

KINETIC APPROACH FOR BERYLLIUM-IRON DECONTAMINATION ION FLOTATION PROCESS

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ABSTRACT

The research aims to study the amenability of removal of iron ions from the leaching liquor solution produced from acid leaching of beryl ore concentrate from the area of Umm Sleimat in the Eastern Desert of Egypt. Ion flotation is unique technique for removal of heavy metal from wastewater. It could be applied to remove iron from liquor solution produced from acid leaching process of beryl ore concentrate. A series of chelation-flotation experiments for removal of iron were carried out using 1-10 phenanthroline as chelating agent and iron sodium lauryl sulphate (SLS) as flotation collector to find out the effect of pH, amount of chelating agent and rate of gas flow.

Applying the chelating flotation optimum condition (at pH 7 for 15 min. flotation time with 0.3 ml/min. 1% SLS solution flow rate and 150 ml/min. nitrogen gas flow rate after 4 ml 1,10-phenanthroline addition) on Umm Sleimat Beryl liquor solution showed that the removal of iron was more than 98% with only 1% beryllium removal in float fraction.

The paper presents a review of kinetic studies on ion flotation methods with first-, second models, to calculate values of flotation rate constants (k), order of flotation (n) and determination coefficients (R).

Data treatment according to the graphical method of kinetic analysis indicated a much better fit to second order kinetics for iron with correlation coefficients higher than 0.998 compared to first order kinetics with correlation coefficients of less than 0.945. As may be noted, the same data in second order result in a very good linearity as compared with the results of the first order kinetics. Variation and implication of ion flotation rate constant are discussed with respect to chemical conditions and amount of chelating agent sufficient for iron present. The obtained kinetics parameter will be further included in mass balance equation for up scaling processes.

1. INTRODUCTION

Beryllium is a rare element; its abundance in the earth's crust is about 6 ppm. However, it occurs in concentrated form in various minerals Harben (2002). The first beryllium mineral commercially exploited was beryl. It is found in mica schist, granite, pegmatite, and argillite Osborn, (1950), Fleischer and Cameron (1955). The crystals often show signs of weathering and they rarely have the ideal composition. Beryl is often obtained as a byproduct of feldspar quarrying.

The demand for beryllium increased around (1950) with the development of nuclear reactors. The nuclear properties and low density of beryllium indicate that the metal would be especially suitable for non-stationary nuclear reactors of the types used in airplanes, spacecraft, ships and submarines Beaver and Lillie (1960).

In Egypt, beryl occurrence is mainly localized in the southern part of the Eastern Desert in Zabara-Umm Debaa where it is associated with quartz, pegmatite veins and mica schists. It has also recently discovered in Sinai in Wadi Ghazala and Wadi El Seih, where it is associated with granite and pegmatite [Sadek, (1952)].

The recovery of beryllia concentrate is generally undertaken directly from its different solution (sulphate and fluoride) by precipitation of beryllium as hydroxide, which would then be converted to the oxide form. These recovery procedures are accompanied by some purification methods to remove co-dissolved impurities and interfering cations. However, refined beryllia, which is lightly pure (nuclear grade), can be recovered by the modern separation techniques; viz, solvent extraction [Abdel Messieh, (1979) and (1993)] or ion exchange [Demerdash, (2006)]. These techniques would directly be applied on the breakdown solutions or else after a primary separation of a beryllia concentrate.

Ion flotation, as a flotation subgroup, is a pre-concentration technique known since the early 1960s [Cabezon L et al., (1984)]. It is a simple and inexpensive method for effective removal of ionic species from dilute aqueous solution [Pavlovskaya G. et al., (2001); Stafilov T. et al., (2000)]. The major advantages of this method are the rapidity and high recoveries of the trace elements [Caballero M., Cela R., (1990)]. Ion flotation has a special position for removal of ions from diluted solutions at concentrations below $1.0 \cdot 10^{-4}$ M. This both method involve removal of surface inactive ions from aqueous solutions by introducing of surface active substances (collectors) and subsequent passage of gas bubble through the solution [Walkowiak W. and Ulewicz M., (1999); Lu Y. and Zhu X., (2001); Zouboulis, A. I. and Matis, K. A. (1987); Sobianowska, K. et al. (2009)]. In recent years, several macrocyclic compounds, such as non-ionizable crown ethers and ionizable lariat ethers, have been applied as new type collectors for removal and separation of several metal ions. [Walkowiak, W. et al. (1999); Koide Y. et al. (1993, 1996); Schulz J.C., and Warr G.G., (1998); Charewicz W., et al., (2001); Ulewicz, M. et al. (2003a, 2003b, 2006); Ulewicz, M. and Maciejewski, P. (2008)]. Koide, Y. et al. (1993) investigated resorcinol-type calix[4]arenes with alkyl side chains as collectors in competitive flotation of alkali metal cations and showed selective flotation of Cs^+ over Na^+ and K^+ cations from dilute aqueous solutions. On the other hand, Koide Y and co-workers, (1996) separated UO_2^{2+} cations from seawater with C-undecylcalix [4] resorcinarene phosphate collectors. Schulz, J. C. and Warr, G. G. (1998) studied alkali metal cation separations from dilute aqueous solutions by neutral macrocyclic complexing agents of a crown ether (18-crown-6) or a cryptand (cryptand 222) in combination with the anionic surfactant bis(2,2')ethylhexyl-sulfosuccinate (AOT). Schulz, J. C. and Warr, G. G. (1998) showed that addition of C222 allows achieving much better separation of alkali metal cations. Separation with AOT only was as follows: $\text{Cs}^+ \geq \text{Rb}^+ \geq \text{K}^+ > \text{Na}^+ > \text{Li}^+$. With equimolar C222 added to AOT, separation was changed as follows: $\text{K}^+, \text{Rb}^+ > \text{Cs}^+ > \text{Na}^+ > \text{Li}^+$. In other approach, Charewicz W., et al., (2001) first employed proton ionizable lariat ethers in combination with octylphenol decaethylene glycol ether, a neutral foaming agent, and reported efficient and selective flotation of Sr^{2+} over Cs^+ and Co^{2+} from very dilute aqueous solutions.

