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The effect of water recovery on the ion flotation process efficiency

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Abstract: The present study deals with nickel ions removal from dilute aqueous solution by ion flotation with emphasizing the process efficiency. The effect of collector structure on ion flotation efficiency and water recovery was evaluated using anionic collectors of sodium dodecyl sulfate (*SDS*) and functionalized graphene oxide by 2,6-diaminopyridine (*AFGO*). The results showed that process efficiency enhanced with the increase in pH and reached to complete removal at pH of 9 and 9.7 for *SDS* and *AFGO*, respectively. The *AFGO* showed the multifunctional bindings for complex formations with nickel ions. A coordinate bond may be formed between nickel ions and *AFGO* at the pH of 9 which increased nickel ion removal. The water recovery as a critical parameter that contributes to removal efficiency was significantly affected by the collector structure. The *AFGO* doesn't have a frothing property and so decreases the water recovery during the process. The *AFGO* had significantly lower water recovery than *SDS* (almost threefold).

Keywords: ion flotation, collector structure, process efficiency, wastewater, water recovery

1. Introduction

Flotation is a process that can be considered the most important mineral processing technique of the past century. The efficiency of this process depends on the recovery of the minerals by both true flotation and entrainment (Cilek, 2009; Neethling and Cilliers, 2009; Safari et al., 2016; Lima et al., 2016; Safari et al., 2020). The recovery of the flotation is strongly dependent on physical and chemical parameters, such as the cell hydrodynamics, pH, reagents types and dosages (Safari et al., 2017; Testa et al., 2017; Hoseinian et al., 2018a; Hoseinian et al., 2018c).

The froth recovery and entrainment during the flotation process play important roles in flotation efficiency which is controlled by the water recovery percentage in the froth phase. Entrainment is strongly dependent on the amount of water carrying medium into the froth phase. Therefore, flotation efficiency is size-dependent separation (Hassanzadeh and Karakaş, 2017; Safari et al., 2018; Safari et al., 2020) and the relationship between the water recovery and the recovery by entrainment shows a linear trend for fine particles (particles smaller than 25 µm). The main mechanism for the recovery of gangue particles to concentrate in the froth flotation is entrainment (Akdemir and Sönmez, 2003; Yang and Aldrich, 2006; Cilek, 2009; Neethling and Cilliers, 2009). In flotation, water is an important component of feed and recovery of water used regularly in the simulation and modeling of flotation, more specifically in kinetics studies. Although water recovery has generally been considered as an independent parameter, it is evident that it is a critical independent process parameter. For example, different collectors and frothers produce different carrying rates of water into the overflow (Moyo et al., 2007). This means that the collector structures can influence water recovery (Cho and Laskowski, 2002; Grau et al., 2005; Gupta et al., 2007; Wang et al., 2020).

The world's population enhancement is led to industrial production increases, which makes plenty amount of industrial wastewater (Krishnan et al., 2010; Ji et al., 2011; Anoop Krishnan et al., 2015; Jang et al., 2017). Sebba in 1962 for the first time introduced "ion flotation" as a process for separation (Sebba, 1962). Generally, in the ion flotation, a surface-active ion with the opposite charge to the ion that needs to be separated is added to the solution (Doyle and Liu, 2003; Zheng et al., 2006; Lemlich, 2012). This process has many advantages such as simplicity, good separation yields, rapid operation, low energy requirement, and economic benefits that make ion flotation a commercial technic for heavy metal ions removal from wastewater with low concentrations (Hoseinian et al., 2018b; Hoseinian et al., 2019b).

