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## Using the Results of Analyzing Solid Waste Products from Ore Processing Industry for Predicting the Chemical Composition of Drainage Water

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### Abstract

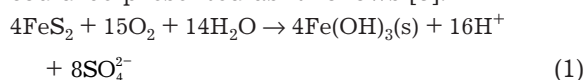
We have studied the substance of the stored waste products of sulphide ore dressing (clinker waste pile of the Belovo Zinc Plant JSC, tailing pits of the Komsomolsk Gold Recovery Plant and the Salagayev ravine of the Salair Integrated Ore-Dressing Plant JSC) and recrements planned for storing in the overburden of the ore bodies of the Vedugino gold deposit. Full-scale experiments have been conducted concerning the drainage of the substances of the objects by naturally occurring neutral water. The amount of sulphate ions passing into solution and metal ions has been estimated. Connection has been demonstrated between experimentally obtained physicochemical analytical data (pH values for substance paste, the acid producing and acid neutralizing potential) and chemical composition of drainage wastewaters (total ion concentration for main technogenous elements such as iron, zinc, copper, cadmium, lead).

**Key words:** waste products, oxidation of sulphides, acidic drainage wastewaters, acid producing potential, neutralizing potential, paste pH value

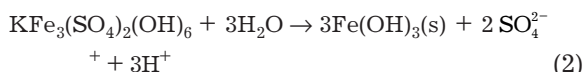
### INTRODUCTION

In ore processing, a lot of waste products is formed (overburden rock, ore-dressing tails, etc.), containing sulphide minerals whose oxidation results in the formation of acidic wastewaters with a high mineralization level [1, 2]. The studies concerning this problem have been started 40 years ago [3] and now they represent an important direction in ecological geochemistry and other sciences [4].

The total process of acid formation in the course of pyrite (FeS<sub>2</sub>) oxidation, which is one of the most widespread sulphide minerals, could be presented as it follows [3]:

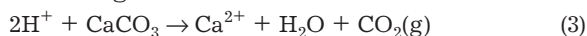


Besides sulphides, there is also another source of hydrogen ions. The substance of waste piles contains minerals producing acid under dissolution and hydrolysis [5]:



These <sup>+K</sup> compounds represent “keepers” of the stored acidity in the substance [5]. Sulphate salts of iron and aluminium concern (cokimbite ((Fe,Al)(SO<sub>4</sub>)<sub>3</sub> · 9H<sub>2</sub>O), copyapite (MgFe<sub>4</sub>(OH)<sub>2</sub>[SO<sub>4</sub>]<sub>6</sub> · 20H<sub>2</sub>O), jarosite (KFe<sub>3</sub>(SO<sub>4</sub>)<sub>2</sub>(OH)<sub>6</sub> etc.), first of all belong to the compounds under consideration.

The main minerals capable to neutralize efficiently acid, are presented by carbonates. They can interact with the acid produced according to the reaction



The neutralizing ability is mainly exhibited by calcite CaCO<sub>3</sub>; however other carbonates (magnesite MgCO<sub>3</sub>, rhodochrosite MnCO<sub>3</sub>, dolomite Ca<sub>0.5</sub>Mg<sub>0.5</sub>CO<sub>3</sub>) can also neutralize acid.

In parallel with monitoring of store facilities for sulphide-containing waste products, of great importance are the studies concerning the

situation resulting from their further interaction with naturally occurring waters as well as the development of methods for prognosis of acidic drainage [5, 6]. The level of potential hazard of waste piles for environment causes the choice of nature protection measures in the manipulation with waste products [7]. Studies on particular objects promotes both obtaining a more complete information concerning them, and using the dependences established for other man-caused systems.

The present work is aimed at: 1) establishing connection between experimentally obtained physicochemical analytical results concerning the substance waste products (substance paste pH values, acid producing and acid neutralizing potentials) and the chemical composition of drainage wastewaters (total ion concentration of main technogenous elements such as iron, zinc, copper, cadmium, lead); 2) potential hazard estimation for the substance of objects under investigation under interaction with naturally occurring waters.

