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KINETICS STUDIES OF IONS FLOTATION

We present a review of kinetic studies on flotation methods with zero-, first-, second- and log-log models. We propose a general kinetic equation to evaluate literature data as well as our own results. To calculate values of rate constants (k) and order of flotation (n) and determination coefficients (R^2) Statistica Program was applied. An original method of floated ions concentration detection vs. time during each experiment by means of radioactive tracers has been proposed. We have verified chosen kinetics literature data showing values of k , n , and R^2 . Some unexpected kinetics calculations were received for data published by Skrylev and Legicenko (1981). We have found, on the example of zinc(II) ions, that where no sublate was precipitated in the aqueous phase, the order of flotation was close to one with good data fitting. In the presence of insoluble sublate in the aqueous solutions a characteristic inflection appears on flotation curves. Much better fitting is reached after dividing those curves into two parts; for the first stage we observed the flotation order to be close zero, and for the second stage the order of flotation was close one.

INTRODUCTION

There are several separation techniques employing adsorption on gas bubbles (Karger et al., 1967). These methods are divided into two categories, i.e. foam separation and nonfoaming adsorptive bubble separation techniques. The foam separation techniques can be subdivided into foam fractionation and flotation. Flotation methods include: ore flotation, macroflotation, colloidal flotation, ion flotation, molecular flotation, and precipitate flotation.

Ion flotation involves the removal of surface inactive ions from dilute aqueous solutions by the introduction of a surfactant and the subsequent passage of bubbles through the solution (Sebba, 1962). As a result of the process, a solid known as the sublate appears on the surface of gas/aqueous solution. Higher concentrations of floated ions and the surfactant may lead to precipitation of sublate in the bulk aqueous solution. If the species form a soluble complex or an ion pair with opposite charged surfactant ion, the process is called a foam fractionation. The foam fractionation is often confused with the ion flotation and sometimes these two processes occur simultaneously (Walkowiak, 1992).

Precipitate flotation is a froth flotation in which a precipitate is removed but the precipitating agent is other than the surfactant. Solvent sublation is a nonfoaming adsorptive bubble separation process where the enriched material adsorbs on a gas bubble surface and is then collected in an organic solvent.

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There are few reviews of foam fractionations, ion flotations, precipitate flotations and solvent sublations (Lemlich, 1972; Golman, 1982; Wilson and Clarke, 1983; Grieves, 1990).

We now present a review of kinetic studies on ion flotation (and/or foam fractionation), precipitate flotation, solvent sublation, and ore flotation. We propose a general kinetic equation to evaluate literature data as well as our own results.

REVIEW OF KINETIC STUDIES OF FLOTATION METHODS

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:

$$\frac{dc}{dt} = -\frac{c_i}{c_i - c_r} k(c - c_r) \quad (1)$$

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

After integration:

$$\log(M - R) = \log M - \frac{0.434c_i}{M} kt \quad (2)$$

where: $R = 1 - \frac{c}{c_i}$, $M = 1 - \frac{c_r}{c_i}$ (maximal flotation removal).

Also 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):

$$\log(M - R) = \log B - m \log t \quad (3)$$

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 sublimate, as a result of interaction of ions and 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 than 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).

Also 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:

$$\log(100 - x) = 2 - \frac{k}{2.3} t \quad (4)$$

where: x is the sublimate 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).

The mathematical model for the solvent sublation process was proposed by Stachurski (1970, 1974). This stochastic model takes into consideration both adsorption and desorption of ions on surface of gas bubbles passing through the aqueous solution to the organic phase. According to this model the mean value of random variable E {n(t)} is:

$$E \{n(t)\} = N (1 - e^{-\alpha t}) \quad (5)$$

where: N - the number of sublimate molecules in solution at time t = 0, n(t) - the number of sublimate molecules floated at time t, α - constant value.

It indicates that the first - order equation is describing the solvent sublation process.

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:

$$\frac{1}{c} - \frac{1}{c_i} = kt \quad (6)$$

It was noticed that floated sublimes 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 in paper (Sviridov, 1986), in which results for flotation of copper(II) tartrates by cetylpyridinium chloride are presented.

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

$$c_i - c = -kt \quad (7)$$

Zivanov et al. (1976) has proposed for precipitate flotation the modified form of equation (3):

$$\log(1 - R) = Z - k_p \log t \quad (8)$$

where: Z and k_p are constants.