The quantification of kinetic parameters is of increasing importance in industrial flotation to shed light on the speed of the process. The kinetic of the reaction varies

widely depending on the metal and the conditioning parameters during the ion flotation process. The rate of collector adsorption can be influenced by many factors, such as the pH and collector concentration.

The first kinetic studies of ion flotation process were carried out by Rubin et al. (1966, 1968). They studied the ion flotation of copper (II) with sodium lauryl sulfate. As a result, a soluble copper (II) cation - surfactant product was floated, for which a first - order equation, can be applied:

$$dc/dt = - c_i/c_i - c_r k (c - c_r) \dots\dots\dots (1)$$

where: c_i – initial concentration of floated ions, c_r – concentration of ions in residual solution, k - kinetic rate constant.

After integration:

$$\text{Log (M-R)} = \text{log M} - 0.434 c_i / M kt \dots\dots\dots (2)$$

Where: $R=1-c/c_i$, $M= 1- c_r / c_i$ (maximal flotation removal).

In addition, the lead (II) and iron (III) cations with sodium lauryl sulfate were floated and semi - log equation was found to fit the data reasonable. The second equation for insoluble systems, i.e. for precipitate flotation, is exponential with the integration form (Rubin, 1968):

$$\text{Log (M-R)} = \text{log B} - m \text{log t} \dots\dots\dots (3)$$

where: m and B are constants. Product of m and B is the rate constant in the differential form of equation 3. The log–log plots of precipitate flotation data for copper (II) and lead (II) hydroxides with sodium lauryl sulfate; show that equation 3 fits these data quite reasonable.

Kinetics studies on the foam separation of thorium (IV) ions from nitric media at pH ranged from 2.6 to 6.5 with sodium lauryl sulphate were done by Shakir and Samy (1979). In experimental conditions, a colloidal sublute, because of interaction of ions, surfactant was formed, and a bulk aqueous solution became turbid. The rate data were found to confirm to the first – order equation 2. This indicates that thorium is being removed by a partition mechanism. Thorium at pH equal to 2.6 is being removed in two stages and the removal rate at the first stage is lower that at the second stage. According to Shakir and Samy (1979), it implies the existence of two types of thorium (IV) cations, which have different removal tendencies. At pH 6.5, where hydrous thorium oxide is precipitated, the rate data are not described by semi-log equation 2 but a reasonable fit for log - log equation 3.

In addition, Skrylev and Amonov (1973) studied kinetics of ion flotation process. They found out the first - order kinetics for flotation of cobalt (II) and nickel (II) cations with sodium lauryl sulfate. According to Sviridov et al. (1982), the flotation data for indium (III) fit reasonably the following kinetic equation:

$$\text{Log}(100-x) = 2 - k/2.3 t \dots\dots\dots (4)$$

where: x is the sublute concentration.

Also first order kinetics was confirmed for flotation of thorium (IV) ions (Skrylev, et al. 1993, 1996), and for flotation of cobalt (II) ions (Skrylev, et al. 1988).

Skrylev et al. (1981) found that kinetics of uranium (VI) carbonates flotation using dioctylamine as a surfactant can be described by a second – order equation:

$$1/c - 1/c_i = kt \dots\dots\dots (5)$$

It was noticed that floated sublutes were in the colloidal form. The second - order equation flotation process was also reported for the flotation of La (III) cations with fatty acids (Skrylev, et al. 1987). It was found that depends on applied surfactants the kinetics is first or second order. Another confirmation of second order kinetics is to float of copper (II) tartrates by cetylpyridinium chloride are presented (Sviridov, 1986).

Goldman (1979) as well as Skrylev and Amonov (1973) have found the kinetic zero-order equation for flotation data of homogenous systems:

$$c_i - c = - kt \dots\dots\dots (6)$$

2. EXPERIMENTAL

2.1. Reagents

All chemicals used in this study were of analytical-reagent grads. All aqueous solutions were prepared in double distilled water. Take 1000 ml of sample into a beaker, add 2ml of (1:1) sulphuric acid, 2-5 ml of 10% hydroxylamine sulphate (reducing agent) and 1-4 ml of 0.01 M 1-10 phenanthroline (chelating agent) solution, and 1ml of 1% sodium lauryl sulphate (SLS). Collect the foam coming out of the separator in a 50 ml beaker, which contained 2 ml of 1-propanol as a foam breaker, pH of the solutions during experiments, was adjusted with 6 M ammonium.

2.2. Apparatus

Ion Flotation Machine (a cylindrical tub of 13 cm length and 4.5 cm inner diameter, provided with a stopcock at the bottom and a stopper at the top).The cell contains micro porous sintered glass. Micro air bubbles produced during passing the air through it. These small bubbles are proper and suitable to be used in ion flotation to float the ion from the solution. It contains two side opening tube, one in the bottom for passing air or nitrogen and the second in top in the side for collecting the float fraction. The pH-values of the solutions were measured using WTW inolab level 1.

