

THE INVESTIGATION OF THE KINETICS OF HYDROCHEMICAL OXIDATION OF METAL SULPHIDES WITH THE AIM OF DETERMINATION OF THE OPTIMAL CONDITIONS FOR THE SELECTIVE EXTRACTION OF MOLYBDENUM FROM ORES

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Abstract

The kinetics of the oxidation of molybdenite, pyrite and sphalerite in solutions of nitric acid, hydrogen peroxide, and sodium hypochlorite was studied by the rotating disk method. The influence of the molar concentration of reagent, pH of solution, temperature, disk rotation frequency, and duration of measurements on the specific rate of hydrochemical oxidation of sulphides was determined. The kinetic models allowing to calculate the dissolution rate of sulphides when these parameters change simultaneously were obtained. The conditions of kinetically and diffusion-controlled processes were detected. The details of mechanism of the studied processes were revealed. The nature of intermediate solid products, the reasons and the conditions of their formation as well as the character of their influence on the kinetics of dissolution processes were determined. The probable schemes of interactions corresponding to the observable kinetic dependences were offered. The conditions of the effective and selective molybdenum leaching directly from ore without its concentration were found.

Keywords: molybdenite, pyrite, sphalerite, dissolution, kinetical models

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1. Introduction

The search of effective reagents for the selective hydrochemical extraction of valuable metals from sulphide raw materials as well as the determination of the kinetic parameters of these dissolution processes with the aim of choice of the optimal technological conditions are the most important directions of physico-chemical investigations in hydrometallurgy. The examples of such sulphide materials are molybdenite ores with the high content of talc, the presence of which impedes their concentration. The complex analysis of the kinetic dependences and the mechanisms of dissolution processes of these ores allows to solve the problem of the selective leaching of molybdenum.

2. Experimental

The most reliable kinetic characteristics of the interactions between sulphide minerals and solutions can be obtained using the rotating disc method. It is known to provide the equal accessibility of a dissolving crystal surface in conditions of regulated convection [1, 2].

The kinetics of oxidative dissolution of molybdenum (IV) sulphide (molybdenite), iron (II) persulphide (pyrite), and zinc sulphide (sphalerite) with solutions of nitric acid, hydrogen peroxide, and sodium hypochlorite was studied using the rotating disc method. The rate of dissolution (W , $\text{mol}\cdot\text{dm}^{-2}\cdot\text{s}^{-1}$) was calculated from the amount of metal cations transferred from unit disk surface area (Q , $\text{mol}\cdot\text{dm}^{-2}$) in unit time. The concentration of zinc ions in solutions was determined photometrically (in the form of the complex with xylenol orange) and polarographically. The concentrations of iron and molybdenum in solutions were determined photometrically only (in the forms of the complexes with 5-sulphosalicylic acid and lumogallion respectively).

The complete factor experiment technique [3] was used to obtain adequate models of mentioned processes. In a general case, there is the following dependence between Q and the most influential factors – the molar concentration of the reagent C ($\text{mol}\cdot\text{dm}^{-3}$), temperature T (K), disk rotation frequency ω (s^{-1}), and duration of measurements τ (s):

$$Q = k \cdot C^n \cdot \omega^m \cdot \tau^p \cdot \exp(-E_a/RT) \quad (1)$$

Taking the logarithm of the expression (1) leads to the linear equation:

$$\ln Q = \ln k + n \cdot \ln C + m \cdot \ln \omega + p \cdot \ln \tau + q/T \quad (2)$$

where $q = -E_a/R$. For the simplification of the form of polynomial models we usually selected $\ln C$, $1/T$, $\ln \omega$, and $\ln \tau$ as the influencing factors and $\ln Q$ as the response function. The following substitution of variables: $y = \ln Q$; $X_1 = \ln C$; $X_2 = 1/T$; $X_3 = \ln \omega$; $X_4 = \ln \tau$ and their subsequent transition to the non-dimensional values of factors x_i by the formula

$$x_i = (X_i - \bar{X}_i) / \Delta X_i$$

allowed, as a result of the complete factor experiment 2^4 , to obtain the polynomial model in the form of

$$y = b_0 + \sum_i b_i x_i + \sum_{i \neq j} b_{ij} x_i x_j \quad (3)$$

(The coefficients at the interactions of third and fourth orders were insignificant in all cases.)

