

# Structural and temperature sensitivity of the chloride leaching of copper, lead and zinc from a mechanically activated complex sulphide

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## Abstract

The hydrometallurgical processing of complex concentrates represents an ecologically attractive alternative with respect to classical pyrometallurgical technologies. The structural and temperature sensitivity of chloride leaching of copper, lead and zinc from a complex CuPbZn sulphide concentrate of Slovak origin which was mechanically activated in an attritor was studied. Mechanical activation influences the leaching kinetics and the recoveries of copper and zinc, but only the leaching kinetics of lead. It has been found that the reaction is sensitive to the increase in surface area and decrease in crystalline phase content of the studied concentrate due to mechanical activation. The demonstration of a linear relationship shows the structural sensitivity of the reaction. For copper and zinc, the reaction temperature sensitivity for the optimum mechanically activated sample in the temperature region 323–363 K showed a change in the rate-determining step at 343 K. Lead showed no change in mechanism over this temperature range. The suggested order of structural sensitivity galena > chalcopyrite > sphalerite is in accordance with their temperature sensitivity.

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**Keywords:** Mechanical activation; Sulphide concentrate; Chloride leaching; Copper; Lead; Zinc

## 1. Introduction

Chloride systems in hydrometallurgy have been used for the treatment and recovery of precious metals for a number of years. In the past few years, the application of chloride hydrometallurgy for the processing of non-ferrous metals in large-scale mineral processing has been considered seriously (Barbery et

al., 1980; Campbell and Ritcey, 1981; Wadsworth, 1982; Canterford, 1983; Winand, 1991).

The oxidation potential of ferric chloride leaching systems results in elemental sulphur as one of the major by-products, a form generally more acceptable environmentally than the sulphur dioxide from pyrometallurgy or sulphate from pressure hydrometallurgy.

The main advantages of application of chloride-based systems over sulphate-based leaching are:

- Chloride leaching is performed at moderate temperature solubilizing all metals.

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- Pyrite is not attacked by chloride salts.
- Most metal chlorides are considerably more soluble than the corresponding sulphate salts.
- Facile regeneration of the leaching reagents in cyclic processes.
- Most sulphide sulphur in the mineral is converted to the elemental sulphur.

However, ferric chloride solutions are corrosive to most metals, although this problem may be readily overcome with the use of plastic materials.

The leaching of metal sulphide minerals in chloride media is a subject of considerable interest and many excellent review articles have been published (Dutrizac, 1978, 1986, 1992; Bobeck and Su, 1985; Majima et al., 1985; Warren et al., 1985; Furstenau et al., 1986; Hirato et al., 1986; Buttinelli et al., 1990; Palencia and Dutrizac, 1991). These studies indicated that the ferric chloride solutions could rapidly, but unselectively leach valuable metals such as copper, lead and zinc from sulphide minerals, leaving the bulk of the pyrite and sulphur. The reaction mechanism of chloride leaching is still very controversial and it is not unambiguous with the reaction mechanism changing with temperature. The chloride leaching of sulphide concentrate has been studied by various researchers (Cathro, 1974; Greig, 1987; Kobayashi et al., 1990; Neou-Singouna and Fournalis, 1990; Mulak and Wawrzak, 1994; Havlík et al., 1995; Havlík and Kammel, 1995; Mulak, 1998).

The mechanical activation of minerals represents nowadays an important contribution to different fields of solid processing technology. In extractive metallurgy, activation by intensive grinding decreases the reaction temperature in pyrometallurgy, increases leaching kinetics in hydrometallurgy and leads to alternative products and potential mitigation of the environmental impact, which is becoming increasingly important with time (Baláž, 2000; Tromans and Meech, 1999, 2001; Welham, 1997, 2001).

A few articles have been published on the chloride leaching of mechanically activated minerals (Björling and Mulak, 1974; Baláž, 1996; Maurice and Hawk, 1998). However, the ferric chloride leaching of copper, lead and zinc from complex CuPbZn sulphide concentrate after mechanical activation has not been studied so far.

This paper investigates the structural and temperature sensitivity of chloride leaching of a mechanically activated complex CuPbZn sulphide concentrate.

## 2. Experimental

### 2.1. Materials

The investigation was carried out using a sample of a complex copper–lead–zinc sulphide concentrate from Banská Hodruša, Slovakia which had the following chemical composition: 353 g t<sup>-1</sup> Au, 170 g t<sup>-1</sup> Ag, 0.93% Cu, 4.08% Pb, 3.57% Zn, 20.06% Fe, 0.2% Sb, 0.17% Hg, 0.02% Bi, 0.12% As, 0.03% Mn, 0.02% Co, 0.07% Mg, 5.5% SiO<sub>2</sub>.