2.3. Procedure

According to the recommended procedure for low iron concentration separation by ion floatation after chelation with 1-10- phenanthroline (chelating agent) ,1000 ml of sample was taken into a beaker with addition 2 ml of (1+1) sulphuric acid, 2-5 ml of 10% hydroxylamine sulphate (reducing agent) and 1-4 ml of 0.01 M chelating agent

solution. Adjust the PH 2-8 with 6M ammonium and stir the solution for 10 min. Pour the solution into the foam separation tube to which nitrogen gas is being fed at a low flow rate. After adjusting the nitrogen-gas flow rate to 150 ml/min, add 1 ml of 1% sodium lauryl sulphate (SLS) aqueous solution from the top of the separation. At the same time, start feeding the 1% SLS solution through the lower side tube at the flow rate starting from 0.1 ml/min. to 0.3 ml/min. collects the foam coming out of the separator in a 50 ml beaker, which contained 2 ml of 1-propanol as a foam breaker. Continue the separation for 20 min. at and measure the metal contents in the solutions obtained. All experiments were carried out at room temperature, (20 + 1°C).

The floatability (F) of Fe (III) was determined from the relationship:

$$F = (C_i - C_f)/C_i \times 100\%$$

where, C_i and C_f denote the initial and final concentrations of Fe (III) ion in mother liquor.

3. RESULTS AND DISCUSSION

A series of chelation-floatation experiments were carried out to find out the effect of pH, amount of chelating agent sufficient for iron present, rate of floating agent flow time of floatation and rate of gas flow (Samia 2012).

3.1. Effect of pH

The hydrogen ion concentration has a significant effect on the formation of chelate and on the floatation process. The experiment was carried out at 15 min. flotation time the results were 0.70%, 0.73% and 0.74% (recovery percent) at pH 5, 6 and 7, respectively. Accordingly, floatability of iron increased markedly in the pH range 5-7 especially in the higher ion concentration experiments (figure 1).

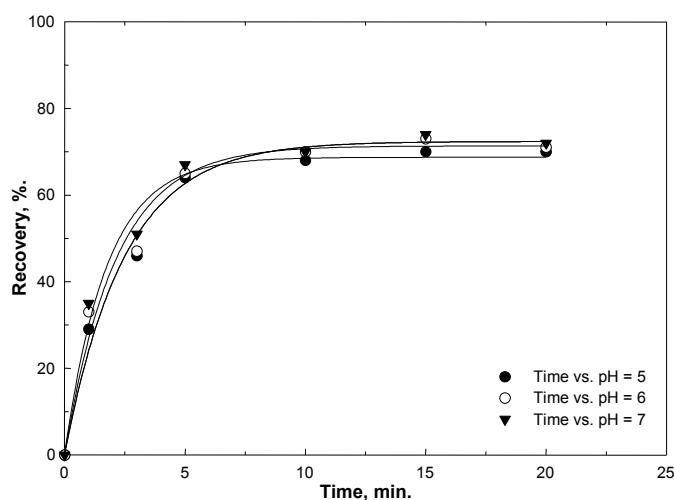


Fig. 1: Floatability % of Fe at different rang of pH using SLS flow rate of 0.2 ml/min. and nitrogen flow rate 150 ml/min. at 3ml chelating agent and 0.005% Fe.

3.2. Reducing Agent, Chelating Agent and Ion Concentration Effect

All previous work on iron apply 5 ml of 10% hydroxylamine sulphate as a reducing agent while in this case, 2 ml of reducing agent proved to be enough to reduce Fe (III) present in solution. Further, 3 ml of 1,10-phenanthroline solution was shown to be sufficient for qualitative results specially with higher ion concentration (0.005%) the effect of the amount of chelating agent, added (1,2,3 and 4 ml of reagent) at 0.005% ion concentration as shown in figure 2.

The figure prevailed that floatability of the ion increased with chelating agent amount increase under all different conditions until nearly constant value after 3ml of the reagent.

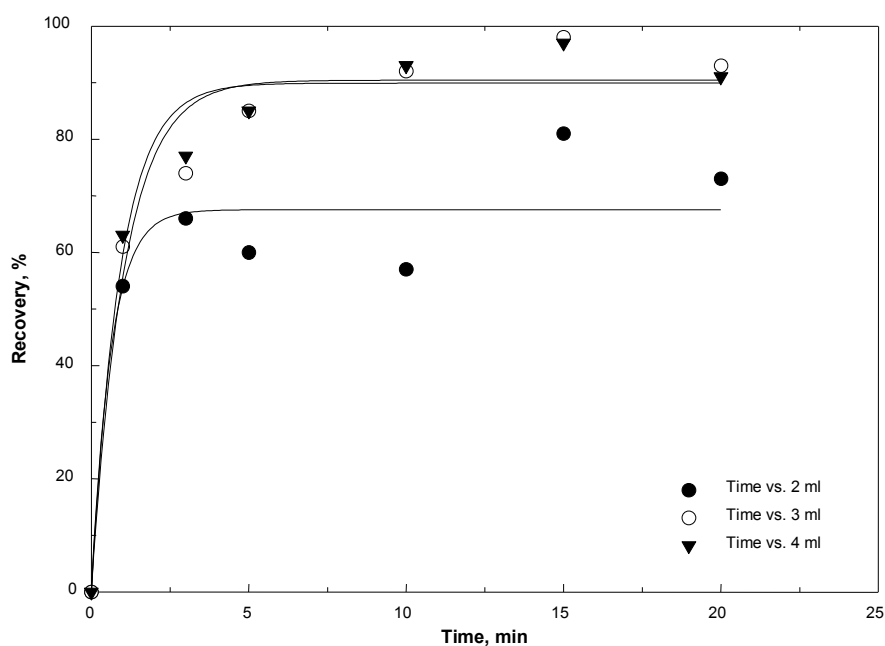


Fig. 2: Floatability % of Fe with SLS, flow rate of 0.2 ml/min. and nitrogen flow rate 150 ml/min., different chelating agent at pH 7 and 0.005% Fe.