The kinetic data for precipitate flotation of copper(II) hydroxide by dodecylbenzene sulfonate were fitting the equation (8) reasonable (Golman, 1979). Preliminary studies on ion flotation kinetics were done in our research group a few years ago (Walkowiak et al., 1990; Walkowiak, 1992).

The first example of mineral processing modelling is in Zuniga paper (1935), who found out that his experimental data could be fitted by an equation of the form:

$$M = M_i(1 - e^{-kt}) \quad (9)$$

where: M is the amount of mineral floated, and M_i is the initial amount of mineral in the cell.

Jowett and Safvi (1960) applied the equation (9) as a basis to assume the flotation rate is govern by first order kinetics. In investigations of Jowett and Safvi (1960); Horst (1958); Bennett and Chapman (1960) the flotation rate is described by an equation of the form:

$$\frac{dM}{dt} = k(M_i - M)^n \quad n > 1 \quad (10)$$

Higher order rates corresponding to $n = 2$ (Jowett and Safvi, 1960; Horst, 1958), and $n = 1.55$ to 2.8 (Matveenko, 1957) were found. Ek (1992) presents a review of mineral flotation kinetics including theoretical approaches and kinetic models. He has shown an example of flotation calculation for copper flotation in a pneumatic cell processing a chalcopyrite ore. These results were studied by a computer program testing out the 9 models, and equation (9) was found to fit the data better than the other ones.

CALCULATIONS AND EXPERIMENTAL

The review presented shows that the kinetics of ion flotation methods is not so far clearly established. In addition, the fitting of experimental data to adequate kinetic equation is shown only in the form of plots. But no statistical measure of flotation data fitting is presented. Our approach to the flotation kinetics is to apply a general equation describing the dependence of floated ions concentration vs. time. We propose the following equation to calculate rate constant and order of flotation:

$$\frac{dc}{dt} = -\frac{c_i}{c_i - c_r} k(c - c_r)^n \quad (11)$$

After integration two equations can be found to calculate values of k and n :

$$k = -\frac{c_i - c_r}{c_i t} \ln \frac{c - c_r}{c_i - c_r} \quad \text{for } n = 1 \quad (12)$$

$$k = \frac{c_i - c_r}{c_i(n-1)t} \left[\frac{1}{(c - c_r)^{n-1}} - \frac{1}{(c_i - c_r)^{n-1}} \right] \quad \text{for } n \neq 1 \quad (13)$$

To calculate values of n and k Statistica Program (Version 5.0) was applied. The procedure „Multiple Regression”, which allows to analyse the relationship between dependent variable, i.e. c , and two independent variables, i.e. n and k , was used. The program also allows to find the determination coefficient (R^2), which is from 0 to 1.0000 and can be treated as a measure of data fitting to equations (12) or (13).

In all reviewed papers researchers were sampling aqueous solutions getting from a few to dozen or so points on a plot of concentration of floated ions vs. time. In our research group an original method of floated ions concentration detection vs. time during each experiment by means of radioactive tracers and gamma radiation spectrometry has been developed. This procedure first time described by Charewicz and Niemiec (1969) was recently improved. The apparatus scheme is presented in Fig. 1.

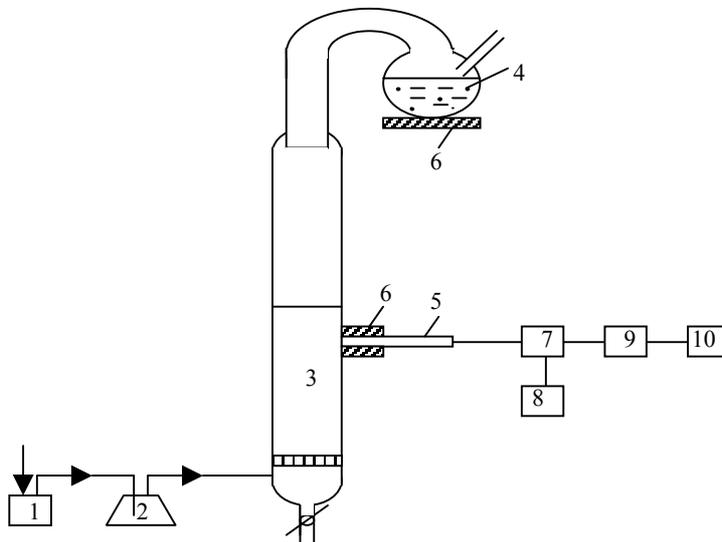


Fig. 1. Apparatus for ion flotation process: 1 - gas flow and pressure meter, 2 - humidifier, 3 - flotation column, 4 - foam receiver, 5 - spectrometric scintillation probe, 6 - lead screen, 7 - one channel gamma spectrometer, 8 - recorder, 9 - analog/digital converter, 10 - computer.