X-ray diffraction (Fig. 1) showed that the major minerals present were chalcopyrite CuFeS<sub>2</sub>, galena PbS, sphalerite ZnS, tetrahedrite Cu<sub>12</sub>Sb<sub>4</sub>S<sub>13</sub>, pyrite FeS<sub>2</sub> and quartz SiO<sub>2</sub> as noted previously (Jágerský, 1999).

### 2.2. Mechanical activation

The mechanical activation was performed in a stirring ball mill (attritor) Molinex PE 075 (Netzsch, Germany) using a 2<sup>3</sup> factorial experiment, extended by use of a second order polynomial for the specific surface area response. These chosen parameters of the attritor as variables studied were: weight of sample—50 and 200 g; revolutions of the milling shaft—600 and 1200 min<sup>-1</sup>; time of mechanical activation—15 and 60 min. The feed concentrate was < 71 µm. The mill had a 500-mL grinding chamber which contained 2000 g of 2-mm diameter steel balls and 200 mL of water; the mill was run at ambient temperature.

### 2.3. Physico-chemical characteristics

#### 2.3.1. Specific surface area

The specific surface area  $S_A$  was determined by the low-temperature nitrogen adsorption method in a Gemini 2360 sorption apparatus (Micromeritics, USA).

#### 2.3.2. X-ray diffraction analysis

The X-ray diffraction of the investigated samples of concentrate was measured on a DRON 2.0 diffrac-

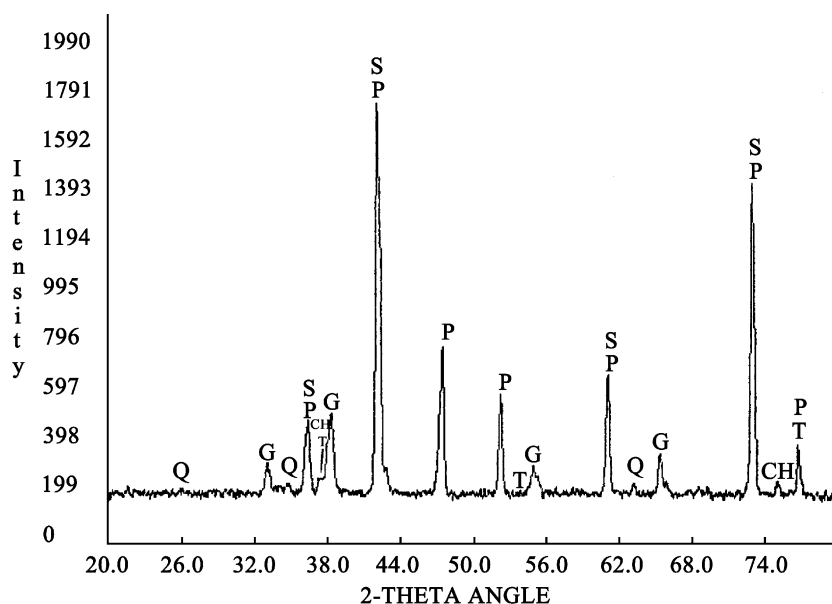


Fig. 1. X-ray diffraction pattern of nonactivated complex CuPbZn sulphide concentrate: pyrite (P), sphalerite (S), tetrahedrite (T), chalcopyrite (CH), galena (G), quartz (Q).

tometer using FeK $\alpha$  radiation operated at 10 mA and 25 kV, the time constant was 2 s and the system ran in  $\theta$ – $2\theta$  mode at  $2^\circ \text{ min}^{-1}$ .

The effect of mechanical activation was evaluated by an increase  $X$  (%) in the X-ray amorphous portion of mineral compared with the reference sample (non-activated), which is assumed to correspond to 100% crystallinity, where  $X$  is defined as

$$X = \frac{U_o}{I_o} \cdot \frac{I_x}{U_x} \cdot 100 \quad (\%) \quad (1)$$

and  $U_o$  and  $U_x$  denote the backgrounds of the reference sample and activated sample while  $I_o$  and  $I_x$  are integral intensities of diffraction lines of the reference sample and activated sample, respectively. The complementary value of amorphization,  $A$ , was calculated by Eq. (2) and used for the calculation of degree of minerals disordering.

$$A = 100 - X \quad (\%) \quad (2)$$

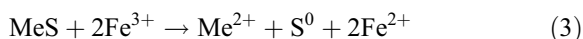
### 2.3.3. Particle size analysis

The particle size distribution of the ground concentrate was measured by a laser beam scattering in a Helos and Rodos granulometer (Sympatec, Germany).