The figure clears that recovery percents of ion at 15 min. flotation time the results were 0.81%, 0.98% and 0.97% at chelating agent 2, 3 and 4, respectively.

3.3. Effect of – SLS and Nitrogen Gas Flow Rate

It was clearly that, 0.2 ml / min. 1% SLS flow rate during 15 min. with 150 ml/min. nitrogen gas flow were the most promising rates of solution and gas flow, respectively. Faster rates may collapse and disturb the floatation system and did not give higher promising floatability results as shown in figure 3.

For the addition of 1% SLS solution, it was necessary to add both from the top at the beginning and continuously from the bottom. This is because of several large scums that may be originated from $[\text{Fe}(\text{phen})]^{+2}(\text{Ls})^{-}$ remains on the surface of the separator and prevented the quantitative floatation in case of addition from the top during all the experiment while N_2 gas for inert atmosphere to prevent Fe oxidation.

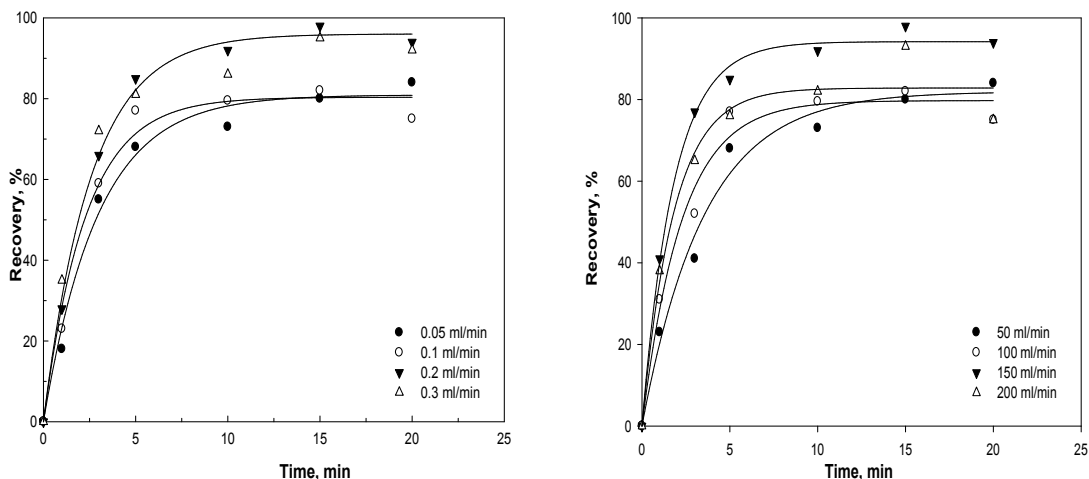


Fig. 3: Flotation recovery of iron as a function of time at different SLS and Nitrogen gas flow rate.

3.4. Flotation Recovery as a Function of Time

Flotation recoveries of iron ion obtained as a function of time are shown in figure 4. The maximum and quantitative recoveries were attained after 15 min. which chosen for separation time by floatation. From figure 4, we can confirm that the floatability of the ion increased with the time of floatation till certain limit under all variable parameters of pH, rate of SLS flow, chelate addition, ion concentration and gas flow.

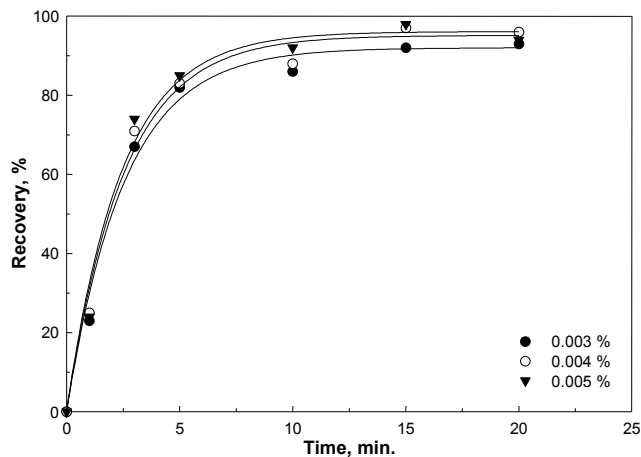


Fig. 4: Flotation recovery of iron as a function of time at different ion concentration.

3.5. First Order Kinetics

The flotation data plotted in figure 4 have been examined from a kinetics point of view. Flotation kinetics can be studied by analogy to chemical kinetics. The rate equation in its simple form may be written as:

$$dc/dt = -k_n C^n \dots\dots\dots (7)$$

in which C is the concentration of particles in the flotation cell, t is time, k is the flotation rate constant and n is an integer determining the order of the rate equation. The rate constant is dependent on a number of variables, others to operating variables such as dosage of reagents. Equation 7, when integrated for the first-order flotation kinetics (Arbiter and Harris, 1962) respectively leads to:

$$R = R_{\infty} (1 - e^{-k_1 t}) \dots\dots\dots (8)$$

where R is the cumulative recovery after time t and R_{∞} is the maximum recovery after prolonged flotation time and k_1 is the first order rate constant, this model has been considered as the first case. Rearranging equation 8 results in the following linear form:

$$\ln [(R_{\infty} - R)/R_{\infty}] = - k_1 t \dots\dots\dots (9)$$

If the first order model is valid, a plot of the left hand side of equation 9 as a function of time should result in a straight line. The experimental data are accordingly plotted in figure 5. As may be noted, results depend on the maximum recovery values adopted. The ion concentration, 0.003%, 0.004% and 0.005% denote the calculated results based on the R_{∞} values adopted in table 1. Correlation coefficients from linear regression show three concentration, one lower than the other. The lower was obtained by using higher R_{∞} values by extrapolation to larger values based on second order kinetics to be discussed below. Regardless of what the values of R_{∞} are, a deviation from linearity is significant. In general, the flotation rate constant decreases with increasing flotation time.