Rys. 1. Aparatura do procesu flotacji jonów.

A single channel, gamma radiation spectrometer with a spectrometric scintillation probe as a detector of radiation intensity of specific activity was used. The specific

activity of aqueous solution was measured continuously by a recorder to obtain the flotation curve. On the other hand, analog impulses were converted into digital signals and collected every 0.5-minute on a file of PC 486 computer. Gamma radioactive isotopes, were used as analytical tracers and their characteristic is shown in table 1. The used isotopes were of sufficiently high-energy (Zn-65, and Pt-193) or carrier free (Co-58) to neglect the effect of carrier concentration.

Table 1. Characteristic of applied radioactive isotopes
Tabela 1. Charakterystyka użytych izotopów promieniotwórczych

Isotope	Half life (days)	Energy of peak (fJ)	Specific energy (GBq/g)
Co - 58	72	129.6	Carrier free
Zn - 65	245	179.4	9.2
Pt - 193	4.3	10.7	30.0

RESULTS AND DISCUSSION

Kinetic curves for ion and precipitate flotation obtained by Rubin (1968), Skrylev et al. (1993), Shakir and Samy (1979), and Zivanov et al. (1976) are presented in Figs. 2 - 6. On plots are shown experimental data and values of k and n obtained from equations (12) or (13) (right side of the plot). In *italic* the values of k , n and R^2 for assumed kinetic orders (i.e. 0.000, 1.000, or 2.000) are presented.

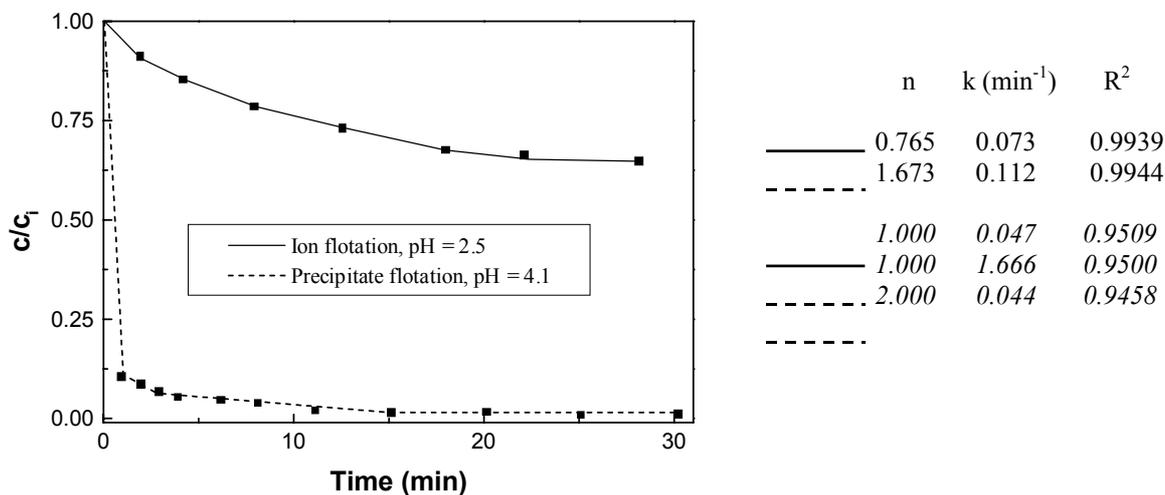


Fig. 2. Kinetic curves for the ion flotation of $2.0 \cdot 10^{-4}$ M iron(III) with sodium lauryl sulfate (Rubin, 1968), and results of kinetic calculations

Rys.2. Krzywe kinetyczne flotacji jonów żelaza(III) sulfonianem dodecylo sodowym oraz wyniki obliczeń kinetycznych

As can be seen from Fig.2 the experimental data for iron(III) flotation at pH = 2.5 fit the equation (12) for $n = 0.765$ with high determination coefficient, i.e. 0.9939. It means that order of ion flotation process is different from 1.000. Assuming that $n = 1.000$ the fitting of 7 experimental points to the equation (11) is

rather poor ($R^2 = 0.9509$). On the other hand, for precipitate flotation data (pH = 4.1) order of this process was found to be 1.673 with $R^2 = 0.9944$.

