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Low-temperature oxidation of sulphide ores of sulphide-polymetallic deposits of Azerbaijan

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Abstract. The article presents the results of experimental studies to determine the oxidative activity for the main industrial types of sulphide ores of sulphide-polymetallic deposits in Azerbaijan. To calculate the amount of heat generated during oxidation, the dependences of the oxygen absorption rate on the temperature and moisture of the ore were established. It was found that with an increase in temperature above 50-80°C, absorption of oxygen slows down with time faster than at low (2.5-50°C) temperatures. If with an increase in temperature from 2.5 to 45-50°C the amount of absorbed oxygen increases by 7.5-12 times, then at a temperature from 45-50°C to 80°C it increases only by 3.6-3.7 times, which is explained by a decrease in moisture of the ore with an increase in temperature, due to evaporation. The time dependence of absorption rate of the oxygen for sulphide ores of sulphide-polymetallic deposits of Azerbaijan has a clearly pronounced break, which indicates a change in the oxidation mechanism. The oxidizing activity of sulphide ores of sulphide-polymetallic deposits of the Balakan ore field of Azerbaijan at low temperatures (2.5-80°C) was studied. Determination of the oxidative activity of the main industrial types of sulphide ores of sulphide-polymetallic deposits in Azerbaijan makes it possible to classify ore reserves according to the degree of tendency to spontaneous combustion, which will allow a scientifically sound approach to planning the sequence of their development and designing mines. In addition, the article presents the results of experimental studies to determine the oxidizing activity of the sulphide ores of the Katsdag, Filizchay and Katekh deposits, carried out in laboratory conditions by the method of Institute of Mining named after A.A. Skochinsky. An indicator of oxidative activity is the oxygen absorption rate constant and labeled by the letter U (ml/g hour). The results of laboratory studies show that the oxidation of the ore leads to an increase in temperature and ignition and depends on a large number of factors such as mineralogical composition, chemical activity, humidity, temperature, fragmentation, etc. The dependence of the total amount of oxygen absorbed by sulphide ores on time at various temperatures is obtained. It has been established that with increasing temperature, the rate of oxygen absorption by sulphide ores increases. The values of the temperature coefficient ($K_t < 2.0$) in the temperature range of 2.5-80°C show that the rate of the oxidation of sulphide ore is controlled by oxygen diffusion, and not by the rate of the chemical reaction. With equal fragmentation and equal distribution of chemical elements in ore samples, the oxidation rate is directly proportional to the outer surface of the sulphide ore.

Key words: sulphide ore, sulphide-polymetallic deposit, oxygen absorption, oxygen absorption rate constant, oxidation, chemical activity.

Низькотемпературне окислення сульфідних руд колчеданно-поліметалевих родовищ Азербайджану

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Анотація. У статті наводяться результати експериментальних досліджень щодо визначення окисної активності для основних промислових типів сульфідних руд колчеданно-поліметалічних родовищ Азербайджану. Для розрахунку кількості тепла, що утворюється при окисненні, встановлені залежності швидкості поглинання кисню від температури та вологості руди. Виявлено, що з підвищенням температури вище 50-80°C поглинання кисню уповільнюється з часом швидше, ніж за низьких (2,5-50°C) температур. Якщо з підвищенням температури від 2,5 до 45-50° С кількість поглиненого кисню збільшується в 7,5-12 разів, то при температурі від 45-50°C до 80°C воно збільшується лише в 3,6-3,7 рази, що пояснюється зменшенням вологості руди із підвищенням температури, внаслідок випаровування. Залежність швидкості поглинання кисню для сульфідних руд колчеданно-поліметалічних родовищ Азербайджану від часу має чітко виражений злам, що свідчить про