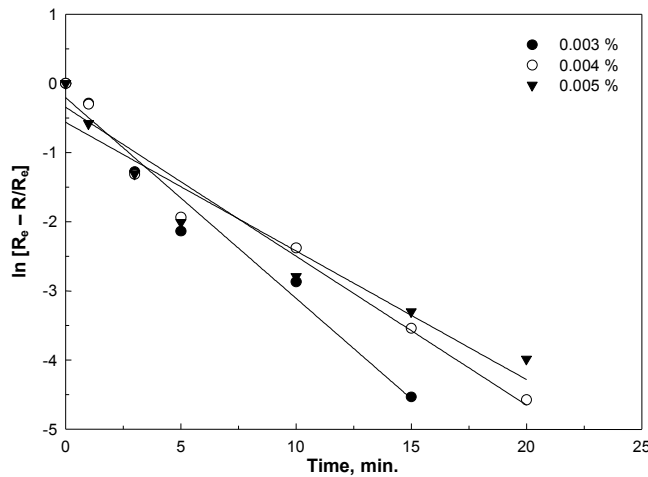


Fig. 5: First-order reaction curve for the different ion concentration.

Table (1): Reaction rate constant and correlation coefficients for the different ion concentration.

Fe Conc. %	K	R ²
0.003	0.2903	0.9857
0.004	0.2154	0.9823
0.005	0.1860	0.9651

The influence of SLS flow rate and nitrogen-gas flow rate on iron is shown through figures 6 and 7. The ion floatability results were shifted to their corresponding higher values under the same conditions. The highest value is at SLS flow rate 0.2 ml/min. and 150 ml/min N₂ gas flow rate.

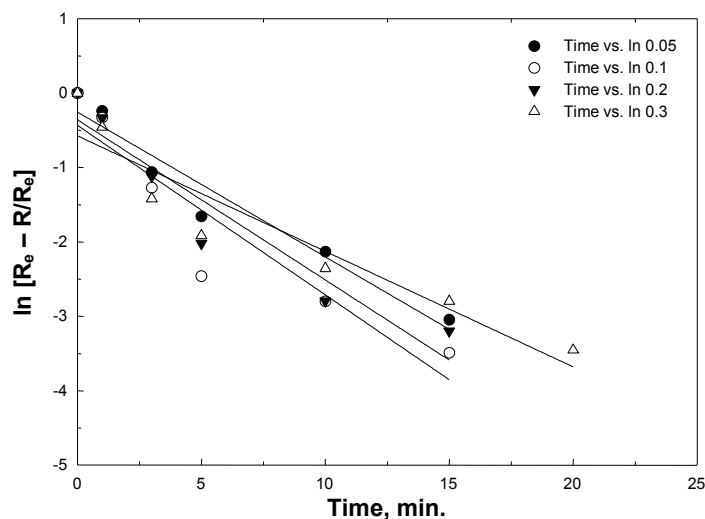


Fig. 6: First-order reaction curve for the different SLS flow rate.

Table (2): Reaction rate constant and correlation coefficients for the different SLS flow rate.

SLS, ml/min	K	R ²
0.05	0.1948	0.9724
0.1	0.2279	0.9351
0.2	0.2155	0.9538
0.3	0.1551	0.9456

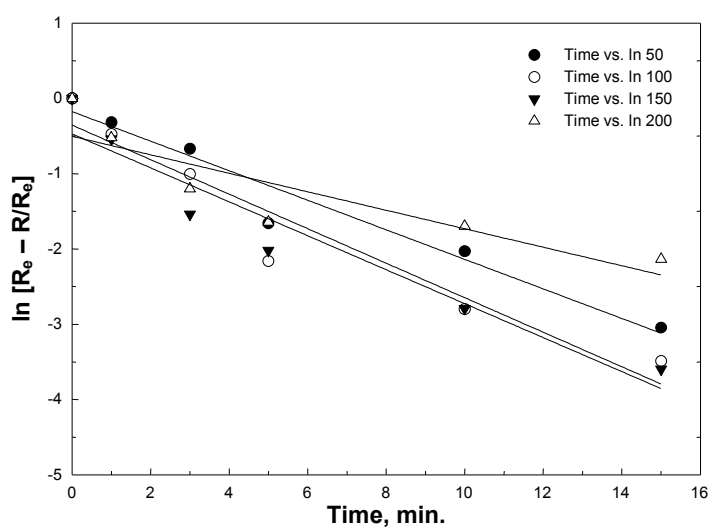


Fig. 7: First-order reaction curve for the different N₂ flow rate.

Table (3): Reaction rate constant and correlation coefficients for the different N₂ flow rate.

N ₂ , ml/min	K	R ²
50	0.1963	0.9767
100	0.2292	0.9630
150	0.2253	0.9640
200	0.1228	0.8857

The influence of chelating agent on iron is shown in figure 8. The ion floatability results were shifted to their corresponding higher values under the same conditions. The highest value is at chelating agent 3 ml.