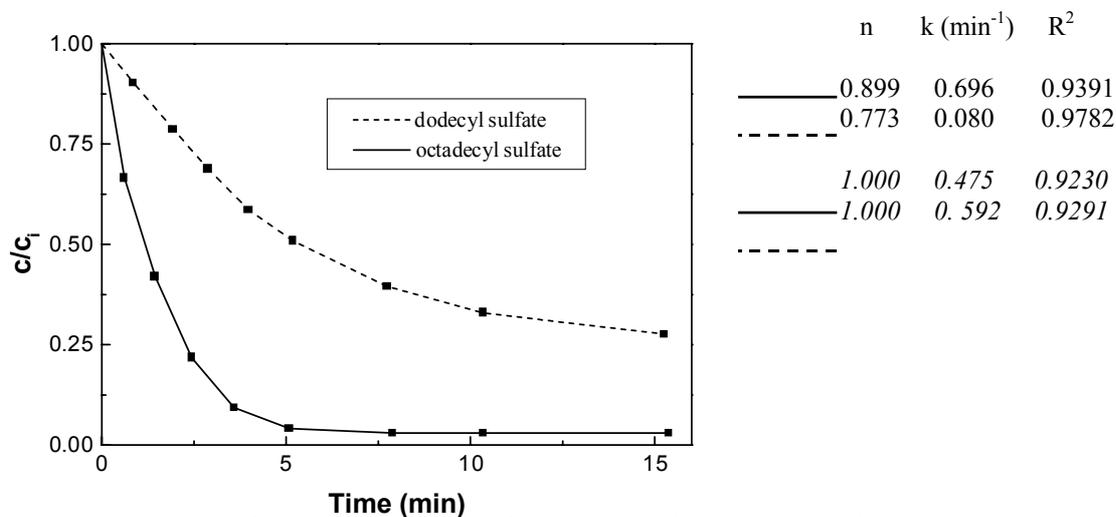


Fig. 3. Kinetic curves for the thorium(IV) ion flotation with alkylsulfates (Skrylev et al., 1993), and results of the kinetic calculations

Rys. 3. Krzywe kinetyczne flotacji jonów toru(IV) za pomocą alkilosulfonianów oraz wyniki obliczeń kinetycznych

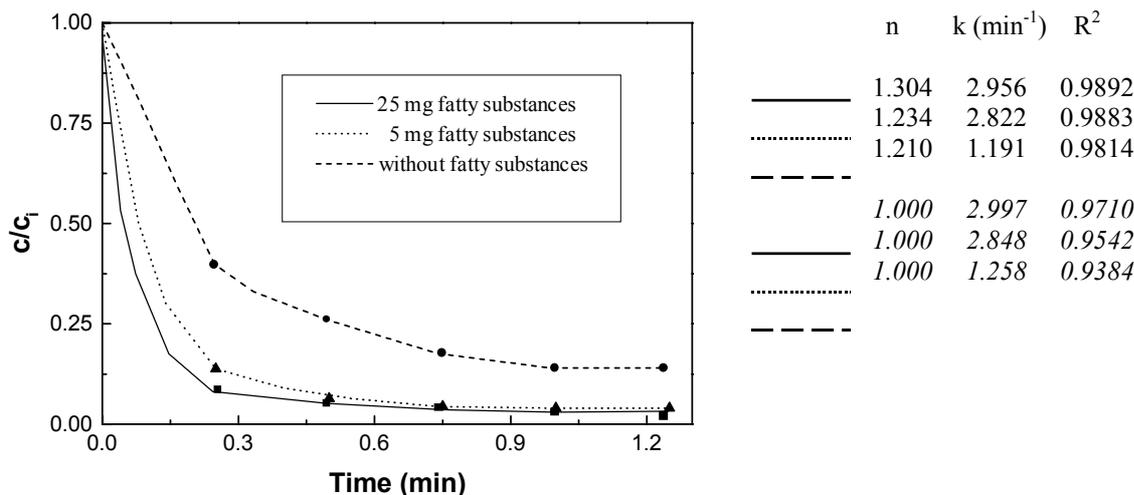


Fig.4. Kinetic curves for the precipitate flotation of $\text{Cu}(\text{OH})_2$ (Zivanov et al., 1976), and results of kinetic calculations