зміну механізму окислення. Вивчено окисну активність сульфідних руд колчеданно-поліметалічних родовищ Білоканського рудного поля Азербайджану за низьких температур (2,5-80°C). Визначення окислювальної активності основних промислових типів сульфідних руд колчеданно-поліметалічних родовищ Азербайджану дає можливість класифікувати запаси руд за рівнем схильності до самозаймання, що дозволить науково обґрунтовано підійти до планування черговості їх освоєння та проектування рудників. Крім того, у статті були наведені результати експериментальних досліджень щодо визначення окисної активності сульфідних руд Кацдаг, Філізчай та Катехського родовищ, проведені в лабораторних умовах методом Інституту гірничої справи імені О.О. Скочинського. Показником окисної активності є константа швидкості поглинання кисню – U (мл/г на годину). Результати лабораторних досліджень показують, що окислення руди призводить до підвищення температури і займання і залежить від великої кількості факторів таких як мінералогічний склад, хімічна активність, вологість, температура, ступінь дроблення та ін. Встановлено, що з підвищенням температури швидкість поглинання кисню сульфідними рудами зростає. Значення температурного коефіцієнта ($K_t < 2,0$) в інтервалі температур 2,5-80°C показують, що швидкість процесу окислення сульфідної руди управляється дифузією кисню, а не швидкістю протікання хімічної реакції. При рівномірному дробленні та рівномірному розподілі хімічних елементів у наважках руди швидкість окислення прямо пропорційна зовнішній поверхні сульфідної руди.

Ключові слова: сульфідна руда, колчеданно-поліметалеве родовище, поглинання кисню, константа швидкості поглинання кисню, окиснення, хімічна активність.

Introduction

Oxidation of sulphide ores in underground mining proceeds under constantly changing thermal and moisture conditions. Since the temperature rises during self-heating in the accumulation of sulphide ores, in order to calculate the amount of heat generated, it is necessary to establish the dependence of the oxygen absorption rate on temperature, ore moisture content, and air flow rate. The results of experimental studies to determine the oxidative activity on temperature for different types of ores of various sulphide-polymetallic deposits of Azerbaijan are given in Eable 1. (Ainbinder et al., 2017; Valiev et al., 2008; Veselovsky et al., 1972; Zaitseva et al., 1972; Rylnikova et al., 2020; Kondratiev et al., 2019; Hong 2020; Fu-qiang, et al., 2014; Teterev et al., 2018).

Cases of self-heating and a significant decrease in the oxygen content in the atmosphere of mining were observed in the explored mining of sulphide ore deposits in Azerbaijan. Due to the fact that sulphide-polymetallic deposits, the ores of which are prone to oxidation and spontaneous combustion, are extremely diverse both in nature and in mining and geological conditions, the approach to their exploitation should be strictly individual. Using the rich experience of similar mines to prevent the oxidation and spontaneous combustion of ores is not possible without knowledge of the properties of ores that characterize their oxidative activity and are determined experimentally.

The article presents the results of experimental studies to determine the oxidative activity and spontaneous combustion of sulphide ores of the Katsdag, Filizchay and Katekh deposits, carried out in laboratory conditions by the method of Institute of Mining named after A.A. Skochinsky, the basis of which is the determination of the oxygen absorption rate constant.

Results

Studies have established that during the oxidation of freshly fragmented ore, the initial absorption of oxygen is fast, and an oxide layer is formed, which prevents further oxidation. Studies carried out over several years have shown that the rate of oxygen absorption by sulphide ores is long-lived and slowly fading, but incessant.

When plotting the obtained values on a chart, the dependence of the oxygen absorption rate on temperature is depicted by exponential curves (Fig. 1). If the rates are plotted on the ordinate axis on a logarithmic scale, then we can expect that the experimental data will fall on straight lines. As we can be seen from figure 2 this assumption is satisfactorily justified.