In opposition to the common flotation practices where the valuable minerals separated selectively, in ion flotation the species which are separated are the metal ions. This means the ion flotation is a process for separating ions selectively. The efficiency of this process normally is measured by the metal content and the amount of water in the concentrate. Therefore, a large ratio of metal ion to water in the concentrate confirms the success of metal ions removal. On the other hand, the selectivity of this process normally is assessed based on both the water and metal ions recoveries during the flotation process. The volume of water in the froth phase is a critical variable that has a high effect on the ion flotation efficiency (Zheng et al., 2006; Polat and Erdogan, 2007). Although there are many studies on ion flotation, the lack of information on the water and metal removals during the flotation process is obvious and only a few studies have investigated these factors (Polat and Erdogan, 2007; Hoseinian et al., 2018c; Hoseinian et al., 2019c). Therefore, understanding of these critical variables is necessary for reaching to maximum ions recovery and minimum water recovery. In this regard, the scope of this study was to investigate the optimum conditions for the removal of nickel ions from wastewaters by ion flotation with particular focus on the nickel ion removal from solution and water recovery during the flotation process. In this study, the effects of critical variables such as pH and collector type were also evaluated.

2. Materials and methods

Sodium dodecyl sulfate (*SDS*) and functionalized graphene oxide by 2,6-diaminopyridine (*AFGO*) were used as anionic collectors. *AFGO* was produced by the method presented in the previous study (Hoseinian et al., 2020). For removal of nickel ions with a positive charge in the solution, the negative charges onto the amino functionalized graphene oxide was created using 2,6-diaminopyridine to modify the properties of amino functionalized graphene oxide. All used reagents were provided from Merck Company and utilized in analytical grade.

Wastewater with the nickel ion concentration of 10 mg/dm^3 was prepared using dissolving nickel(II) nitrate ($(\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O})$) in double-distilled water. The primary nickel concentration in all experiments was 10 mg/dm^3 . The ion flotation experiments were carried out in a Denver type laboratory mechanical flotation cell with a 1 dm^3 volume. In all experiments, an impeller speed of 800 rpm was considered according to the previous study for the energy input effect on the ion flotation process efficiency (Hoseinian et al., 2019b).

Firstly, the pH of wastewater was adjusted to the desired value by HCl and NaOH and stirred at 800 rpm for 5 min. In the nickel ion flotation with *AFGO* as a collector, after the pH adjustment, 0.1 g/dm^3 of *AFGO* was added to the solution and agitated for 6 min, then, 0.05 g/dm^3 of *SDS* was added and stirred for 3 min. In the nickel ion flotation with *SDS* as a collector, the 0.1351 g/dm^3 of *SDS* was added to the solution and agitated for 3 min, thereafter, 0.020 g/dm^3 Dowfroth 250 as a frother was added and stirred for 3 min. After the preparation time, the flotation was begun by introducing $1 \text{ dm}^3/\text{min}$ air. At the end of nickel ion flotation, the nickel ion concentration in the solution was measured using the atomic absorption spectrometry (AAS, Perkin Elmer AA300 model). Then, the formed precipitate (sublate) in the froth phase was studied. Fourier-transform infrared spectroscopy (FTIR, Perkin Elmer-E100 Company) was used to assess the effect of collector type and chemical interactions on the removal process. Furthermore, sublate morphology characterization in the optimal conditions was studied by scanning electron microscope (SEM, The Philips XL30 model).

3. Results and discussion

Ion flotation as a practically promising method has been widely applied for wastewater treatment and mineral processing applications to recover and or remove different types of ions with a low

concentration (Doyle, 2003). The successful ion flotation with high efficiency simultaneously depended on the high ion removal and low water recovery. Accurately estimating water recovery is crucial when evaluating the performance of the ion flotation process as it is essential to specify the ion removal achieved. The collector type plays the main role in the removal efficiency of ions using ion flotation (Peng et al., 2019). On the other hand, change of the solution pH varies the nickel ion species and complex solubility of collector and nickel ions. In this regard, the effects of collector type and pH on the recovery of water and process efficiency of nickel ion flotation were evaluated.