#### OBJECTS UNDER INVESTIGATION

The analysis of the substance of sulphide waste products was carried out by the example of waste pile clinkers formed in the course of pyrometallurgical processing of zinc blende concentrate at the Belovo Zinc Plant (BZP) [8, 9];

waste products resulted from cyanidation at the Komsomolsk Gold Recovery Plant (KGRP) [10, 11]; waste products resulted from flotation stored in tailing pits of the Salagaevo ravine of the Salair Integrated Ore-Dressing Plant (SIODP) [12, 13] (Kemerovo Region); the samples of recrements planned for storing in the overburden of the ore bodies of the Vedugino gold deposit (Krasnoyarsk Territory) [14, 15]. The objects under investigation differ from each other in the content of sulphide and carbonate minerals (Table 1), in the age of storehouses, in the technological process of ore-dressing and, as a consequence, in the chemical composition of wastewaters and the solutions of sludge ponds (Table 2).

The age of waste products determines the duration of their interaction with atmospheric air, atmospheric precipitations and, hence, the oxidation level of sulphides [11]. For the objects under investigation, there is a considerable time scatter observed in this parameter: from 20 and 10 years since the moment of waste pile formation end (for waste products from the BZP and the KGRP, respectively) up to the working tailing pit of the Salagayevo ravine and recrements of the Vedugino deposit just planned for warehousing.

In order to estimate the composition of waste pile drainage waters of the Vedugino deposit we have used kinetic experimental data. According to the results obtained, samples were divided into two groups (see Table 2).

TABLE 1

Comparative characteristics for objects under investigation concerning the qualitative and quantitative content of minerals

| Minerals under investigation   | Content, % |
|--|------------|
| <i>Clinkers from the BZP</i>   |            |
| Main sulphides (zinc blende ZnS, chalcopyrite CuFeS <sub>2</sub> )                 | ~1 %       |
| Carbonates   | Traces     |
| <i>Waste products from the KGRP</i>  |            |
| Main sulphides (pyrite FeS <sub>2</sub> , arsenopyrite FeAsS)                      | ~1.5 %     |
| Carbonates (calcite CaCO <sub>3</sub> )  | ~5 %       |
| <i>Tailing pit from the Salagayevo ravine</i>                                      |            |
| Main sulphides (pyrite, zinc blende, halenit PbS)                                  | ~3 %       |
| Carbonates (calcite CaCO <sub>3</sub> )  | ~7 %       |
| <i>Recrements from the Vedugino gold deposit</i>                                   |            |
| Basic sulphides (pyrite, pyrrhotine Fe <sub>7</sub> S <sub>8</sub> , arsenopyrite) | 0.02–2.6 % |
| Carbonates (calcite CaCO <sub>3</sub> , dolomite Mg(Ca)CO <sub>3</sub> )           | 0.01–9 %   |

Note. Here and in Tables 2, 3: BZP – Belovo Zinc Plant, KGRP – Komsomolsk Gold Recovery Plant.

TABLE 2

Comparative characteristics for the composition of drainage wastewaters and sludge pond solutions