The mean particle diameter was calculated as the first moment of the volume size distribution function.

### 2.4. Chloride leaching

The leaching of complex copper–lead–zinc sulphide concentrate may be described by the Eq. (3)



where Me is Cu, Pb or Zn.

The kinetics of leaching were investigated using a 500-mL glass reactor into which 400 mL of leaching solution (1 M FeCl $_3$  + 0.2 M HCl) and 5 g of concentrate were added. The leaching was performed at 323–363 K, pH < 1 and stirring rate  $460 \text{ min}^{-1}$  for up to 30 min. Aliquots (5 mL) of the solution were withdrawn at appropriate time intervals for determination of the content of dissolved copper, lead and zinc by atomic absorption spectrophotometer.

#### 2.4.1. Processing of kinetic data

The leaching kinetics of copper, lead and zinc can be described by the following rate Eq. (4)

$$e_{\text{Me}} = k_1 + k_3 t - k_1 e^{-k_2 t} \quad (4)$$

where  $\varepsilon_{\text{Me}}$  is recovery of metal (Cu, Zn, Pb) into the leach solution,  $t_L$  is time of leaching (s),  $k_1$ ,  $k_2$  and  $k_3$  are the rate constants ( $\text{s}^{-1}$ ), the meaning of which results from the paper by Tkáčová and Baláž (1988).  $k_0$  is the initial rate constant which is defined in the form

$$k_0 = k_3 + k_1 k_2 \quad (5)$$

### 3. Results and discussion

#### 3.1. Physico-chemical changes in mechanically activated complex CuPbZn concentrate

The mechanical activation performed in an attritor causes both surface and bulk structural changes in the studied concentrate. The primary effect of mechanical activation is comminution of particles which results in changes in a great number of physico-chemical properties of the minerals in the concentrate.

The values of specific surface area  $S_A$  of non-activated and the selected samples of mechanically activated CuPbZn concentrate (samples of factorial experiment) are summarized in Table 1. It can be seen that  $S_A$  increases with the time of mechanical activation.

In regard to the particle size analysis, the results are summarized in Table 2. The fraction of 0.9–10  $\mu\text{m}$  particles and the surface area  $S_G$  increased with the increasing energy input of grinding  $E_M$ . For instance, if we compare the nonactivated sample ( $E_M = 0 \text{ kW h t}^{-1}$ ) with the sample of mechanically activated concentrate at energy input of grinding  $E_M = 82 \text{ kW h t}^{-1}$ , we can find that in the nonactivated sample, only

Table 2

Energy input of grinding,  $E_M$ , granulometric surface area,  $S_G$ , and fraction of particles less than  $x \mu\text{m}$ ,  $Q(x)$ , of nonactivated and the selected samples of mechanically activated samples complex CuPbZn sulphide concentrate

$E_M$ ( $\text{kW h t}^{-1}$ )	$S_G \times 10^3$ ( $\text{m}^2 \text{kg}^{-1}$ )	$Q(10)$ (%)	$Q(7.5)$ (%)	$Q(5)$ (%)	$Q(2.6)$ (%)	$Q(1.1)$ (%)
0	0.11	13.51	10.13	7.05	3.87	1.25
82	0.48	84.8	71.86	51.56	26.36	8.05
303	0.98	100	100	96.91	71.42	26.63
404	0.99	100	99.86	96.99	71.87	27.50
524	1.08	100	99.54	95.36	77.48	33.43
808	1.17	100	100	100	86.18	37.10

14% of particles are  $< 10 \mu\text{m}$ , whereas in the activated sample it was 85%,  $< 10 \mu\text{m}$ . The samples with higher energy inputs were 100%,  $< 10 \mu\text{m}$ .

The changes in surface area are accompanied by bulk changes of the concentrate as well (Table 1). The amorphization,  $A$ , of minerals creating the constituents of the concentrate was calculated by Eq. (2). The calculated values for individual sulphide minerals increase with the time of mechanical activation but only up to 37 min. In the case of samples activated for 60 and 64 min, the amorphization decreases with the time of mechanical activation. This may be due to mechanochemical equilibrium. In this case, the formation of agglomerates was not observed.