3.1. Effect of collector type and pH

The ion flotation process is a separation method based on ion transport onto the interface of the solution-vapor using an ionic collector with the opposite charge of ions in the solution. Ni^{2+} and $\text{Ni}(\text{OH})^+$ with positive charges are the predominant ion species at pH values less than 9.7 (Hoseinian et al., 2019a). Thus, the range of pH of 3-9.7 was used to investigate nickel ion flotation using anionic collectors of *AFGO* and *SDS* with negative charges. The optimum conditions of removal of nickel ions using a common collector of *SDS* and a novel nano-collector of *AFGO* were completely evaluated in the previous studies (Hoseinian et al., 2018c; Hoseinian et al., 2020). The effects of pH and collector type on the nickel ion flotation and water recovery are shown in Figs. 1 and 2. The nickel ion removal enhanced with the increasing pH and the maximum removal of nickel ions was obtained at pH 9.7 and 9 for *SDS* and *AFGO*, respectively. As can be seen from Figs. 1 and 2, both collectors *AFGO* and *SDS* can be effectively used to remove high nickel ions in the optimum conditions. The water recovery during the process using *SDS* as the collector increased with the increasing solution pH from 3 to 5.5 and then decreased. While the water recovery in the presence of *AFGO* decreased with the increasing solution pH. The water recovery using the *AFGO* as the collector in the nickel ion flotation in optimum conditions was approximately 26.5% lower than the water recovery using the *SDS* as the collector.

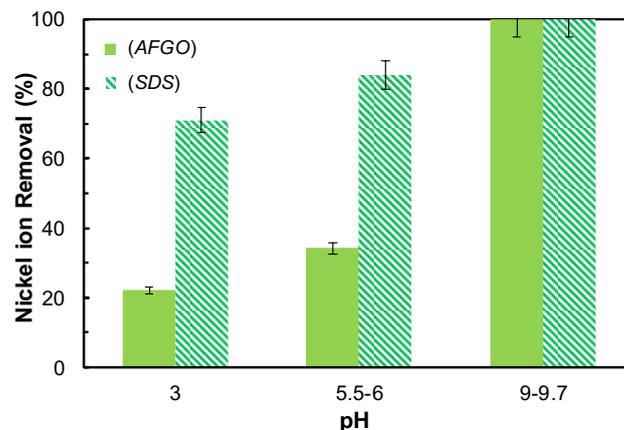


Fig. 1. The effect of collector type and pH on the nickel ion flotation

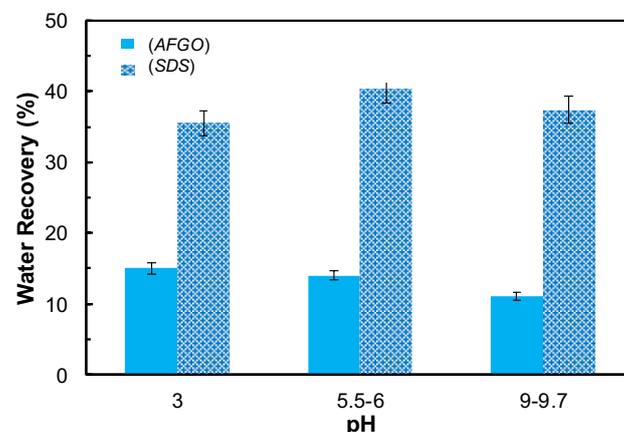


Fig. 2. The effect of collector type and pH on the water recovery

3.1.1. Effect of the collector structure

The collector structures have a significant effect on the process efficiency and the required collector concentration to remove ions. The collector structures of *SDS* and *AFGO* are shown in Fig. 3.

SDS has been successfully used as a collector for ion flotation of various ions in the literature (Niraula et al., 2014). Moreover, *SDS* has a frothing property that allows it to form foam in the ion flotation process, and so act as a frother. On the other hand, the adequate collector concentration for achieving high nickel ion removal depends on the magnitude of nickel ion charges in the solution (Shakir et al., 2010). There is a stoichiometric relation between *SDS* collector ions and nickel ions for maximum removal of nickel ion which led to an enhancement in the *SDS* concentration. The required *SDS* concentration to remove nearly complete nickel ions should be at least stoichiometric value and below its critical micelle concentration. The removal percentage of nickel ions increases with enhancing the *SDS* concentration. Nevertheless, high *SDS* concentration is not appropriate in the process and decreases ion removal due to the micelle or hemi-micelle formation, large foam losses, higher water recovery and competitive adsorption between the nickel ion -*SDS* complexes and unreacted *SDS* ions on the bubble surface (Hoseinian et al., 2018c).