To carry out the physico-chemical discussion of the obtained polynomial model we transformed it to the rate equation. When the pair effects were absent (i.e., $b_{ij} = 0$), for example, when the process was kinetically or diffusion-controlled, after the transition to the natural variables the polynomial equation (3) assumed the form of the equation (2). The exponentiation of the equation (2) leads to the expression in the form of (1). The differentiation of the equation (1) by τ allowed to get the following equation for the calculation of the specific dissolution rate:

$$W = \delta Q / \delta \tau = p \cdot k \cdot C^n \cdot \omega^m \cdot \tau^{p-1} \cdot \exp(q/T) \quad (4)$$

When the process proceeded in a mixed mode, some coefficients b_{ij} in the polynomial equation (3) were found to be significant. Studying the dissolution kinetics of rotating disks of sulphides, we observed the pair effects “ $C - \omega$ ”, “ $C - T$ ”, and “ $T - \omega$ ”. In the case of their simultaneous presence, the form of the equation (4) became complicated:

$$W = p_1 \cdot k_1 \cdot C^{n_1+q_2/T+m_2 \ln \omega} \cdot \omega^{m_1+q_3/T} \cdot \tau^{p_1-1} \cdot \exp(q_1/T) \quad (5)$$

or, that is equivalent,

$$W = p_1 \cdot k_1 \cdot C^{n_1+q_2/T} \cdot \omega^{m_1+m_2 \ln C + q_3/T} \cdot \tau^{p_1-1} \cdot \exp(q_1/T) \quad (5')$$

or

$$W = p_1 \cdot k_1 \cdot C^{n_1+m_2 \ln \omega} \cdot \omega^{m_1} \cdot \tau^{p_1-1} \cdot \exp[(q_1 + q_2 \ln C + q_3 \ln \omega)/T] \quad (5'')$$

where n_1 , m_1 , and q_1 are the transformed coefficients at the linear members of the equation (3); m_2 , q_2 , and q_3 are the coefficients characterizing the pair interactions “ $C - \omega$ ”, “ $C - T$ ”, and “ $T - \omega$ ” respectively. The equation (5'') demonstrates that, when the dissolution process proceeds in a mixed mode, the character of the temperature's influence on the rate changes depending from the combination of C and ω . As a result, one succeeds in obtaining an additional information allowing to evaluate the trends of the macromechanism's conversions depending on the reagent concentration and disk rotation frequency.

The rate constant of the dissolution process (K_T) was calculated from the equation (4) at the following conditions: $C = 1 \text{ mol}\cdot\text{dm}^{-3}$, $\omega = 1 \text{ s}^{-1}$. The empiric value of the activation energy E_a was found from the temperature dependence of the rate constant.

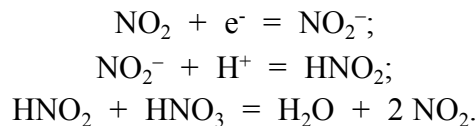
3. Results and discussion

3.1. Molybdenite dissolution

The kinetics of *molybdenite dissolution in solutions of nitric acid* was studied for $0,5 \leq C \leq 6,0 \text{ mol}\cdot\text{dm}^{-3}$, $298 \leq T \leq 318 \text{ K}$, and $1,6 \leq \omega \leq 10,0 \text{ s}^{-1}$ [4]. The obtained equation

$$W = 5.8 \cdot 10^{-3} \cdot C^{13.1 - 3700/T} \cdot \omega^{-0.37} \cdot \exp(-3454/T) \quad (6)$$

corresponds to $K_{298} = 5,4 \cdot 10^{-8} \text{ mol}^{0,3} \cdot \text{s}^{-1,4}$ and $E_a = 28,9 \text{ kJ} \cdot \text{mol}^{-1}$. The order of the process with respect to HNO_3 increases with the rise of temperature from 0,7 (at $T = 298 \text{ K}$) to 1,5 (at 318 K). The process proceeds in a mixed mode with an autocatalytic action of reaction products on the rate ($W \sim \omega^{-0,37}$). The mechanism of an autocatalytic action of NO_2 can be expressed by the following equations:



The process of *molybdenite oxidation in solutions of hydrogen peroxide* (under the conditions $0,05 \leq C \leq 1,0 \text{ mol} \cdot \text{dm}^{-3}$, $288 \leq T \leq 318 \text{ K}$, $4,0 \leq \omega \leq 25,0 \text{ s}^{-1}$, and $\text{pH} = 3,5$) was described by the adequate model

$$W = 664 \cdot C \cdot \omega^0 \cdot \exp(-6850 / T) \quad (7)$$

According to the equation (7), the process is chemically controlled ($K_{298} = 6,9 \cdot 10^{-8} \text{ dm}^3 \cdot \text{s}^{-1}$; $E_a = 56,9 \text{ kJ} \cdot \text{mol}^{-1}$; $W \sim \omega^0$). The limiting stage is the first-order (with respect to H_2O_2) chemical reaction on the surface of MoS_2 .

The rate of *molybdenite oxidation in solutions of sodium hypochlorite* doesn't depend on their alkalinity at $\text{pH} = 8 \div 12$ [5]. The rise of pH value above 12,5 leads to the deceleration of the process. For $C = 1,0 \text{--} 6,0 \text{ mol} \cdot \text{dm}^{-3}$, $T = 293 \text{--} 313 \text{ K}$, $\omega = 1,6 \text{--} 10,0 \text{ s}^{-1}$, and $\text{pH} = 8 \text{--} 12$ the interaction of MoS_2 with NaOCl can be described by the equation

$$W = 0,11 \cdot C^{0,74} \cdot \omega^0 \cdot \exp(-2705 / T) \quad (8)$$

According to (8), the process is chemically controlled: $K_{298} = 1,2 \cdot 10^{-5} \text{ mol}^{0,26} \cdot \text{dm}^{0,22} \cdot \text{s}^{-1}$; $E_a = 22,6 \text{ kJ} \cdot \text{mol}^{-1}$; $W \sim C^{0,74}$; $W \sim \omega^0$. The found rate constant is in about 220 times more than the value of K_{298} calculated for the process of molybdenite's interaction with nitric acid (equation (6)). It testifies to the higher efficiency of oxidation of MoS_2 by alkaline solutions of NaOCl in comparison with solutions of HNO_3 .

At the geotechnological processing of molybdenite ores it is necessary to know the kinetic characteristics of the processes of molybdenite's interaction with diluted weak alkaline solutions of sodium hypochlorite. The adequate model obtained for $0,02 \leq C \leq 0,1 \text{ mol}\cdot\text{dm}^{-3}$ and $8 \leq \text{pH} \leq 10$

$$W = 1.86 \cdot 10^{-2} \cdot C^{1.11} \cdot \omega^{0.22} \cdot \exp(-2072/T) \quad (9)$$

indicates that dissolution of MoS_2 proceeds in a mixed mode ($K_{298} = 1,8 \cdot 10^{-5} \text{ mol}^{-0,11} \cdot \text{dm}^{1,33} \cdot \text{s}^{-0,78}$). In comparison with the model (8) constructed for more concentrated solutions of NaOCl , the appearance of outer-diffusional limitations (the decrease of E_a to $17,2 \text{ kJ}\cdot\text{mol}^{-1}$ in combination with the appreciable influence of an agitation intensity on the interaction rate ($W \sim \omega^{0,22}$)) is marked. The pH decrease from 10 to 8 does not influence on the rate value, however a further pH reduction leads to the intensification of hypochlorite's decomposition.