#### 3.2. Chloride leaching of mechanically activated concentrate

##### 3.2.1. Structural sensitivity of the Cu, Pb and Zn leaching

Figs. 2–4 present the plots of the fraction leached for copper, lead and zinc as a function of leaching

Table 1

Energy input of grinding,  $E_M$ , time of mechanical activation,  $t_M$ , specific surface area,  $S_A$ , and amorphization,  $A$ , of the selected samples of mechanically activated complex CuPbZn sulphide concentrate (the values of  $A$  are calculated for the selected peaks at given  $d$  for mineral components of the concentrate)

$E_M$ ( $\text{kW h t}^{-1}$ )	$t_M$ (min)	$S_A \times 10^3$ ( $\text{m}^2 \text{kg}^{-1}$ )	CuFeS <sub>2</sub>	PbS	ZnS	FeS <sub>2</sub>
			$A$ (%)			
			$d = 0.3030 \text{ nm}$	$d = 0.3429 \text{ nm}$	$d = 0.3123 \text{ nm}$	$d = 0.2423 \text{ nm}$
82	10	2.77	36	27	38	66
404	15	4.48	49	59	40	64
303	37	5.33	60	78	59	72
808	60	6.91	53	47	48	71
524	64	8.59	43	35	51	70

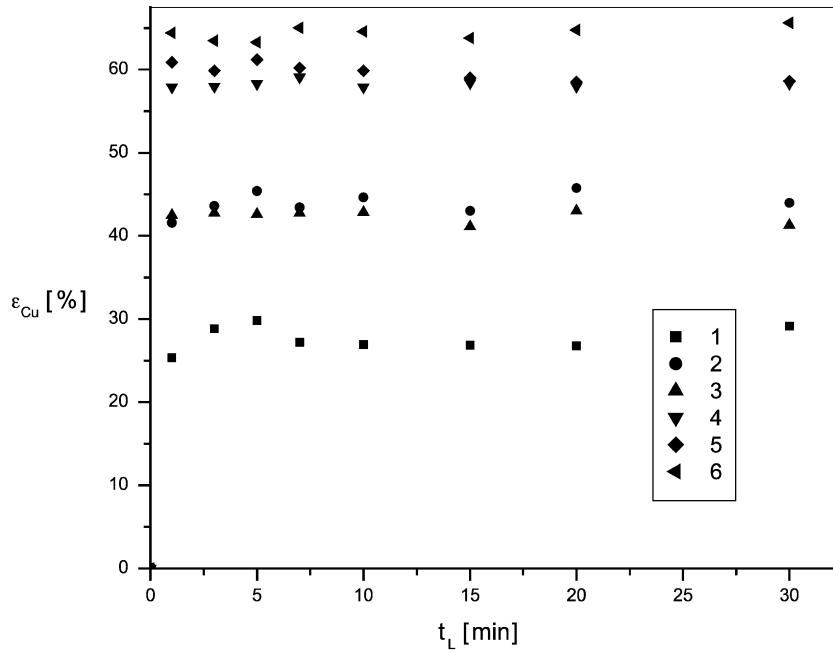


Fig. 2. Recovery of copper,  $\varepsilon_{\text{Cu}}$  vs. leaching time,  $t_L$ . Mechanical activation: (1) 0 min; (2) 10 min; (3) 15 min; (4) 37 min; (5) 60 min; (6) 64 min.

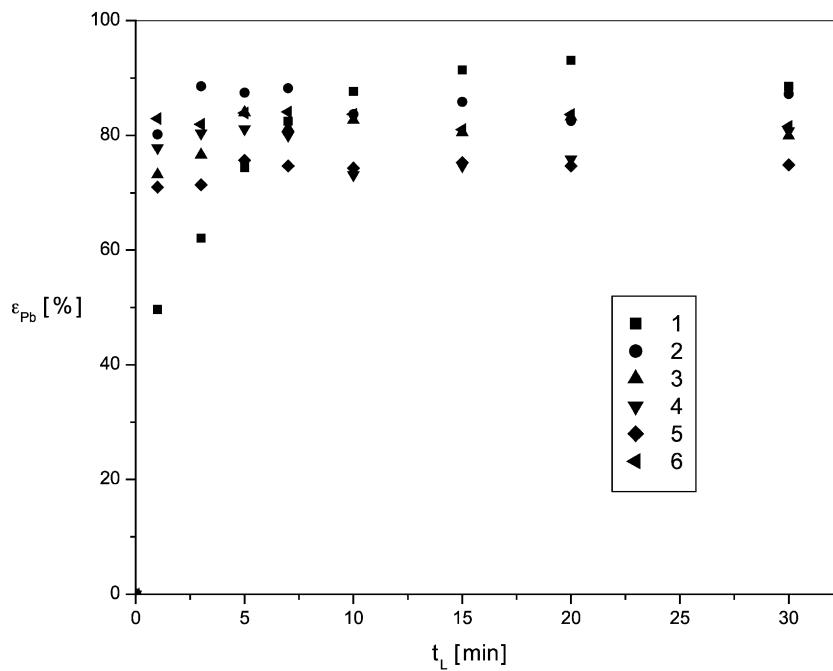


Fig. 3. Recovery of lead,  $\varepsilon_{\text{Pb}}$  vs. leaching time,  $t_L$ . Mechanical activation: (1) 0 min; (2) 10 min; (3) 15 min; (4) 37 min; (5) 60 min; (6) 64 min.