| Object under investigation   | Kurlov's formula  |
|--|---|
| Waste pile drainage of the BZP                                     | $M_{2.7} \frac{\text{SO}_4^{2-} 91 \text{Cl}^- 8.6}{\text{Ca}^{2+} 30 \text{Na}^+ 23 \text{Mg}^{2+} 22 \text{Cu}^{2+} 11 \text{Zn}^{2+} 11} \text{pH } 5.8$                                 |
| Sludge pond of the KGRP tailing pit                                | $M_{0.5} \frac{\text{SO}_4^{2-} 84 \text{HCO}_3^- 7.7 \text{NO}_3^- 3.8 \text{Cl}^- 3.0 \text{F}^- 1.5}{\text{Na}^+ 41 \text{Mg}^{2+} 27 \text{Ca}^{2+} 27 \text{K}^+ 4.7} \text{pH } 8.15$ |
| Sludge pond tailing pit of the Salagayevov ravine                  | $M_{3.8} \frac{\text{SO}_4^{2-} 96 \text{HCO}_3^- 2.2 \text{Cl}^- 1.4}{\text{Ca}^{2+} 60 \text{Mg}^{2+} 33 \text{Na}^+ 5.6} \text{pH } 7.75$  |
| Model drainage solutions of the recrements of the Vedugino deposit | $M_{0.7} \frac{\text{SO}_4^{2-} 70 \text{HCO}_3^- 24 \text{Cl}^- 3}{\text{Ca}^{2+} 58 \text{Mg}^{2+} 27 \text{K}^+ 14 \text{Na}^+ 1.5} \text{pH } 7.61$                                     |
|  | $M_{1.9} \frac{\text{SO}_4^{2-} 98 \text{HCO}_3^- 1.4}{\text{Fe}^{2+} 63 \text{Ca}^{2+} 12 \text{Mn}^{2+} 9.4 \text{Mg} 8.0 \text{K}^+ 6.2} \text{pH } 4.24$                                |

Note. For designations see Table 1.

## EXPERIMENTAL

The samples of waste pile substances from the BZP, waste products from tailing pits of the KGRP and the Salagayevov ravine were taken according to an identical scheme. In studying the substance of potential recrements resulting from the Vedugino deposit we used four representative samples reflecting the geochemical structure of rocks (Table 3, samples Nos. 1–4) given by the Amikan Co., Ltd. (Krasnoyarsk) were used.

In our work we used various methods for the investigation of rock acid productivity those are used both by foreign [5, 16, 17], and Russian researchers [18, 19].

For all the samples we determined paste pH values, acid producing and acid neutralizing potentials (AP and NP, respectively). The flow-through experiments with the waste pile substances of the BZP, the waste products resulting from the KGRP and the Salagayevov ravine were carried out employing a unified technique.

### Determining the paste pH values

On the basis of data concerning the acidity of paste one could estimate the ability of the substance under investigation to produce acid as well as to neutralize acid in the course of interaction with water. "Paste" is meant to be a

highly concentrated suspension consisting of fine-dispersed sample and distilled water. Preparing the pastes and measuring pH values were carried out employing a technique described in [16]. All the measurements concerning pH values were performed by means of "Ekspert-001-3" with the use of an ESK-10601/7 combined glass electrode.

### Analysis of acid-base ratio

The analysis of the acid-base ratio used for the estimation of the potential environment of drainage includes the determination of AP and NP and their comparison among themselves.

The acid producing potential represents the estimation of the maximal acid formation level in the substance of waste products. It is supposed, that the process of oxidation occurs according to reaction (1), whereas the neutralization process occurs *via* reaction (3). The value of AP is determined from the content of sulphur in the form of sulphides [17]. The content of sulphide sulphur ( $S_s$ ) was determined by a standard technique [20]. The AP value can be expressed as the amount of  $\text{CaCO}_3$  (in kg/t) necessary for  $\text{H}^+$  neutralization [16, 17]:

$$\text{AP} = S_s(\%) \times 31.25 \quad (4)$$

The acid neutralizing potential allows one to estimate the ability of rocks to function as neutralizing barriers. The technique for NP esti-

mation is described in [16]; it consists in the determination of the content of neutralizing minerals in substance employing the method of back acid-base titration. The value of NP is determined by the amount of  $\text{CaCO}_3$  (in kg/t); this value always exceeds or corresponds to the content of calcite in substance.

When  $\text{NP} < \text{AP}$ ,  $\text{NP}/\text{AP} < 1$ , the substance interacting with oxygen forms acid and when  $\text{NP} > \text{AP}$ ,  $\text{NP}/\text{AP} > 1$  all the formed acid is neutralized, thus the pH value for drainage wastewaters is be neutral [4].