To decrease the required collector concentration, a collector with high ion adsorption capacity in a low collector concentration is required. The anionic collector of *SDS* with a negative charge includes two sections of a 12-carbon tail and a sulphate group. It has oxygen atoms in its charge group. Whereas, the anionic nano-collector of *AFGO* with a lot of negative charges contains a structure of hexagonal carbon including the groups of oxygen-based functional like the alkoxy, carbonyl hydroxyl and carboxylic acid. It has multiple nitrogen and oxygen atoms in its structure. The atoms of nitrogen and oxygen can form the bonds of dipolar and hydrogen with the nickel ions with a positive charge in the solution. In other words, the absorption between the nickel ions with a positive charge and anionic collectors with a negative charge may be more than the adsorption by Coulomb attraction. Furthermore, *AFGO* doesn't have a frothing property which leads to high process efficiency due to low water recovery during the process.

The results indicated that the *AFGO* nano-collector showed more efficiency rather than *SDS* for ion removal. The interaction mechanisms of nickel ion removal with *AFGO* and *SDS* are shown in Fig. 4.

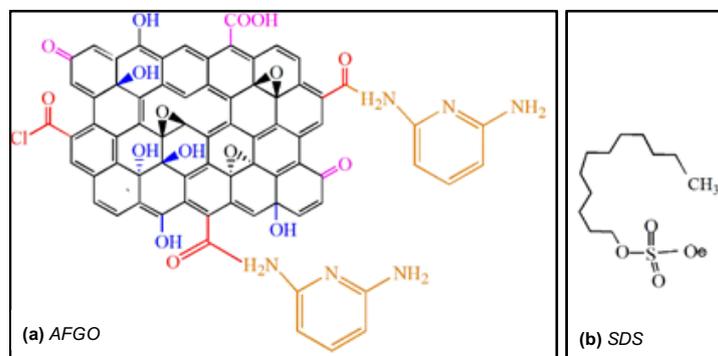


Fig. 3. The collector structures of (a) *AFGO* (b) *SDS*

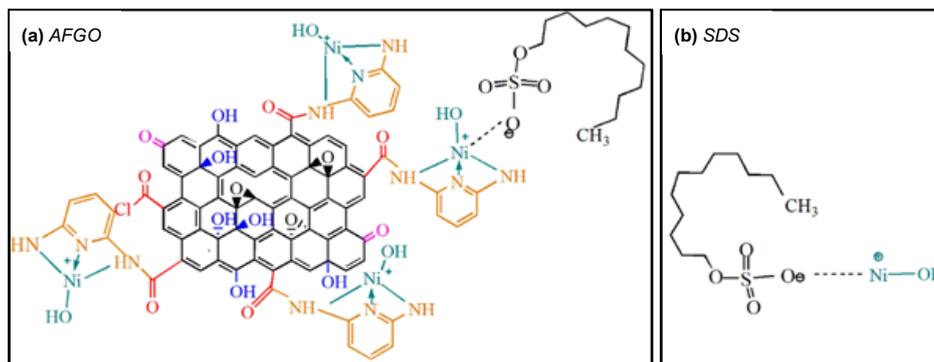


Fig. 4. The mechanism of interaction of nickel ions with (a) *AFGO* (b) *SDS*

As can be seen from Fig. 4 that the *AFGO* has appropriate multifunctional bindings for complex formations with nickel ions. At a pH 9, a coordinate bond may be formed between nickel ions and *AFGO* which increases the removal of nickel ion (Świątek-Kozłowska et al., 2002).

3.1.2. Fourier-transform infrared spectroscopy (FTIR) analysis

To evaluate the collector type effect on the nickel ion flotation, the formed sublates during the process were analyzed by *FTIR*. The *FTIR* analyses of collectors before and after the ion flotation and distinguished peaks of *FTIR* spectra are shown in Fig. 5 and Table 1, respectively. The OH absorption band in the spectra of *AFGO* after the flotation was broader and stronger than that of *SDS* spectra, denoting the absorbed percentages of OH bond in the hydrolyzed species. The OH bands in the *AFGO* after flotation shifted from 3424 cm^{-1} to 3416 cm^{-1} which can be ascribed to an appropriate interaction between the *AFGO*, species of hydrolysis nickel ions, *SDS*, and molecules of water. The presence of C-N and N-H bonds in the *AFGO* confirmed that the successfully functionalized of graphene oxides by 2, 6- diaminopyridine. The nitrogen and oxygen atoms in the *AFGO* exhibited an appropriate performance to remove nickel ions due to providing numerous active sites to adsorb nickel ions on the *AFGO* sheets.