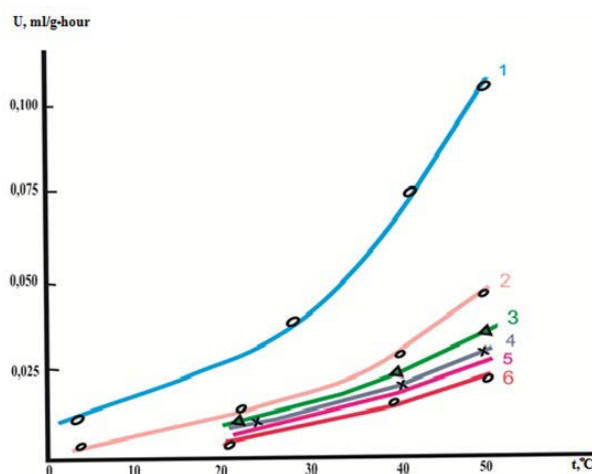


Fig. 1. Dependence of the oxygen absorption rate constant on temperature

1. Massive pyrrhotite ore of the Katsdag deposit
2. Stringer-disseminated quartz – chalcopyritic ore of the Katsdag deposit
3. Banded pyrite ore of the Filizchay deposit
4. Massive galena-sphalerite-pyrite ore of the Katekh deposit
5. Massive pyrite ore of the Filizchay deposit
6. Spotted-disseminated pyrite ore of the Filizchay deposit

Table 1. Dependence of the chemical activity of sulphide ores of sulphide-polymetallic deposits of Azerbaijan on temperature

Types of ores	The oxygen absorption rate constant, ml/g, hour								Temperature coefficient		Energy activ- ity E_k , cal/ mol
	t=2,0°C		t=20°C			t=40°C	t=50°C				
	K_{24}	U_{250}	K_{24}	U_{250}	K_{24}	U_{250}	K_{24}	U_{250}	K_t^k	K_t^u	
2	3	4	5	6	7	8	9	10	11	12	13
Massive pyr- rhotite			0.0825	0.0296		0.0690	0.7933	0.1053	2.29	1.61 1.574 1.533	15485
	0.0373	0.01107	0.0693	0.0532					1.36	2.13	28978
			0.1751	0.0492				0.1125		1.32	
Stringer-dis- seminated quartz-chal- copyritic			0.0196	0.0128	0.0353	0.0294	0.1253	0.04764	1.94	1.6	12275
			0.0131	0.0093	0.0261	0.0245	0.0439	0.03425	1.46 1.54 1.68	1.664 1.574 1.425	7994
Banded py- rite	0.0884	0.001715	0.4844	0.0081					2.34	1.725	16873
			0.0285	0.0096		0.0235	0.099	0.03566	1.56	1.62 1.572 1.526	15421
Massive – pyrite			0.0153	0.0074			0.058	0.02709	1.62	1.59	8897
			0.0114	0.0059			0.0449	0.0261	1.63	1.59	9041
Spotted-dis- seminated pyrite			0.0088	0.0058			0.0451	0.0229	1.80	1.56	10807
			0.0182 0.0066				0.0603	0.0235	1.51	1.57	7923

Deposits	Types of ores	The oxygen absorption rate constant, ml/g, hour								Temperature coefficient		Energy activity	
		t=2,0°C		t=20°C		t=40°C		t=50°C		K_t^k	K_t^u	E_k , cal/mol	E_y , cal/mol
		K_{24}	U_{250}	K_{24}	U_{250}	K_{24}	U_{250}	K_{24}	U_{250}				
1	2	3	4	5	6	7	8	9	10	11	12	13	14
Katekh	Massive gale-na-sphalerite-pyrite			0.0404	0.009144		0.0202	0.0858	0.0302	1.31	1.57 1.54 1.50	4977	4895
				0.0131	0.0071			0.0289	0.0266	1.33	1.60	5231	8233
	Concretionary massive gale-na-sphalerite-pyrite			0.0077	0.0061			0.0417	0.0201	1.83	1.53	9652	7863
				0.0119	0.00636			0.0819	0.0239	2.00	1.6	12771	3752
	Stockwork-stringer gale-na-sphalerite-pyrite			0.0088	0.0058			0.0414	0.0219	1.74	1.58	10239	8840
				0.0045	0.0018			0.0181	0.0065	1.64	0.58	9142	2364