### Flow-through experiments

Carrying out full-scale experiments on draining waste products by natural water is considered a convenient approach in determining the scale of washing-off the elements from waste piles [19]. In flow-through experiments we simulated the interaction of waste piles with rain water. The duration of the experiment amounted to about 24 h, which excluded essential oxidation of sulphides within so short time interval. Hence, all the acid washed off was produced in substance earlier. The technique of the flow-through experiment is similar to that described in [19]. For carrying out the experiments, we took substances of waste products from the BZP, the KGRP and the SIODP, 100 g each. The flow rate of water amounted to 1.1–1.5 L/h. The concentration of metal ions in water samples was determined employing the method of atomic emission spectrometry with inductively coupled plasma at the Analytical Centre of the Institute

of Geology and Mineralogy, SB RAS (Novosibirsk). The measurement error amounted to  $\pm 10\%$ . The determination of the content of sulphate ions was carried out employing a turbidimetric method according a standard technique (RD 52.24.405–95). The measurement error amounted to  $\pm 10\%$ .

### Kinetic experiments

Kinetic experiments adequately modelling the interaction of rock with water flows [21] were carried out with the substance of the Vedugino deposit recrements. The conditions of the experiments were aimed at the acceleration of sulphide oxidation in order to extrapolate the process for long term. In the experiments, we used columns 10 cm in height and 6.5 cm in diameter wherein we loaded samples of 0.3 kg in weight (the particle size being  $< 0.25$  mm). Within 20 weeks, samples were every day blown with air (0.01 mL/min); every 6 days the samples were washed with distilled water (50 mL) and then the pH value of drain medium and the content of macro- and microelements therein.

## RESULTS AND DISCUSSION

### Analysis results for the substance of waste products from the BZP

The data concerning the paste pH value for clinkers (see Table 3) indicate an “accumulated” acidity in substance, which is in a good agreement coordinated with the results of the flow-through experiment in whose beginning out-

TABLE 3

Results of analysis for paste pH value, acid producing (AP) and acid neutralizing (NP) potentials, kg of  $\text{CaCO}_3$  per 1 t of rock

| Object                                | pH   | AP | NP  | NP/AP |
|---------------------------------------|------|----|-----|-------|
| Waste products of the BZP             | 2.41 | 31 | 0   | 0.0   |
| Tailing pit of the KGRP               | 8.09 | 43 | 87  | 2.0   |
| Tailing pit of the Salagayevov ravine | 7.92 | 94 | 76  | 0.81  |
| Recrements of the Vedugino deposit:   |      |    |     |       |
| Sample No. 1                          | 8.20 | 18 | 27  | 1.5   |
| Sample No. 2                          | 8.45 | 33 | 63  | 1.9   |
| Sample No. 3                          | 5.30 | 69 | 3.4 | 0.049 |
| Sample No. 4                          | 6.60 | 44 | 8.8 | 0.20  |

Note. For designations see Table 1.

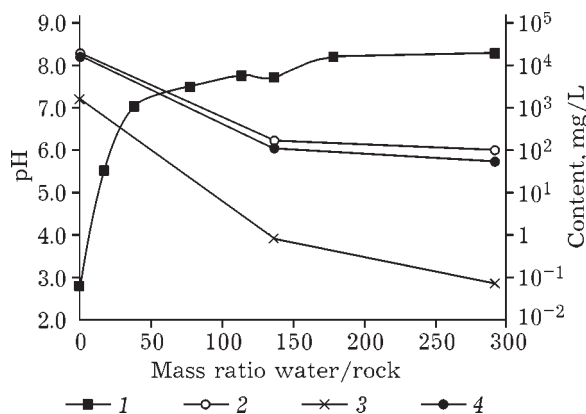


Fig. 1. Change in the chemical composition of out-flowing waters in the flow-through experiment with the substance of waste products from the BZP: 1 – pH value; 2 – mineralization level, mg/L; 3, 4 – the content of iron ions (3) and of sulphate ions (4).