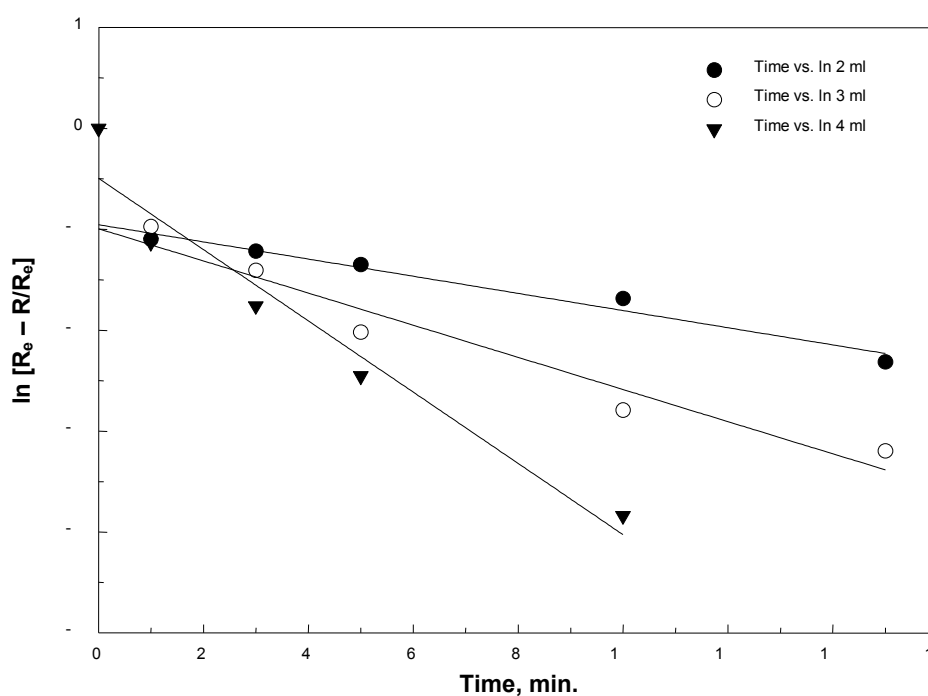


Fig. 8: First-order reaction curve for the different chelating agent.

Table (4): Reaction rate constant and correlation coefficients for the different chelating Agent.

Chelating agent, ml	K	R ²
2	0.0851	0.9865
3	0.1594	0.9755
4	0.3531	0.9731

Further experiments were carried out at different pH: 5, 6 and 7 with solution of 1,10-phenanthroline. The influence of pH on the chelating agent adsorption on iron is shown in figure 9. Adsorption increases with increasing pH until it reached the highest value at pH = 7, after that the adsorption began to decline.

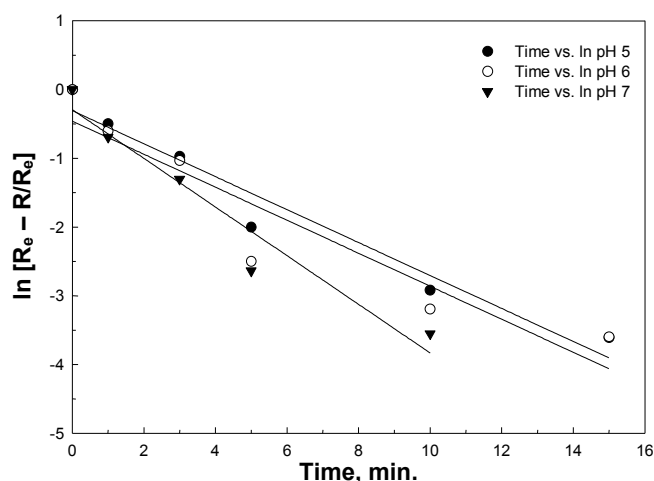


Fig. 9: First-order reaction curve for the different pH.

Table (5): Reaction rate constant and correlation coefficients for the different pH.

pH	K	R ²
5	0.2396	0.9761
6	0.2401	0.9400
7	0.3539	0.9697

3.6. Second Order Kinetics

Since treatment of data according to the first order kinetics has resulted in a significant degree of scatter and apparent inconsistencies, applicability of the data for second order flotation kinetics needed to be tested. Equation 7, when integrated for the second order flotation kinetics respectively yields the following (Arbiter and Harris, 1962):

$$R = \frac{R_{\infty}^2 k_2 t}{1 + R_{\infty} k_2 t} \dots\dots\dots (10)$$

where k_2 is the second order rate constant. Re-arranging equation 10 one can get:

$$\frac{t}{R} = \frac{t}{R_{\infty}} + \frac{1}{R_{\infty}^2 k_2} \dots\dots\dots (11)$$

If the second order equation is valid, a plot of “t/R, flotation time/recovery” against time t (cumulatively for both cases) should result in a straight line. The results plotted accordingly are shown in figure 10. As may be noted, the same data now result in a very good linearity as compared with the results of the first order kinetics (figure 5). The R_{∞} values obtained from slopes and second order rate constants, k_2 obtained from the intercept values for each case are tabulated in table 6. The correlation coefficient (R) obtained for each case is greater than 0.998 compared to 0.945 or less for the first order case (table 3). Due to this excellent data fit, the alternate ultimate recoveries for the first order kinetics (i.e., R_{∞} in table 6 were the R_{∞} values obtained from the second order model (table 7) demonstrating sensitivity of k_1 and R values to variation in the R_{∞}

values. Model fit of the iron data to second order kinetics is also much better than to that of first order kinetics.