Rys.4. Krzywe kinetyczne flotacji osadów $\text{Cu}(\text{OH})_2$ oraz wyniki obliczeń kinetycznych

Also according to Skrylev et al. (1993) the order of ion flotation process is one; their experimental data are shown in Fig. 3. For both surfactants used the calculated order of flotation process was found to be different from 1.000, i.e. 0.899 for tetradecyl sulfate and 0.773 for octadecyl sulfate. But determination coefficients were not so good, i.e. 0.9391, and 0.9782, respectively. Assuming that kinetic order is equal to 1.000 we received not so good data fitting since determination coefficients dropped to 0.9230, and 0.9291, respectively. In Fig. 4 are presented the kinetic experimental data of Zivanow et al. (1976), who suggested the log – log equation (8) as the best fitting of copper(II) hydroxide

flotation data. We obtained the order of flotation for kinetic curves to be equal from 1.304 to 1.210 with good data fitting.

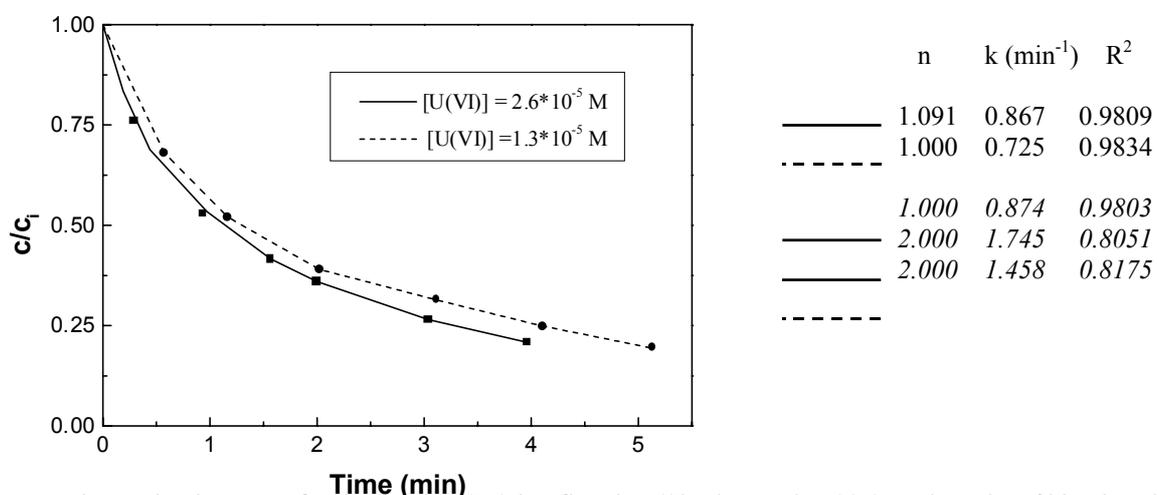


Fig.5. Kinetic curves for the uranium(VI) ion flotation (Skrylev et al., 1981), and results of kinetic calculations
Rys. 5. Krzywe kinetyczne flotacji jonów uranu(VI) oraz wyniki obliczeń kinetycznych