The peaks observed at 581 cm^{-1} and 620 cm^{-1} in the spectra of the *AFGO* after the flotation, respectively, are attributed to the presence of Ni-O and Ni-OH bonds. The intensity order of these bonds in the *AFGO* spectra is stronger than that of *SDS* spectra which confirms the more efficiency of *AFGO* nano-collector for nickel ion removal in the lower collector concentration (Silverstein and Bassler, 1962; Shahane et al., 2010).

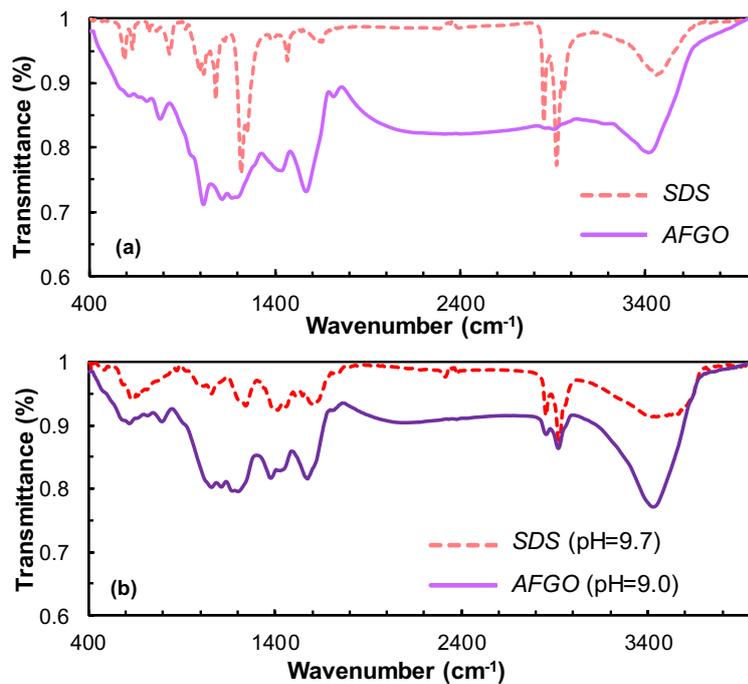


Fig. 5. *FTIR* analysis of (a) *AFGO* and *SDS* before flotation and (b) *AFGO* and *SDS* as after nickel ion flotation in the optimum conditions

3.1.3. Scanning electron microscope (SEM) analysis

The sublata morphology characterization in the optimal conditions was studied by *SEM* (Fig. 6). The *SEM* image of *AFGO* after nickel ion flotation demonstrates that the nickel ion complexes efficiently adsorbed on the *AFGO* separated thin sheets. While the *SEM* images of *SDS* after nickel ion flotation demonstrates that the sublata morphology was compressed with layers of the sheet mode.

3.2. Ion flotation efficiency

In the fine particle flotation system, the fine particles are transferred to the froth phase by two phenomena of true flotation and entrainment. The true flotation of fine particles occurs when fine parti-

Table 1. Presence of different functional groups in various cases

Bands Assignments	Peaks for SDS (cm ⁻¹)	Peaks for AFGO (cm ⁻¹)	Peaks for SDS (pH=9.7) (cm ⁻¹)	Peaks for AFGO (pH=9) (cm ⁻¹)
C=O		1718		1709
C-H	2957, 2919, 2851, 1388	2867	2853, 2924	2920 -2855
-OH	3454	3424	3428	3416
C=C	1635	1566	1609	1577
CH ₂	1465, 723	-	1466	-
S=O	1224	-	1244	1204, 1378
C-O-S	1019, 835	-	-	-
C-S	632	-	636	626
C-O	1083	1000-1200		1000-1200
C-N	-	1000-1200, 1300	-	1000-1200
N-H	-	1570, 3175, 783	-	795, 1570
Ni-O	-	-	419	581
Ni-OH	-	-	485	620