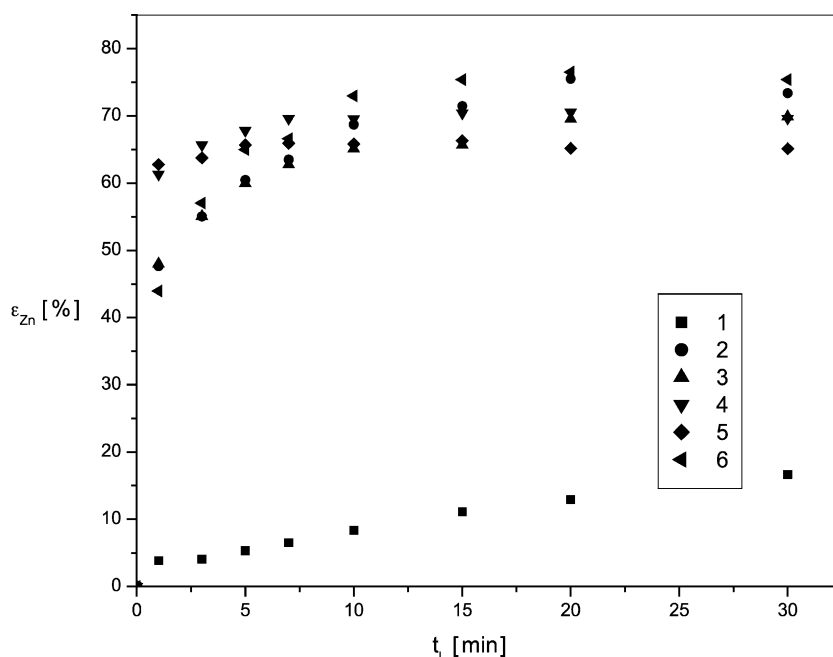


Fig. 4. Recovery of zinc,  $\varepsilon_{Zn}$  vs. leaching time,  $t_L$ . Mechanical activation: (1) 0 min; (2) 10 min; (3) 15 min; (4) 37 min; (5) 60 min; (6) 64 min.

time. It is clear that while the mechanical activation of the complex concentrate affects the leaching kinetics and increases the copper and zinc recoveries, in the case of lead, the mechanical activation affects only the leaching kinetics.

An earlier paper (Tkáčová and Baláž, 1988) derived the following Eq. (6) for the rate of chalcopryrite leaching as a function of structural sensitivity

$$k_0 = (1.387 + 1.281 \cdot S_A/X) \times 10^{-4} \quad (6)$$

where  $k_0$  is initial rate constant ( $s^{-1}$ ),  $S_A$  is specific surface area ( $m^2 \text{ kg}^{-1}$ ) and  $X$  is crystalline phase content of mechanically activated  $\text{CuFeS}_2$ . Eq. (6) can be generalized and the specific rate constant  $k_S$  can be written in the form of Eq. (7)

$$k_S = a + b/X \quad (7)$$

where  $a$  and  $b$  are parameters,  $X$  is crystalline phase content of sulphide, while  $1/X$  (the structural parameter) is calculated from data given in Table 1. The rate constants  $k_0$  are calculated from the leaching plots in

Figs. 2–4 according to Eq. (5) and the specific rate constant  $k_S$  can be defined in the form

$$k_S = k_0/S_A \quad (8)$$

We can investigate the structure sensitivity of chloride leaching of copper, lead and zinc if we eliminate the influence of initial surface area by introducing the specific rate constant as calculated by Eq. (7).

The experimental verification of Eq. (7) for the leaching of copper, lead and zinc from mechanically activated concentrate is shown in Fig. 5. Clearly, there is an equal influence of surface area and structural disordering on the leaching rate of copper, lead and zinc, thus providing evidence for the structural sensitivity of reaction (3). Based on the slopes in Fig. 5, the following sequence of structural sensitivity of individual minerals towards ferric chloride leaching can be obtained:

galena > chalcopryrite > sphalerite.