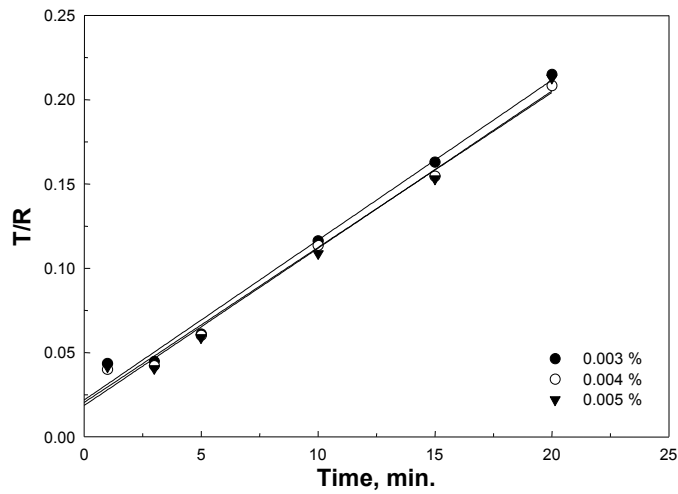


Fig. 10: Experimental data plotted according to second order flotation kinetics for the different ion concentration.

Table (6): Reaction rate constant and correlation coefficients for the different ion concentration.

Fe Conc. %	R	R _{max.}	K
0.003	0.9948	105.26	0.0041
0.004	0.9955	108.69	0.0041
0.005	0.9924	107.52	0.0047

The influence of SLS flow rate and nitrogen-gas flow rate on iron is shown through figures 11 and 12.

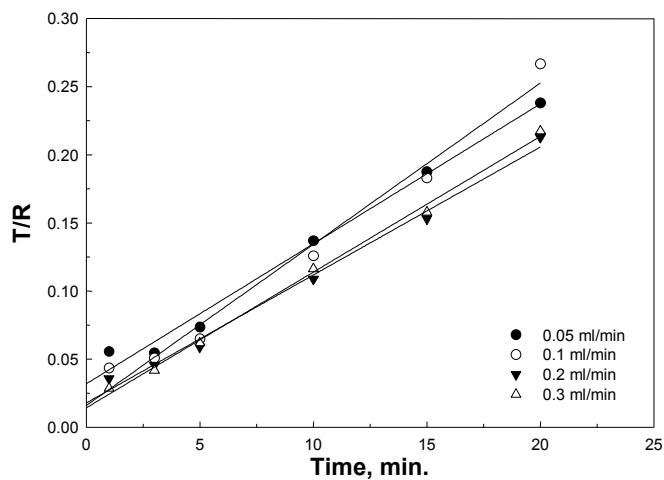


Fig. 11: Experimental data plotted according to second order flotation kinetics for the different SLS flow rate.

Table (7): Reaction rate constant and correlation coefficients for the different SLS flow rate.

SLS, ml/min	R	R _{max.}	K
0.05	0.9940	97.09	0.0033
0.1	0.9906	84.75	0.0088
0.2	0.9959	106.38	0.0049
0.3	0.9984	100	0.0070

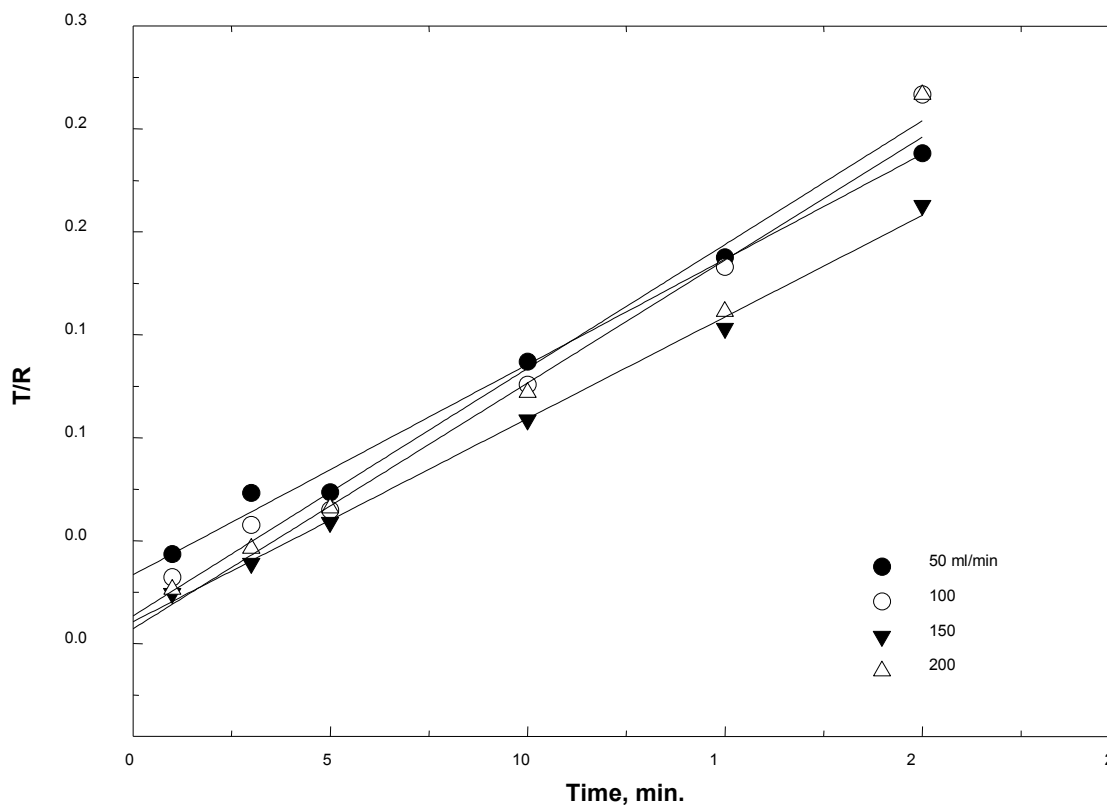


Fig. 12: Experimental data plotted according to second order flotation kinetics for the different N₂ flow rate.