flowing solutions exhibit an acidic pH value (Fig. 1). For the substance of waste piles  $NP = 0$ , which could be caused by a complete exhaustion of neutralizing minerals because of intense acid formation, whereas AP value is low (see Table 3). Combustion processes in waste piles accelerate the oxidation of sulphides and the formation of acid [8], and nowadays the main source of acid is presented by sulphate salts ( $KFe_3(SO_4)_2(OH)_6$ ,  $Zn_3Al[SO_4]_9 \cdot 36H_2O$  [9] *etc.*) rather than the processes of sulphide oxidation. The dissolution of sulphates results in a decrease of the pH values for drainage solutions according to reaction (2) as well as in occurring high concentration of sulphate ions in wastewater.

In the course of the flow-through experiment the acid-base status of the medium of solutions changes from acidic value (pH 2.80) to alkaline (pH 8.28), whereas the general mineralization decreases more than by two orders of magnitude (from 19 to 0.1 g/L) (see Fig. 1). The content of  $SO_4^{2-}$  ions is reduced from 17 to 0.05 g/L. The content of iron and hydrogen ions varies in a similar manner (see Fig. 1). Basing on this fact one could draw a conclusion that the ions of iron,  $H^+$  and  $SO_4^{2-}$  exist in waste piles in the form of the same soluble compounds.

In the interaction of clinkers with naturally occurring waters, the formation of acidic solutions is observed up to the ratio water : rock = 50 : 1, and 1 kg of waste products results in about 50 g of sulphate ions and 5 g iron ions passing into solution.

The results of flow-through experiment (see Fig. 1) indicate that for washing out the acid the ratio water: rock should amount to ~50 : 1 (according to other data ~170 : 1 [22]). Provided mid-annual level of atmospheric precipitations in the Kemerovo Region approximately equal to 500 mm [23] and the areas occupied by waste piles (~4 ha), up to 20 million litres of drainage wastewaters with  $pH \leq 3.0$  could be formed within a year; in this case acid would be washed only from 400 t of waste piles (with wastes stored about 900 thousand ton).

#### Analysis results for the substance of waste products from the KGRP

The data concerning the paste pH value of waste products from the KGRP (see Table 3) indicate the absence of compounds producing acid, which is in accordance with the results of the flow-through experiment (Fig. 2). Besides carbonates, the reagents used in the technological process of gold extraction that occurs with the use of NaOH according to production schedules contribute the NP value. For the waste products of KGRP, an important role is played not only by the ratio between acid producing and acid neutralizing minerals, but also by previous processing by chemical reagents that influences the acidity of the environment of waste products in the course of their storage.

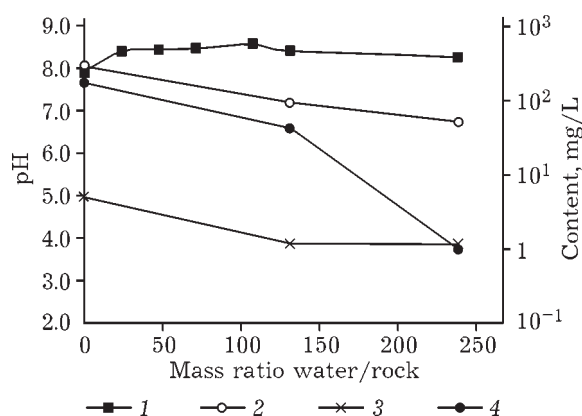


Fig. 2. Change in the chemical composition of out-flowing waters in the flow-through experiment with the substance of waste products from the KGRP: 1 – pH value; 2 – mineralization level, mg/L; 3, 4 – the content of potassium ions (3) and sulphate ions (4).

In the flow-through experiment with the substance from the KGRP storage the value of solution pH almost does not change, whereas the mineralization level is reduced (see Fig. 2). The content of potassium and sulphate ions decreases twofold. In the interaction between the waste products and naturally occurring water, 1 kg of waste products results in 1 g of sulphate ions passing into solution.