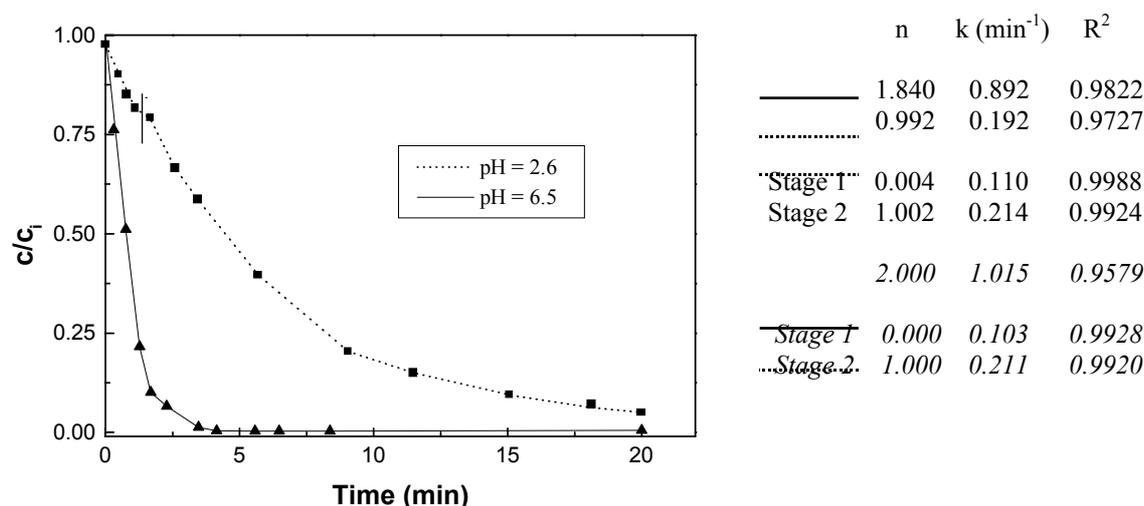


Fig.6. Kinetic curves for the thorium(IV) ions with sodium lauryl sulphate (Shakir and Samy, 1979), and results of the kinetic calculations

Rys. 6. Krzywe kinetyczne flotacji jonów toru(IV) siarczanem laurylo sodowym oraz wyniki obliczeń kinetycznych

Unexpected kinetic calculations were received for data presented in Fig. 5. According to Skrylev and Legicenko (1981), the kinetic order of flotation is two. But we obtained values of n to be close or equal 1.000 with good fitting of experimental data (with R^2 0.9809 and 0.9834, respectively). Assuming that order of flotation is equal 2.000 the fitting of Skrylev and Legicenko data (1981) to equation (6) is not good since determination coefficients were equal to 0.8051 and 0.8175.

An interesting results of kinetic calculations are received from Shakir and Samy (1979) (Fig. 6). At pH = 2.6 the thorium(IV) ions are floated in two stages. Our calculations confirm these observations. For the whole flotation curve the kinetics order was equal to 0.992 with not quite good data fitting ($R^2 = 0.9727$). But dividing these flotation curves into two parts we received very well fitting of

both stages. For the first stage n was equal to 0.004 ($R^2 = 0.9988$), while for the second stage n was equal to 1.002 ($R^2 = 0.9924$).

We also verify the kinetic data of ore flotation obtained by Ek (1992). According to him the first – order equation fits the data the best. Our calculations show that applying equation (13) the best fitting is for $n = 0.894$ ($R^2 = 0.9950$). For assumed $n = 1.000$ determination coefficient was still very high, i.e. 0.9922.

Flotation curves, which are received by us, are shown in Figures 7-9. Previously it was found (Walkowiak and Bartecki, 1973; Walkowiak, 1980, 1992) that the presence of sublate in the bulk aqueous solution is main factor influencing the ion flotation runs. Also our kinetic studies confirm it and curves shown in Fig. 7 are quite different from those in Figs. 8 and 9. The main reason of observed feature is that curves in Fig. 7 are for flotations where no sublate was precipitated in the aqueous phase. In this case the order of flotation was found to be from 0.654 to 0.999, at good data fitting (R^2 from 0.9962 to 0.9971).

In the presence of insoluble sublate in the aqueous solution a characteristic inflection appears on flotation curves (Figs. 8 and 9). This is a reason why flotation data fitting for those curves is not good (R^2 are 0.7935 and 0.8276 for PtCl_6^{2-} , as well as 0.8776 and 0.9473 for $\text{Co}(\text{CN})_6^{3-}$, respectively). Much better fitting is reached after dividing those curves into two parts.