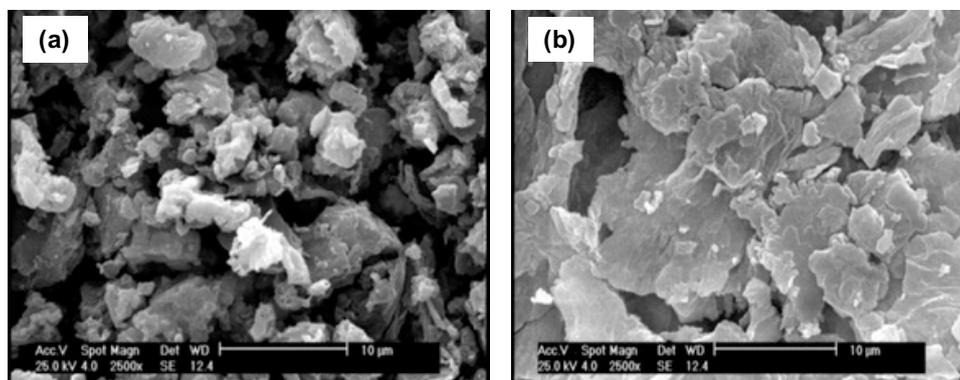


Fig. 6. SEM images of sublates formed using (a) AFGO and (b) SDS as collectors after nickel ion flotation in the optimum conditions

cles selectively attach to the rising bubble surfaces and transfers to the froth phase. The entrainment of fine particles occurs when fine particles non-selectively transfer to the froth phase by dragging the fine particles from the pulp phase into the froth phase using the water of interstitial and wakes of the rising bubbles. The efficiency of fine flotation depends on decreasing the entrainment. Water is the main constituent of feed in the flotation system which transfers to the froth phase along with rising bubbles. Entrainment is a function of water recovery during the process (George et al., 2004; Zheng et al., 2006).

Ion flotation as an efficient process extensively examined to remove ions from a solution. In this process, an insoluble complex produces resulting in the interaction between the metal ions and the collector and then removes by rising bubbles. Hoseinian et al. (2019c) showed that the insoluble complex size during nickel ion flotation was nearly lower than 5 µm (Hoseinian et al., 2019c). It can be assumed that the removal of insoluble ionic complexes in the ion flotation process is similar to the removal of fine particles in the mineral flotation. Therefore, the removal of ions in the ion flotation process can also be carried out by two phenomena of true flotation and entrainment.

The successful ion flotation is achieved not only by a high ion removal but also by the low water recovery. The complexes of ion-collector usually transfer to the froth phase predominantly by true ion flotation, but the ions in the solution can also be transferred by entrainment phenomena in the inter water between rising bubbles, along with ion complexes. Both phenomena of true ion flotation and entrainment may simultaneously carry out during the ion flotation process depending on the separation mechanism type and water recovery.

The successful ion flotation can be achieved by considering both of high ion removal and low water recovery. In this regard, the ion removal should be plot versus the water recovery in a diagram to investigate the process efficiency. The 50/50 split line in this diagram (the diagonal line) is ascribed to

no ion enrichment in the phase of froth or the solution. The points above the split line are ascribed to the ion enrichment in the froth, whereas the points below the split line are ascribed to the ion enrichment in the residual solution in the cell (Polat and Erdogan, 2007).

The effect of collector type and pH on the process efficiency is shown in Fig. 7. The result shows that the efficiency of nickel ion flotation using the *AFGO* at a pH 9 is significantly higher than that by *SDS* as a collector at a pH 9.7. The efficiency process using the *SDS* collector gradually enhanced with pH increasing from 3 to 9.7. Additionally, the efficiency process using the *AFGO* collector considerably increased with the increasing pH.

