design are shown in Table 2. The degree of significance of each factor is represented in Table 2 by its *p*-value; when a factor has a *p*-value of less than 0.05 it influences the process in a significant way for a confidence level of 0.95. The results obtained show that all of factors--type and concentration of frother, pH solution and air flow rate except type and concentration of collector--were significant (*p*-value < 0.05). The results indicated that the type and concentration of frother were the major contributing factors to the yield of calcium ion removal from aqueous solution.

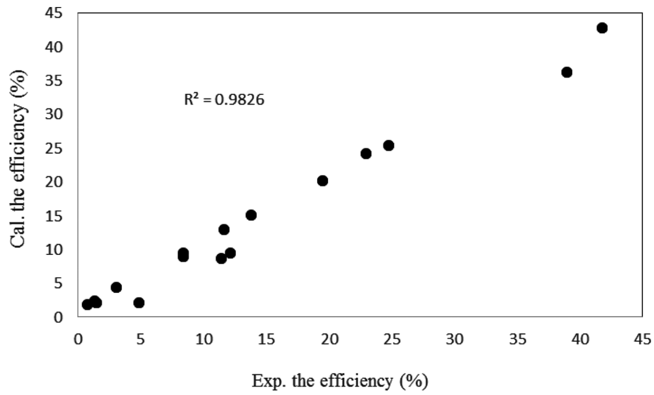

Fig. 3. The relationship between the calculated the calcium ion removal efficiency and experimental data.

The quality of the experimental of design developed was evaluated based on the value of coefficient of determination (R^2). Coefficient of determination (R^2) is defined as the ratio of the explained variation to the total variation and used to measure the degree of fitness. In this case, the R^2 value was 0.983, that Joglekar and May [26] suggested for a good fit of a model, R^2 should be at least 0.80. This implied that 98.26% of the variation for the yield of calcium ion removal is explained by the independent variables and only 1.74% of the total variability in the response is not explained by the model. The relatively high value of R^2 (0.983) demonstrates a high degree of agreement between the experimental observations and predicted values (Fig. 3).

3-3. Optimum conditions and model verification

Optimum conditions for the efficiency of calcium ion removal purification were obtained using the Taguchi method as presented in Table 3. Under such conditions, the yield of calcium ion removal process was predicted to be 46.87%.

The suitability of the Taguchi orthogonal array for predicting the optimum response value was tested by additional independent experiments using the recommended optimum conditions. The results indicate that the yield of calcium ion removal from aqueous solution (45.67%) is not significantly different from the predicted value (46.87%) (Table 3).

Table 2. Analysis of variance of the regression parameters for the Taguchi experimental design

Source	D. F.	Sum of Square	Mean Square	*F*-value	*p*-value
type & con. of collector	3	236.58	78.86	5.66	0.094
type & con. of frother	3	1034.68	344.89	24.75	0.013
pH	3	581.19	193.73	13.90	0.029
air flow rate	3	504.29	168.10	12.06	0.035
Residual Error	3	41.80	13.93		
Total	15	2398.54			

Table 3. Optimum conditions of the calcium ion removal efficiency (%), experimental and predicted from the Taguchi experimental design

Optimum Condition				The TPH removal efficiency (%)	
type & con. of collector (ppm)	type & con. of frother (%v/v)	pH	air flow rate (ml/min)	Cal. Value	Expt. value
SS (10)	1-But (0.5)	11	5	46.87	45.67

4. Conclusions

This work presents the use of ion flotation processes as an alternative tool for water softening. Several parameters, such as type and concentration of collector, type and concentration of frother, pH and air flow rate were tested to determine the optimum flotation conditions on a small scale. The results, which were analyzed by Minitab software, showed that the SS as collector, 1-butanol as frother, pH = 11 and air flow rate = 5 ml/min were optimal conditions in the ion flotation of Ca (II) ion. In these conditions, the recovery of Ca (II) ion was achieved at 45.67%. The study showed that ion flotation has good performance for the removal of cations such as Ca (II) ion from aqueous solution.

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