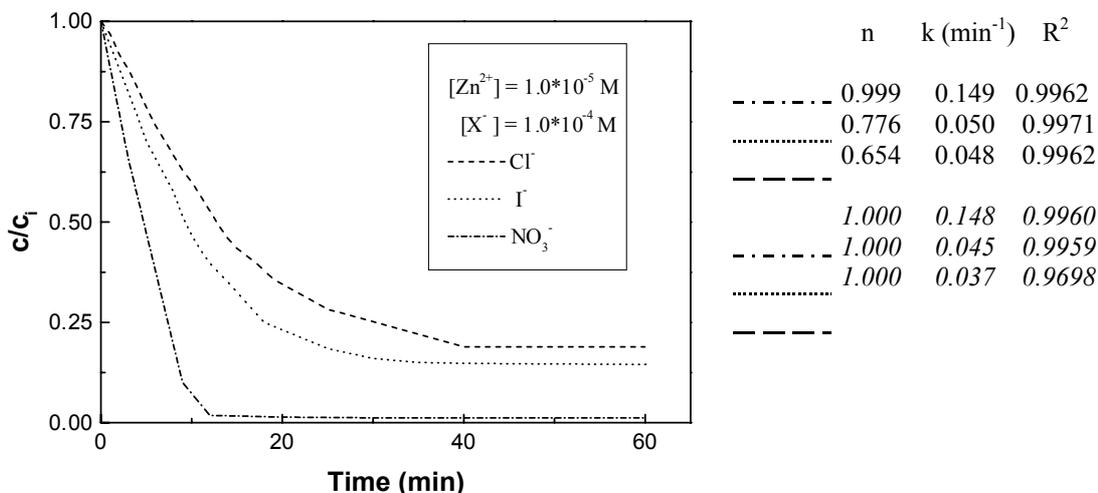


Fig.7. Flotation curves for the zinc(II) ions by $2.0 \cdot 10^{-4}$ M sodium dodecylbenzenesulfonate with kinetic calculation results

Rys. 7. Krzywe flotacyjne jonów cynku(II) sulfonianem dodecylo sodowym oraz wyniki obliczeń kinetycznych

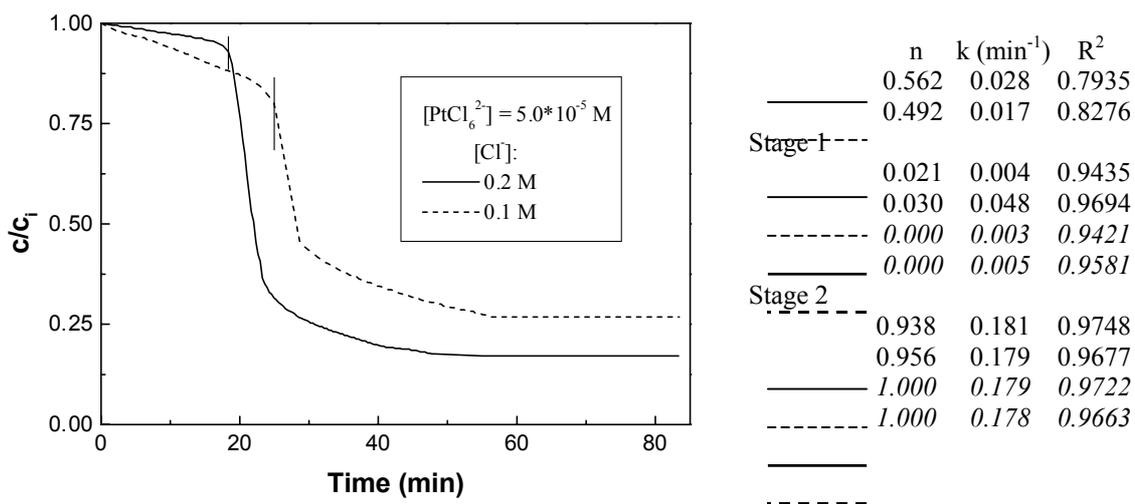


Fig. 8. Flotation curves for the PtCl_6^{2-} ions by $2.0 \cdot 10^{-4}$ M cetylpyridinium chloride with kinetic calculation results
 Fig. 8. Krzywe flotacyjne jonów PtCl_6^{2-} chlorkiem cetylpirydyniowym oraz wyniki obliczeń kinetycznych