3.2. Pyrite dissolution

A pyrite is the most widespread accompanying sulphide in molybdenite ores. The dependence of the rate of *pyrite oxidation in solutions of nitric acid* on HNO_3 concentration has a maximum at $C \cong 11 \text{ mol}\cdot\text{dm}^{-3}$ [6]. For $C < 11 \text{ mol}\cdot\text{dm}^{-3}$ (at $T = 303\text{-}333 \text{ K}$ and $\omega = 1,6\text{-}10,0 \text{ s}^{-1}$) the following kinetic model was obtained:

$$W = 1.92 \cdot 10^3 \cdot C^{2.81} \cdot \omega^{-0.20} \cdot \exp(-8230/T) \quad (10)$$

Its analysis indicates that the process proceeds in a mixed mode close to kinetically controlled with an autocatalytic action of reaction products on the rate: $K_{298} = 1,95 \cdot 10^{-9} \text{ mol}^{-1,81} \cdot \text{dm}^{6,43} \cdot \text{s}^{-1,20}$; $E_a = 68,4 \text{ kJ}\cdot\text{mol}^{-1}$; $W \sim C^{2,81}$; $W \sim \omega^{-0,20}$. At $C > 11 \text{ mol}\cdot\text{dm}^{-3}$ ($T = 303\text{-}333 \text{ K}$; $\omega = 1,6\text{-}10,0 \text{ c}^{-1}$) the obtained kinetic equation

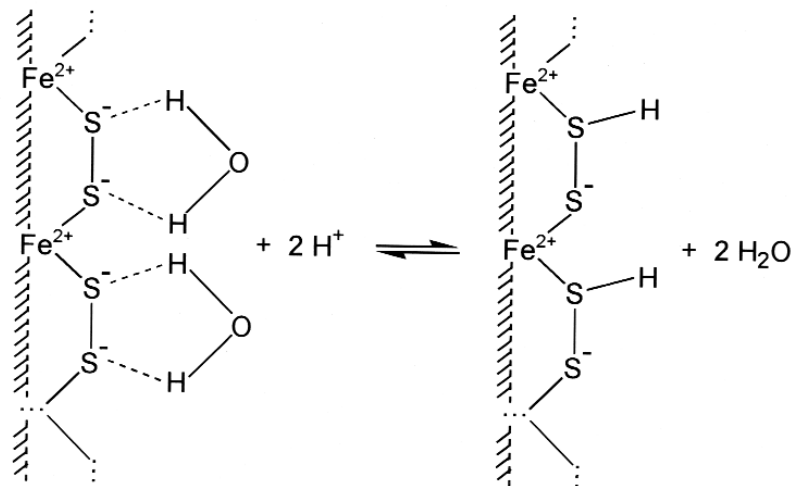
$$W = 4.69 \cdot 10^7 \cdot C^{-2.49} \cdot \omega^{3.73-1150/T} \cdot \exp(-7230/T) \quad (11)$$

is the evidence that the studied process proceeds in a mixed mode close to outer diffusion-controlled. The reason why pyrite reacted with concentrated HNO_3 at a low rate ($W \sim C^{-2,49}$) was a decrease in the solubility of reaction products.