Based on figure 2, the dependence of the oxygen absorption rate on temperature can be expressed by the following equation.

$$\lg U = \lg U_0 + K_t \cdot t \text{ or } U = U_0 e^{2,3K_t \cdot t} \quad (1)$$

On a logarithmic scale, the dependence of the temperature coefficient on temperature is also described by straight lines (Fig. 2).

$$\lg \frac{\Delta K}{\Delta t} = \lg \left(\frac{\Delta K}{\Delta t_0} \right) + Bt$$

As we can be seen from figure 2 inclination angles for all types of sulfide ores are almost the same. The tangents of these angles can be calculated from the above equations and determined graphically based on the figure.

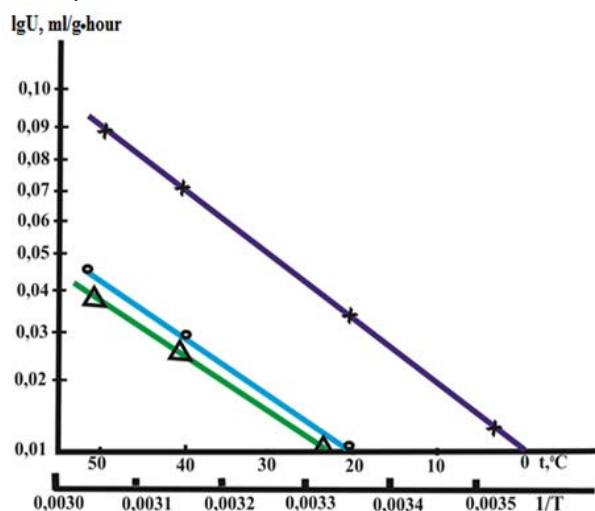


Fig. 2. Dependence of the oxygen absorption rate constant between sulphide ores and the value of $1/T$

x – ore of the Katsdag deposit
o – ore of the Filizchay deposit
Δ – ore of the Katekh deposit

The inclination angle can be calculated by the following equation:

$$\lg U_1 - \lg U_2 = tg\alpha \left(\frac{1}{T_2} - \frac{1}{T_1} \right) \quad (3)$$

For example, for ores of the Filizchay deposit the chemical activity at temperatures $t_1=20^\circ\text{C}$ and $t_2=40^\circ\text{C}$ correspondingly consist of $U_1=0,00962\text{ml/g/hour}$ and

$$U_2=0,0235\text{ml/g/hour}; \quad \frac{U_1}{U_2} = 0,41 \quad \text{and} \quad \lg 0,41 = -0,3872,$$

$$\frac{1}{T_1} - \frac{1}{T_2} = 0,0002.$$

$$\text{Then } tg\alpha = \frac{\lg 0,41}{0,0002} = \frac{-0,3872}{0,0002} = -1936$$

We measure on the diagram $\Delta \lg U = 79\text{mm}$,

$$\Delta \frac{1}{T} = 58 \text{ mm}.$$

$$\text{Hence } tg\alpha = \frac{79}{58} = 1,362$$

However, scale is $100\text{mm} = 0,001 \frac{1}{T}$, that is 1000 times more, than a logarithmic unit along the ordinate axis. That is why it is necessary to divide obtained

value of $\Delta \frac{1}{T}$ by 1000. Then

$$tg\alpha = 1,362 \cdot 1000 = 1362.$$

To get the exponential equation, we turn to natural logarithms.

$$\frac{U_1}{U_2} = e^{2,3 tg\alpha \left(\frac{1}{T_2} - \frac{1}{T_1} \right)} \quad (3)$$

On the other hand, to characterize various physical processes, the activation energy is widely used, which is calculated by the Arrhenius equation according to the following formula:

$$\ln U = \ln U_0 - \frac{E}{RT} \quad (4)$$

Comparing equation (3) and (4) we get

$$2,3 \cdot 1362 = 3132,6 = \frac{E}{R}$$

Hence $E = 3132,6 \cdot R = 313,6 \cdot 1,986 = 6221,3 \text{ cal/moldeg}$. Thus, we get the fictitious activation energy of 6221,3 cal, that is significantly less, than for the chemical reactions.