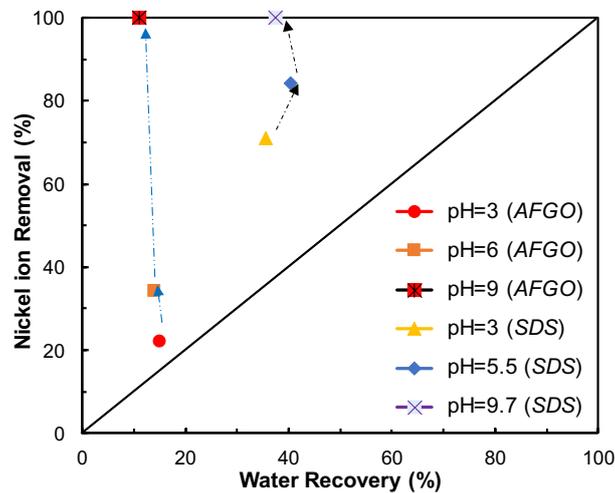


Fig. 7. Nickel removal versus water recovery as functions of collector type and pH

During the nickel ion flotation process, a part of the water is transferred along with the complexes of nickel ions and collector ions from the solution to the froth phase by the rising bubble. The amount of water transferred to the froth phase can be measured by a factor of transport (*T*) (Micheau et al., 2018). The factor of transport is explained as the ratio of the volume of collapsed foam to the volume of the initial foaming solution. It can give significant information regarding the efficiency ion flotation. The transport factors for collectors of *AFGO* and *SDS* are shown in Fig. 8. The results indicate that the transport factor for the *AFGO* is remarkably lower than that for the *SDS*. It significantly decreased with increasing pH by *AFGO*. The results indicated that the *AFGO* is favorable for ion flotation with high process efficiency.

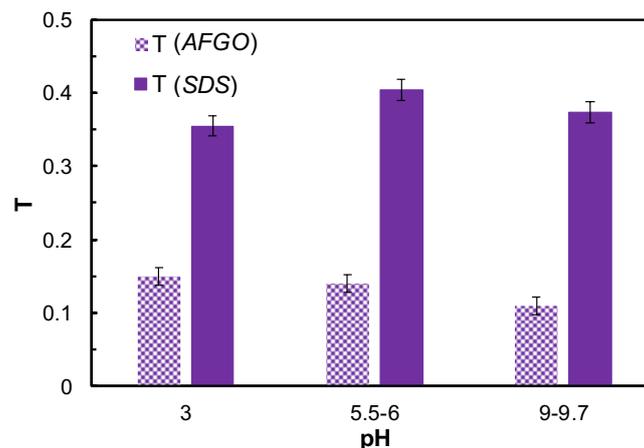


Fig. 8. The effect of collector type and pH on the transport factor

4. Conclusions

The separation efficiency of the ion flotation is strongly influenced by differences in the collector's structure properties. A set of experiments, based on initial flotation experiments, was used to investigate

how the flotation performance responds to changes in some critical parameters such as pH and collector type. The SDS and AFGO were used as collectors. These results showed that over 99.9% of nickel ions were removed with both collectors at the best conditions which are 0.1 g/dm³ of AFGO at pH 9 and 0.135 g/dm³ of SDS at pH 9.7. Based on the results, the collector structure had a major impact on water recovery. A comparison of observed results demonstrated that the AFGO had a higher separation efficiency than SDS in terms of water recovery. It was observed that it was possible to find optimum condition while using AFGO as a collector to concentrate nickel ions with over 99.9 % recovery in concentrate which holds around 11% of the initial water. Given the fact of data reproducibility, obtained results are highly encouraging. At pH 9, the nickel ions and AFGO may be formed a coordinate bond which increases nickel ion removal. The initial flotation experiments demonstrated that the water recovery was less sensitive to pH. The data showed that the water recovery was almost threefold for SDS compare to AFGO. The AFGO doesn't have a frothing property which leads to a decrease in the water recovery during the process. This was a good indication to show that the nickel ion flotation efficiency using the AFGO as a collector at a pH of 9 is significantly higher than that by SDS as a collector at a pH of 9.7.

Acknowledgments

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