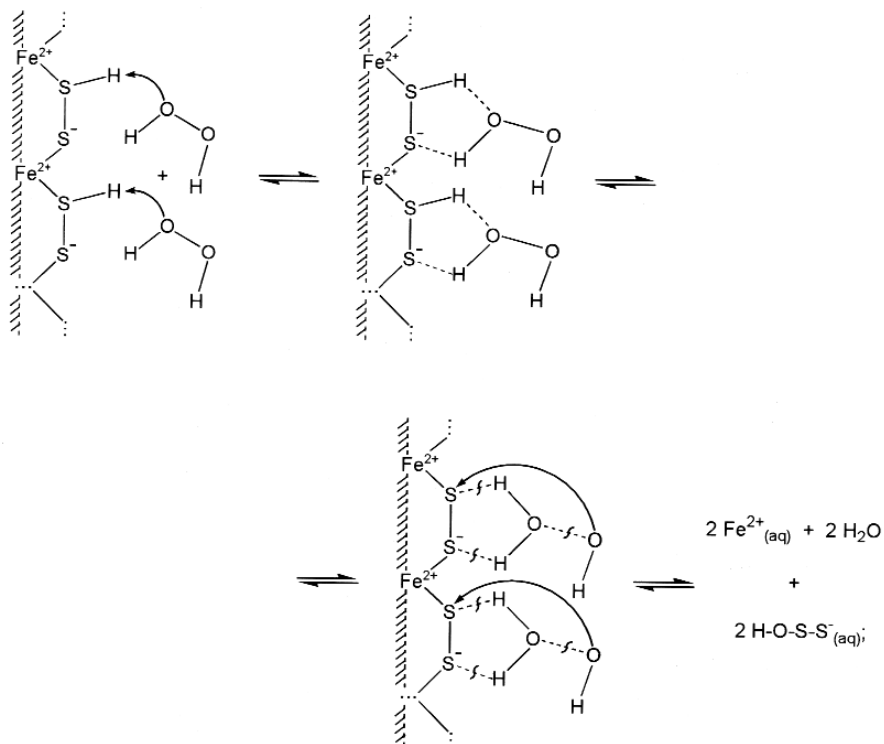
The kinetics of *pyrite oxidation in solutions of hydrogen peroxide* can be described by the model

$$W = 7.58 \cdot 10^3 \cdot C^{0.96} \cdot \omega^0 \cdot \exp(-7513/T) \quad (12)$$

which is adequate for $C = 3,0-11,5 \text{ mol}\cdot\text{dm}^{-3}$, $\text{pH} = 0-4$, $T = 296-333 \text{ K}$, and $\omega = 1,6-10,0 \text{ s}^{-1}$ [7]. It indicates that the examined process is kinetically controlled: $K_{298} = 8,52 \cdot 10^{-8} \text{ mol}^{0,04} \cdot \text{dm}^{0,88} \cdot \text{s}^{-1}$; $E_a = 62,5 \text{ kJ}\cdot\text{mol}^{-1}$; $W \sim C^{0,96}$; $W \sim \omega^0$. The pH change from 0 to 4 doesn't influence on the W value. A further pH increase from 4 to 8 decreased the rate of the reaction, which became first-order in H^+ . The probable mechanism of the interaction between FeS_2 and H_2O_2 includes the following stages: the protonization of the hydrated pyrite surface (it is a limiting stage at $\text{pH} > 4$):



the adsorption of H_2O_2 molecules onto the protonized pyrite surface with the formation of intermediate structures stabilized with hydrogen bonds (this stage is limiting at $\text{pH} = 0 \div 4$):



the oxidation of forming Fe²⁺ and HO-S-S⁻ ions to Fe³⁺ and SO₄²⁻ respectively.

The dependence of the rate of *pyrite oxidation with sodium hypochlorite solutions* on pH has a complicated character [8]. At pH < 7 the *W* value is maximum and independent on H⁺ ions activity; the process is outer diffusion-controlled (*C* = 0,005-0,1 mol·dm⁻³, *T* = 293-333 K, and *ω* = 1,6-10,0 s⁻¹):

$$W = 0.98 \cdot C \cdot \omega^{0.51} \cdot \exp(-3310/T) \quad (13)$$

According to (13), *K*₂₉₈ = 1,48·10⁻⁵ dm·s^{-0,49}; *E*_a = 27,5 kJ·mol⁻¹; *W* ~ *C*¹; *W* ~ *ω*^{0,51}. The pH increase from 6 to 10 leads to the sharp decrease of the interaction rate. Under conditions pH = 10 ÷ 12, the *W* value doesn't depend on the [H⁺]; the process is internal diffusion-controlled (*E*_a = 10,5 kJ·mol⁻¹;

$W \sim C^1$; $W \sim \tau^{-1/2}$; $W \sim \omega^0$). The limiting stage of dissolution is a diffusion through pores of the new phase film represented $\alpha\text{-Fe}_2\text{O}_3$ and $\alpha\text{-FeOOH}$ (the absence of sulphur in it was proved by X-ray photoelectron spectroscopy).