Using the described relationships, one can easily take into account the dependence of heat generation on temperature for a self-heating accumulation of sulphide ores. To do this, it is necessary to know the value of chemical activity at a certain temperature and the average temperature in the accumulation of ore over the considered period. Based on these data in Fig. 3, one can determine the value of U for this average temperature.

The increase in the temperature coefficient of chemical activity of sulphide ores as a result of oxidation is of considerable interest. This is explained by the fact that for sulphide ores at a temperature of 80°C , the absorption of oxygen slows down with time faster, less than at 2.5 or 20°C , since in the first case there is a significant destruction of the oxidized layer that impedes the flow of oxygen. (Iliyas et al., 2011; Boon, 2001; Liu, et al., 2011).

For sulphide ores, the temperature coefficient in the range from 2.5 to 80°C has a value of about 1.32-1.73 (except for two samples of the Katsdag deposit).

An increase in temperature from 2.5°C to $45-50^\circ\text{C}$ increases the amount of absorbed oxygen by 7.5-12 times, and an increase in temperature from $45-50^\circ\text{C}$ to 80°C increases the amount of absorbed oxygen only by 3.6-3.7 times, which is explained by a decrease in moisture when the temperature rises due to evaporation (Rao, 2020; Yuan, 2015; Somot et al., 2010; Rylnikova et al., 2020; Skochinsky et al., 2011; Wang et al., 2013; Skjold et al., 2016).

The experimental data carried out showed that the total amount of oxygen absorbed by sulphide ores is subject to regularity.

$$Q_{abs.o_2} = A \tau_a, \text{ ml/g} \quad (5)$$

where $Q_{abs.o_2}$ – the total amount of oxygen absorbed by sulphide ores, ml;

A – coefficient, equal to amount of oxygen absorbed by sulphide ores at $\tau = 1$ hour, ml/g hour, so, in this case $Q_{abs} = A \cdot \tau$ – time contact of sulphide ores with oxygen of air, hour;

a – coefficient, characterizing the intensity of slowing down the absorption rate of oxygen of the air over time.

Processing the results of experimental studies showed that the coefficient A and a largely depend on temperature. As the temperature rises, A increases, and a decreases. The dependence of these coefficients on temperature is well depicted by straight lines on a semilogarithmic scale (Fig. 3).

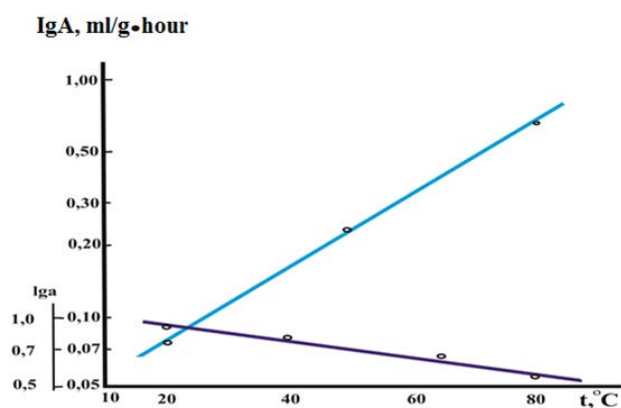


Fig. 3. The dependence of the coefficients (A) and (a) on temperature

Some studies (Veselovsky et al., 1972; Pihlak, 1974; Boon, 2001; Rylnikova et al., 2020) have established a significant dependence of oxygen absorption by sulphides on moisture. This dependence is also confirmed by our studies for sulphide ores of sulphide-polymetallic deposits in Azerbaijan. During long-term storage in air of samples of sulphide ore from the Chirag-Derin deposit, they do not undergo visible changes, and the rate of absorption of atmospheric oxygen by them decreases, which is explained by the appearance of a very thin layer of oxidation products on ore surface. Ores of the Katsdag, massive pyrite ores of the Filizchay and Katekh and banded pyrite ore of the Filizchay deposits behave differently. These ores (especially the pyrrhotite ores of the Katsdag deposit) acquire a red tint in a humid atmosphere; quickly covered with a layer of iron oxides.