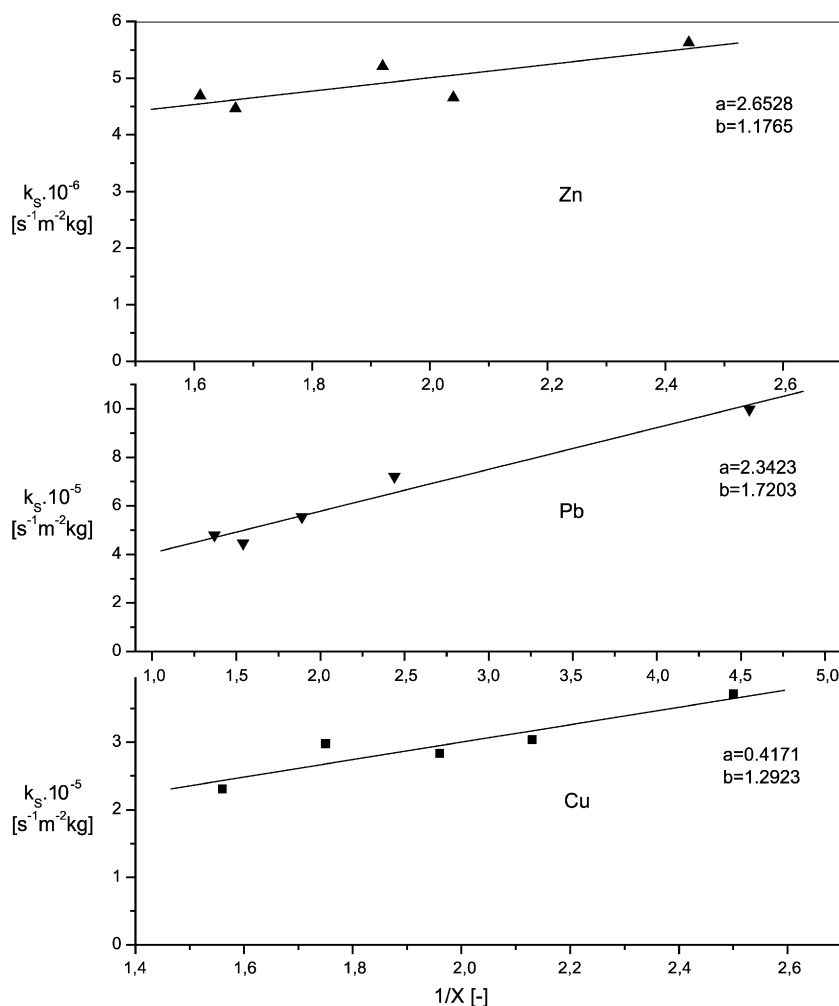


Fig. 5. Specific rate constant of metals (copper, lead and zinc) leaching,  $k_s$  vs. structure parameter,  $1/X$ .

### 3.2.2. Temperature sensitivity of Cu, Pb and Zn leaching

The optimum mechanically activated sample of the studied concentrate ( $S_A = 8.59 \times 10^3 \text{ m}^2 \text{ kg}^{-1}$  at  $E_M = 524 \text{ kW h t}^{-1}$ ) was used for investigating the temperature sensitivity of Cu, Pb and Zn leaching in the temperature region 323–363 K. The dependence of Cu, Pb and Zn recoveries on leaching time is plotted in Figs. 6–8. Clearly, increasing temperature accelerates the extraction of all metals into solution. The corresponding Arrhenius plots and activation energies are shown in Fig. 9.

In the case of copper leaching, the break in the Arrhenius plot (Fig. 9, line 1) indicates that the reaction mechanism changes with temperature. While for  $T = 323\text{--}343 \text{ K}$ , the activation energy  $E_A = 25 \text{ kJ mol}^{-1}$  is typical for mixed regime reactions governed by the rate diffusion control and surface chemical control (Habashi, 1978), at higher temperatures ( $T = 343\text{--}363 \text{ K}$ ) the rate-controlling step is diffusion.

A similar mechanism can be assumed for the zinc leaching (Fig. 9, line 3). The calculated value of  $E_A = 27 \text{ kJ mol}^{-1}$  in the lower temperature region indicates mixed regime and  $E_A = 10 \text{ kJ mol}^{-1}$  in the