3.3. Sphalerite dissolution

Another sulphide accompanying molybdenite in ores is a sphalerite. The rate of *sphalerite oxidation in solutions of nitric acid* increases with the rise of C , reaching a maximum at $C \sim 13 \text{ mol}\cdot\text{dm}^{-3}$. A further concentration increase ($C > 13 \text{ mol}\cdot\text{dm}^{-3}$) doesn't change the W value. At $C < 13 \text{ mol}\cdot\text{dm}^{-3}$ ($T = 292\text{-}333 \text{ K}$; $\omega = 1,6\text{-}10,0 \text{ c}^{-1}$) the following kinetic equation was obtained:

$$W = 1.30 \cdot 10^{-3} \cdot C^{2.65} \cdot \omega^{0.33 \ln C - 0.92} \cdot \exp(-3310/T) \quad (14)$$

The analysis of (14) demonstrates that the process proceeds in a mixed mode close to kinetically controlled with an autocatalytic action of reaction products on its rate: $K_{298} = 1,26 \cdot 10^{-9} \text{ mol}^{-1,65} \cdot \text{dm}^{5,95} \cdot \text{s}^{-1,92}$; $E_a = 68,6 \text{ kJ}\cdot\text{mol}^{-1}$; $W \sim C^{2,65}$; $W \sim \omega^{(0,33 \dots 0,09)}$. The intermediate solid phase formed at $C = 9 \div 13,5 \text{ mol}\cdot\text{dm}^{-3}$ and $T > 313 \text{ K}$ and identified by the derivatography as the elementary sulphur does not complicate the process kinetics. At $C > 13 \text{ mol}\cdot\text{dm}^{-3}$ ($293 \leq T \leq 333 \text{ K}$; $1,6 \leq \omega \leq 10,0 \text{ s}^{-1}$) the process is chemically controlled:

$$W = 4.91 \cdot 10^{-5} \cdot C^0 \cdot \omega^0 \cdot \exp(-8050/T) \quad (15)$$

and $K_{298} = 9,11 \cdot 10^{-7} \text{ mol}\cdot\text{dm}^{-2} \cdot \text{s}^{-1}$; $E_a = 66,9 \text{ kJ}\cdot\text{mol}^{-1}$; $W \sim C^0$; $W \sim \omega^0$. The zero orders of the rate with respect to C and ω are caused by the saturation of sulphide's reaction surface with catalytically active products of HNO_3 reduction.

The dependence of the rate of *sphalerite oxidation in solutions of hydrogen peroxide* on pH has a complicated character. Under conditions $\text{pH} = 1 \div 6$, the activity of H^+ ions virtually doesn't influence on W . A rise of acidity ($\text{pH} < 1$) leads to an increase of the interaction rate; the order W with respect to H^+ is 0,5. A decrease of H^+ ions activity ($\text{pH} > 6$) causes a decrease of W according to the first-order dependence. The interaction between ZnS and solutions of

hydrochloric acid was found to be the first order with respect to the H^+ ions activity too. At $pH > 1$ the oxidative mechanism of dissolution predominates, however at $pH < 1$ the rate of hydrolytic process became comparable with the rate of oxidative one. For $C = 0,5-6,0 \text{ mol}\cdot\text{dm}^{-3}$, $pH = 2-5$, $\omega = 292-335 \text{ K}$, and $\omega = 1,6-10,0 \text{ s}^{-1}$ the interaction of ZnS and H_2O_2 can be described by the kinetic model

$$W = 0.28 \cdot C^{2.50 - 583/T} \cdot \omega^{0.10} \cdot \exp(-4940/T) \quad (16)$$