Since the slowdown in oxygen absorption is caused by the growth of a layer of oxidation products on the surface of the grains, it can be expected

that this layer can be dissolved with sulphuric acid and the oxygen absorption rate will increase. For this purpose, experiments were carried out with samples of sulphide ores from sulphide-polymetallic deposits in Azerbaijan and the following results were obtained (Table 2).

Table 2. Influence of H_2SO_4 on the oxygen absorption rate by sulphide ores of sulphide-polymetallic deposits of Azerbaijan.

The absorption rate of oxygen constant U , ml/gh	Ores			
	Ordinary	humidified with distilled water	wetted with 10% solution of H_2SO_4	wetted with 20% solution of H_2SO_4
K_{24}	0.0082		0.0757	0.1693
U_{250}	0.0059		0.0087	0.0376
K_{24}	0.0074	0.0117	0.1857	
U_{250}	0.0046	0.0029	0.1160	

Discussion

Thus, sulphuric acid accelerated the oxygen absorption process. However, a similar experiment with samples of Dehtar pyrite showed opposite results. Based on this, the authors of this work decided that the sulphuric acid formed during pyrite should participate in slowing down the absorption of oxygen, which, in the authors' opinion, is erroneous. (Ismailov, 1975; Rylnikova et al., 2019; Chao et al., 2005).

There is an influence of the value of the free reacting surface of the ore on the oxidation process. It is of a great interest. Studies carried out with samples of ores of various sizes showed that at the beginning the rate of oxygen absorption for fine fractions is greater than for coarse fractions. However, for fine fractions, it decreases faster with time and after approximately 700 hours from the start of the experiment, the curves of the oxygen absorption rate along time intersect for different fractions, after which the fine ore fraction becomes less active than the coarse one. This allows us to make a very important conclusion that coarse fractions are the most dangerous for spontaneous combustion than the powder. A similar result was obtained by Prof. Veselovsky et al. for coals (Veselovsky et al., 1972). The results of chemical analysis showed that with equal fracturing and equal distribution of chemical elements in ore samples, the oxidation rate is directly proportional to the outer surface of the sulphide ore (Table 2), while some researchers denied direct proportionality between the chemical activity to atmospheric oxygen and the oxidation surface.

Conclusions

1. The quantitative and qualitative influence of temperature on the process of oxidation of the main industrial types of sulphide ores of sulphide-polymetallic deposits of Azerbaijan has been established.

2. The values of the temperature coefficient ($K_t < 2.0$) in the temperature range of 2.5–80°C show that the rate of the sulfide ore oxidation process is controlled by oxygen diffusion, but not by the rate of the chemical reaction.

3. Smaller fractions – 0.25 + 0.0 mm, – 0.5 + 0.25 mm are deactivated faster than coarse ones. This allows us to make a very important conclusion that

coarse fractions are the most dangerous for spontaneous combustion than the powder.

4. With equal fracturing and distribution of chemical elements within ore samples, the oxidation rate is directly proportional to the outer surface of the sulphide ore.

5. The performed studies on oxidative activity have shown that sulphide ores of sulphide-polymetallic deposits of Azerbaijan are characterized by high oxidative activity. The most active in relation to atmospheric oxygen are the ores of the Katsdag deposit ($U=0.072-0.016$ ml/g·hour), the ores of the Katekh deposit are less active ($U=0.0102-0.0041$ ml/g·hour), the ores of the Filizchay deposit occupy intermediate position ($U=0.0304-0.0052$ ml/g·hour).

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