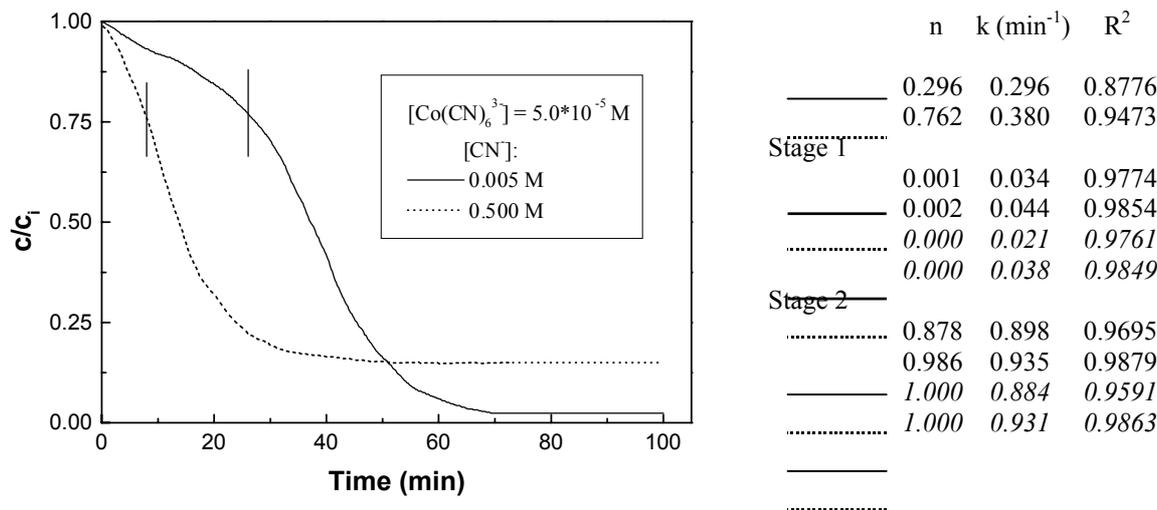


Fig.9. Flotation curves for the of $\text{Co}(\text{CN})_6^{3-}$ ions by $2.0 \cdot 10^{-4}$ M cetylpyridinium chloride with kinetic calculation results

Rys. 9. Krzywe flotacyjne jonów $\text{Co}(\text{CN})_6^{3-}$ chlorkiem cetylpirydyniowym oraz wyniki obliczeń kinetycznych

For the first stage we observed the flotation order to be close zero, i.e. 0.021 or 0.030 for PtCl_6^{2-} , and 0.001 or 0.002 for $\text{Co}(\text{CN})_6^{3-}$ with much better data fitting than for whole curves. On the other hand, for the second stage the order of flotation is close one, i.e. 0.938 or 0.956 for PtCl_6^{2-} , and 0.878 or 0.986 for $\text{Co}(\text{CN})_6^{3-}$ with quite good data fitting. The same feature was observed previously by Shakir and Samy (1979).

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Dokonano przeglądu literatury kinetyki metod flotacyjnych opisując modele flotacji: zero-, pierwszo- i drugorzędowy a także tzw. model podwójnie logarytmiczny. Zaproponowano ciągłą metodę uzyskiwania krzywych kinetycznych poprzez zastosowanie gamma radioaktywnych wskaźników izotopowych oraz odpowiedniego oprogramowania umożliwiającego zapisywanie do pliku krzywej flotacji w postaci kilkudziesięciu punktów pomiarowych. Ponadto autorzy proponują uniwersalny model kinetyczny w którym stężenie flotowanych jonów jest funkcją czasu dla nieznannej wartości stałej kinetycznej k i rzędu flotacji n . Program "Statistica" użyto do obliczania wartości k i n oraz współczynnika determinacji. Zweryfikowano wybrane literaturowe dane kinetyczne pokazując wartości k i n oraz współczynniki determinacji. Nie zawsze uzyskano zgodność obliczonych wartości rzędowości flotacji z poglądami autorów cytowanych prac; w szczególności nie potwierdzono drugiego rzędu flotacji proponowanego przez Skryleva i Legiczenko (1981). W pracy pokazano też wyniki obliczeń własnych krzywych

kinetycznych dla jonów Zn^{2+} , $Co(CN)_6^{3-}$ i $PtCl_6^{2-}$. Stwierdzono, iż w przypadku nieobecności sublatu w objętości roztworu kinetyka flotacji jest bliska pierwszorzędowej natomiast w przypadku pojawiania się sublatu w objętości roztworu na krzywych kinetycznych pojawiają się charakterystyczne przegięcia ze słabym dopasowaniem do równania kinetycznego. Dopiero podzielenie krzywych na dwa odcinki prowadzi do dobrego dopasowania tj. części pierwszej i drugiej krzywej według odpowiednio równania zero – i pierwszorzędowego.