According to the equation (16), the process proceeds in a mixed mode close to kinetically controlled with the appearance of diffusional limitations at a decrease of C and an increase of T simultaneously ($K_{298} = 1,74 \cdot 10^{-8} \text{ mol}^{0,46} \cdot \text{dm}^{-0,38} \cdot \text{s}^{-0,90}$; $E_a = 41,1 \text{ kJ}\cdot\text{mol}^{-1}$; $W \sim \omega^{0,10}$). The order of the process with respect to C changes with a temperature rise from 0,5 (at $T = 292 \text{ K}$) to 0,76 (at 335 K). The probable mechanism of the interaction between ZnS and H_2O_2 includes the following stages: the protonization of the hydrated sphalerite surface (it is limiting stage at $pH > 6$); the adsorption of H_2O_2 molecules onto the protonized sphalerite surface with the formation of intermediate structures stabilized with hydrogen bonds (this stage is limiting at $pH = 1 \div 6$); the oxidation of forming sulphur-containing ions to SO_4^{2-} . At $pH < 1$ the limiting stage is most likely to be the interaction of the protonized surface of ZnS with $H_3O_2^+$ ions.

The rate of *sphalerite oxidation with solutions of sodium hypochlorite* is maximum at $pH < 6$ and independent on pH . Under the conditions: $0,01 \leq C \leq 0,15 \text{ mol}\cdot\text{dm}^{-3}$, $293 \leq T \leq 333 \text{ K}$, and $1,6 \leq \omega \leq 10,0 \text{ s}^{-1}$, the process is outer diffusion-controlled:

$$W = 4.68 \cdot 10^{-2} \cdot C \cdot \omega^{0.53} \cdot \exp(-2110/T) \quad (17)$$

from which $K_{298} = 3,94 \cdot 10^{-5} \text{ dm}\cdot\text{s}^{-0,47}$; $E_a = 17,5 \text{ kJ}\cdot\text{mol}^{-1}$; $W \sim C^1$; $W \sim \omega^{0,53}$. The limiting stage is a diffusion of $HOCl$ molecules to the disk surface. An increase in the pH from 6 to 10 caused an decrease in the interaction rate. At $pH = 10 \div 12$ the rate is pH -independent. According to the obtained kinetic

equation

$$W = 4.27 \cdot 10^{-5} \cdot C \cdot \omega^0 \cdot \exp(-1420/T) \quad (18)$$

which is correct for $C = 0,004-0,425 \text{ mol}\cdot\text{dm}^{-3}$, $T = 293-333 \text{ K}$, and $\omega = 1,6-10,0 \text{ s}^{-1}$, the process of sphalerite dissolution is internal diffusion-controlled: $E_a = 11,8 \text{ kJ}\cdot\text{mol}^{-1}$; $W \sim C^1$; $W \sim \tau^{-1/2}$; $W \sim \omega^0$. The limiting stage is a diffusion through pores of the new phase film consisted of ZnO , $\text{Zn}(\text{OH})_2$ and $(\text{ZnOH})_2\text{O}$. At $\text{pH} > 12$ an increase of the process rate caused by the dissolution of the passivating film was observed.

4. Conclusions

The applied approach for the description of minerals dissolution kinetics consisting in the obtaining and the physico-chemical interpretation of kinetic models allows to clearly present the character of the influence of the dissolution process conditions on its rate. So, for example, under the conditions of a mixed mode, the mutual influence of the parameters in the equation (14) can be well illustrated with the three-dimensional diagrams presented on Fig. 1 and Fig. 2. One can see that the increase of each studied factors (C , T , ω) leads to the rise of W . As a result, at the investigated regions of influencing parameters the maximum rate of dissolution is reached at the maximum values of the reagent concentration, temperature, and disk rotation frequency. The extent of the influence of factors mentioned above on W become lower in the following order: $T > C > \omega$.