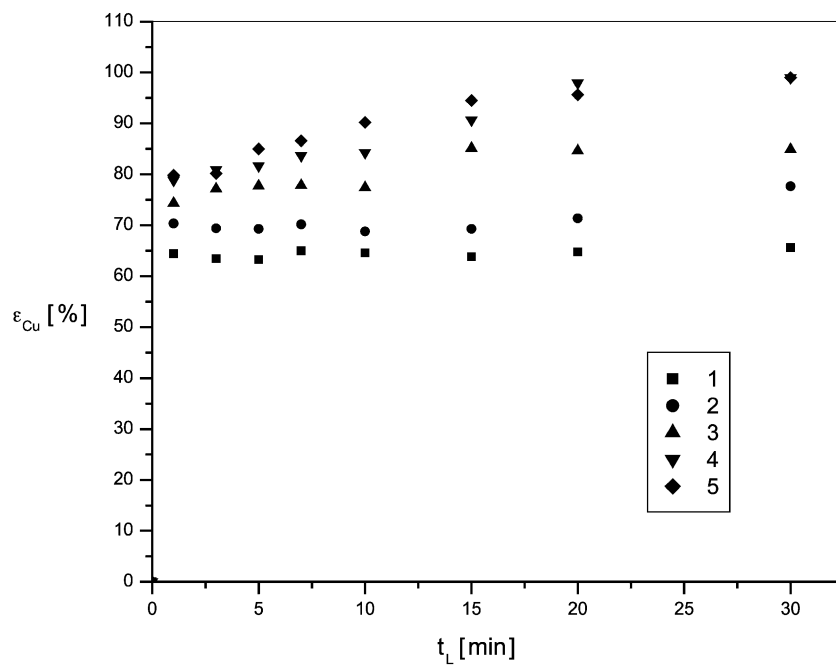


Fig. 6. Recovery of copper,  $\varepsilon_{\text{Cu}}$  vs. leaching time,  $t_L$ . Reaction temperature: (1) 323 K; (2) 333 K; (3) 343 K; (4) 353 K; (5) 363 K.

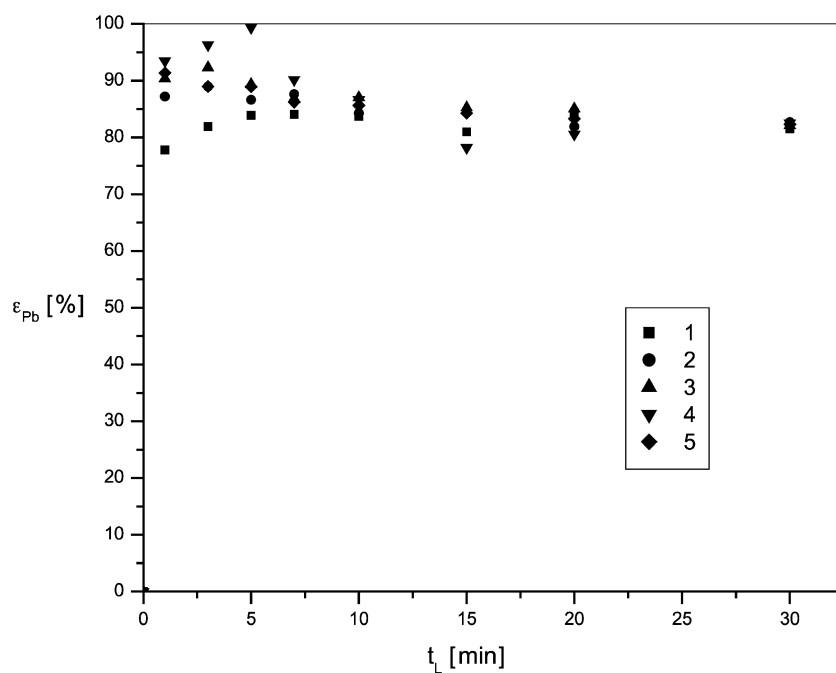


Fig. 7. Recovery of lead,  $\varepsilon_{\text{Pb}}$  vs. leaching time,  $t_L$ . Reaction temperature: (1) 323 K; (2) 333 K; (3) 343 K; (4) 353 K; (5) 363 K.