Table (8): Reaction rate constant and correlation coefficients for the different N₂ flow rate.

N ₂ , ml/min	R	R _{max.}	K
50	0.9964	98.04	0.0031
100	0.9934	83.33	0.0106
150	0.9986	101.01	0.0092
200	0.9858	101.01	0.0092

The influence of chelating agent on iron is shown in figure 13.

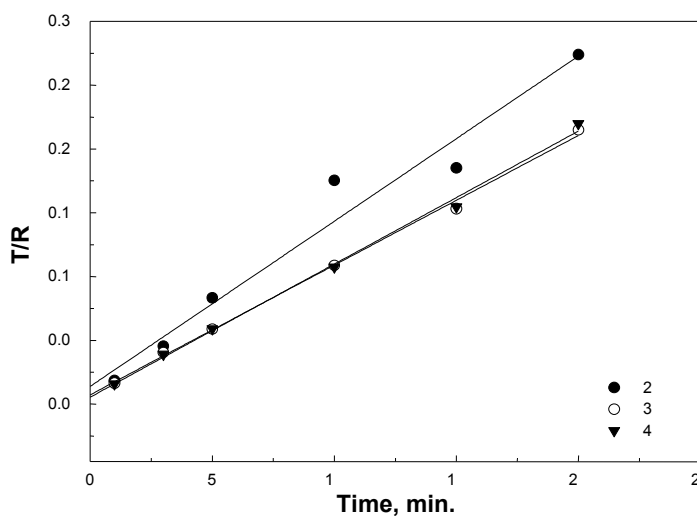


Fig. 13: Experimental data plotted according to second order flotation kinetics for the different chelating agent.

Table (9): Reaction rate constant and correlation coefficients for the different chelating agent.

Chelating agent, ml	R	R _{max.}	K
2	0.9820	77.52	0.0121
3	0.9987	98.04	0.0143
4	0.9984	96.15	0.0200

Further experiments were carried out at different pH: 5, 6 and 7 with solution of 1,10-phenanthroline. The influence of pH on the chelating agent adsorption on iron is shown in figure 14. Adsorption increases with increasing pH until it reached the highest value at pH = 7, after that the adsorption began to decline.

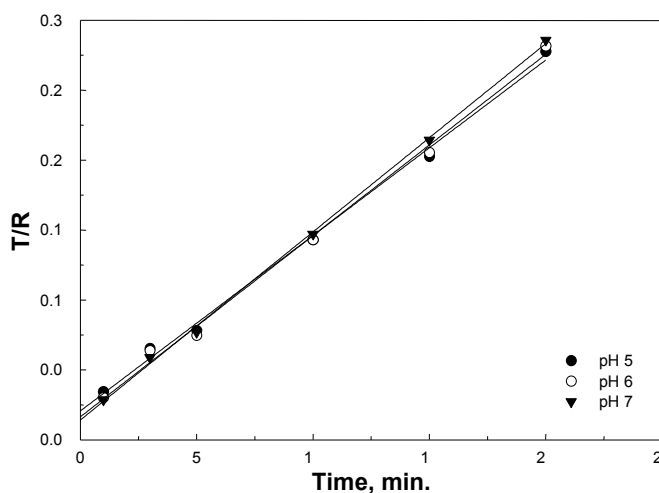


Fig. 14: Experimental data plotted according to second order flotation kinetics for the different pH.

Table (10): Reaction rate constant and correlation coefficients for the different pH.

pH	R	R _{max.}	K
5	0.9981	80	0.0075
6	0.9979	76.92	0.0102
7	0.9995	74.07	0.0129

4. CONCLUSION

In a comparative study on ion flotation kinetics of removal of iron, involving two models, first order and second order, second order model was demonstrated to represent the data better than the first order model. In the literature, very few studies report second order kinetics for flotation. The current work demonstrates one for recovery of beryllium from ion flotation of iron under experimental conditions detailed out, which indicated correlation coefficients of 0.998 or higher because the second order rate constants result in a very good linearity as compared with the results of the first order kinetics.

5. REFERENCES

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المنهج الحركي لتطهير محلول البريليوم من ملوث الحديد باستخدام عملية التعويم الأيوني

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ص.ب.: ٥٣٠ المعادي - هيئة المواد النووية

في دراسة مقارنة عن حركية التعويم الأيوني لإزالة الحديد، التي تتطوي على نموذجين، الترتيب الأول والثاني، تم إظهار نموذج الترتيب الثاني لتمثيل البيانات بشكل أفضل من نموذج النظام الأول. في الدراسات السابقة هناك عدد قليل جداً من الدراسات التي تبلغ حركية الترتيب الثاني للتعويم الأيوني. يوضح العمل الحالي استرداد البريليوم من التعويم الأيوني لأيون الحديد في ظروف تجريبية مفصلة، والتي أشارت معامل الارتباط أن يكون ٠,٩٩٨، أو أعلى لأن معدل الثوابت للترتيب الثاني يؤدي إلى خطى جيدة جداً بالمقارنة مع نتائج حركية النظام الأول.