The complex analysis of the kinetic dependences of the behaviour of MoS_2 , FeS_2 , ZnS and other sulphides during their oxidative dissolution in the presence of HNO_3 , H_2O_2 and NaOCl allows to determine the conditions of the effective and selective molybdenum leaching directly from ore without its concentration as well as to work out the way of the geotechnological extraction of Mo from sulphide ores with the high content of talc with sodium hypochlorite at $\text{pH} = 8\div 12$. The selectivity of molybdenum extraction under these conditions is illustrated on Fig. 3.

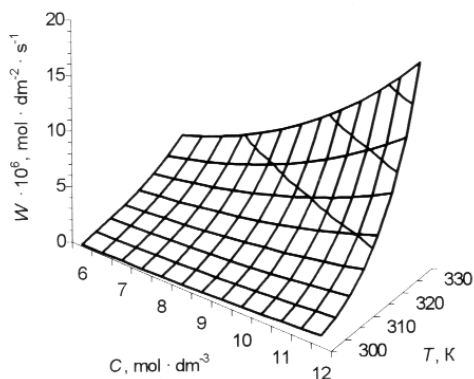


Fig. 1. The influence of HNO_3 concentration C and temperature T on the dissolution rate of ZnS W ($\omega = 1,6 \text{ s}^{-1}$)

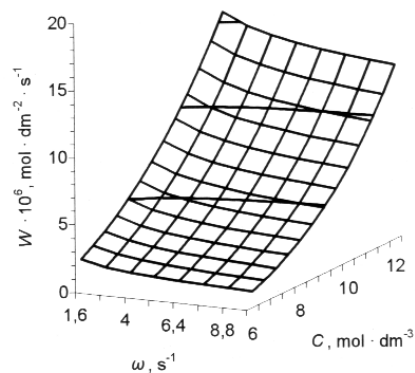


Fig. 2. The influence of HNO_3 concentration C and disk rotation frequency ω on the dissolution rate of ZnS W ($T = 333 \text{ K}$)

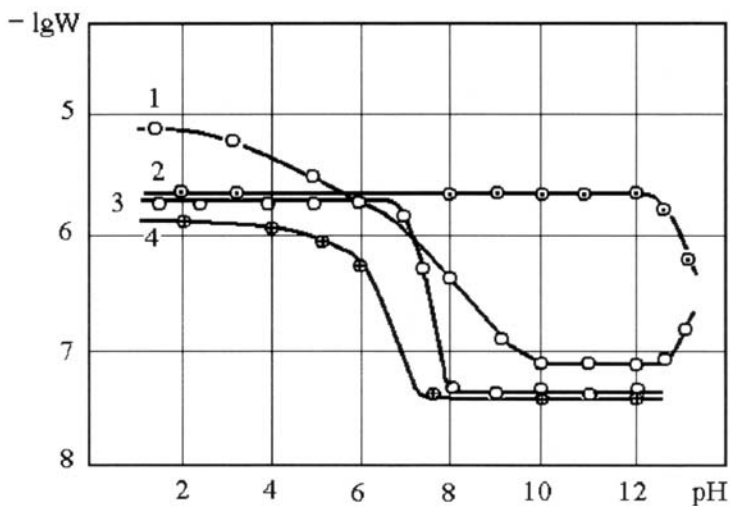


Fig. 3. The influence of pH on the dissolution rate of sphalerite (1), molybdenite (2), pyrite (3), and chalcopyrite (4) at the presence of sodium hypochlorite: $C_{\text{NaOCl}} = 0,1 \text{ mol} \cdot \text{dm}^{-3}$; $T = 298 \text{ K}$; $\omega = 4 \text{ s}^{-1}$

After the percolation leaching of molybdenum from the ore reduced to fragments with size 0-10 mm with alkaline NaOCl solution, the degree of extraction $\alpha = 95,8\%$ was reached during 23 days.

The obtained kinetic models and the revealed stages of mechanisms of the hydrochemical oxidation processes allow to optimize the existing technologies of metal extraction from the sulphide ores and to develop the new ones.

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