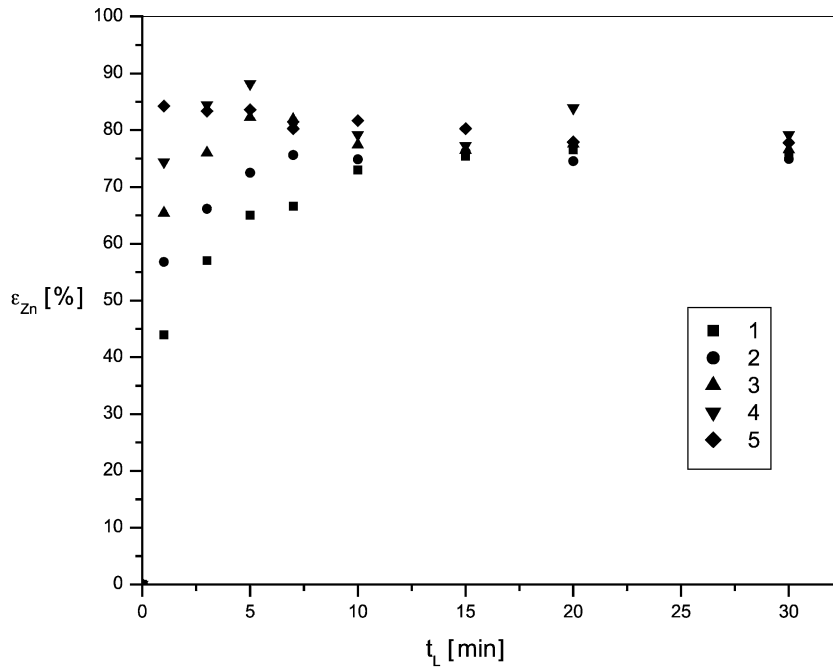


Fig. 8. Recovery of zinc,  $\epsilon_{Zn}$  vs. leaching time,  $t_L$ . Reaction temperature: (1) 323 K; (2) 333 K; (3) 343 K; (4) 353 K; (5) 363 K.

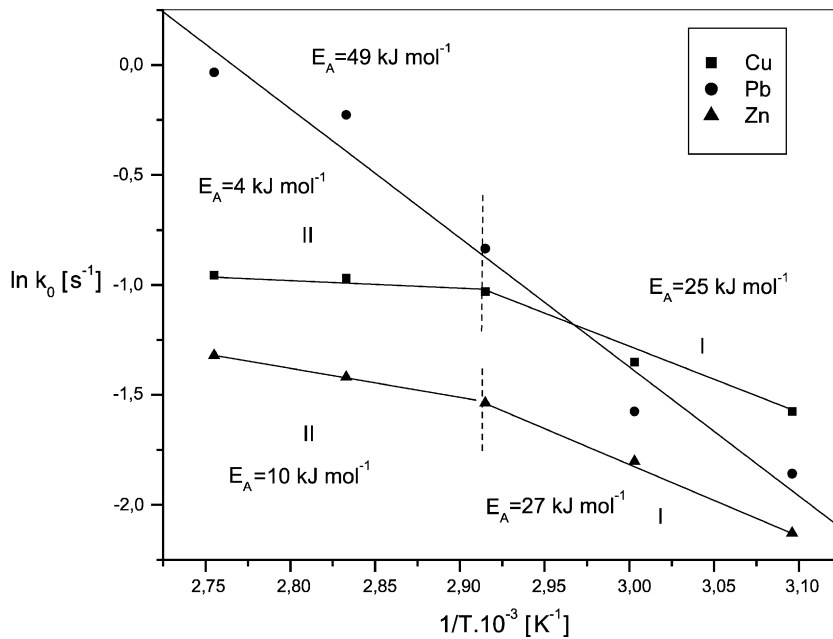


Fig. 9. Arrhenius plots for reactions of Cu, Pb and Zn leaching of optimum mechanically activated sample of complex CuPbZn sulphide concentrate.

higher temperature region is typical for diffusion control.

It can be observed in Fig. 9, line 2 that the plot for lead leaching is linear over the entire temperature range, indicating that the mechanism of lead leaching does not change. The activation energy of  $E_A = 49 \text{ kJ mol}^{-1}$  shows that the rate-determining step of the leaching reaction is chemical reaction.

These results are in agreement with literature data (Tkáčová and Baláž, 1988) that the rate of chemical reaction may be favourably affected by the structure-disordering, provided that the rate of chemical reaction is the rate-determining step. Both the amount of energy accumulated by defect structure and the type of defects involving this energy are of great importance from the point of view of kinetics (Avvakumov, 1986).

From the point of view of temperature sensitivity of copper, lead and zinc leaching for optimum mechanically activated sample, the following order can be suggested:

galena > chalcopyrite > sphalerite.

#### 4. Conclusions

- The structural disordering and increase in the surface area of the investigated concentrate manifests itself in the subsequent leaching behaviour of copper, lead and zinc.
- The mechanical activation affects the leaching kinetics and recoveries of copper and zinc, but only the kinetics of lead leaching.
- The leaching of copper, lead and zinc from the studied concentrate with ferric chloride solution are structure-sensitive reactions.
- The activation energy of copper, lead and zinc leaching with ferric chloride is sensitive to the temperature. Changes in reaction mechanism were observed, mainly at higher temperature, in the case of zinc and copper, with the rate-determining step changing from mixed regime to diffusion control.
- The Arrhenius plot for the lead leaching indicates that reaction mechanism does not change in the temperature interval under study. The value of calculated activation energy shows that the rate-determining step was chemical reaction.

- The suggested order of the structural sensitivity of minerals is the same as the suggested order of their temperature sensitivity.

- The investigation of chloride leaching of mechanically activated copper–lead–zinc concentrate showed that mechanism of ferric chloride leaching is not unambiguous and it can be changed depending on the reaction conditions.

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