#### *Analysis results for the substance of tailing pits from the Salagayevov ravine*

For the substance of tailing pits at a high level of AP (due to non-oxidized sulphides) the acid-base status of the paste was almost neutral (pH 6–8). High NP value could be caused by a rather high content of carbonates.

The results of flow-through experiment (Fig. 3) are similar to the results of the experiment with the substance of the KGRP tailing pit. As water passing through, the value of solution pH changes poorly (pH 7.7–8.4) (see Fig. 3). Iron is washed out of the substance uniformly, and its content in solutions decreases by 30 times (from 1.92 to 0.06 mg/L). The majority of elements (Ca, Mg, Na, Mn, Zn, and  $\text{SO}_4^{2-}$ ) is to a maximum extent washed out not in the beginning of the experiment, but with some delay, which is reflected in the total mineralization level (see Fig. 3).

Using the general development laws for tailing pits [12], one could assume that in the

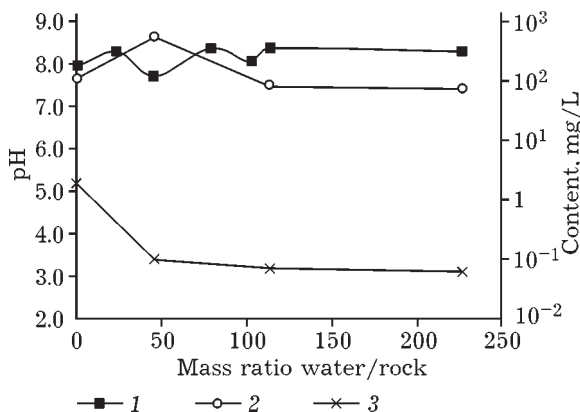


Fig. 3. Change in the chemical composition of out-flowing waters in the flow-through experiment with the substance from the tailing pit of the Salagayevov ravine: 1 – pH value; 2 – mineralization level, mg/L; 3 – the content of iron ions.

future, after the storage conservation and the decrease of the watering level, the oxidation of sulphides would occur in a more intensive manner. After the exhaustion of neutralizing minerals, the drainage wastewaters would exhibit acidic pH value.

#### *Analysis for the substance of potential recrements from the Vedugino deposit*

The samples of substance of this object differ from the other by the fact that they do not contain any reagents used for ore-dressing those can influence the pH value of drainage waters. The acid-base status for the paste of the majority of samples (see Table 3) is close to neutral pH value, as well as the acid-base status for solution media during the first week in the kinetic experiments, which could be caused by a low oxidation level of sulphides in the substance. For samples Nos. 3, 4 AP > NP (see Table 3).

In the kinetic experiments, not only the acidity accumulated in substance was washed out, but also the acidity formed during the experiment in the course of sulphide oxidation. An additional aeration and a significant grinding level of the substances accelerated the reactions. In this connection, for the kinetic experiments one could note a decrease in solution pH values (Fig. 4, curves 3, 4), as against the flow-through experiments where the value of pH could increase only up to a background value (the formation of new portions of acid was impossible).

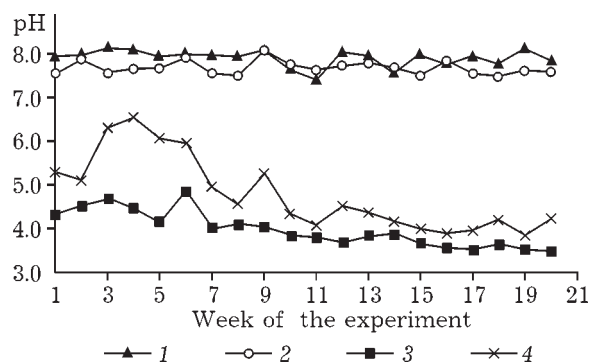


Fig. 4. pH values of out-flowing solutions in the kinetic experiment with recrements from the Vedugino gold deposit: 1–4 – samples Nos. 1–4, respectively.

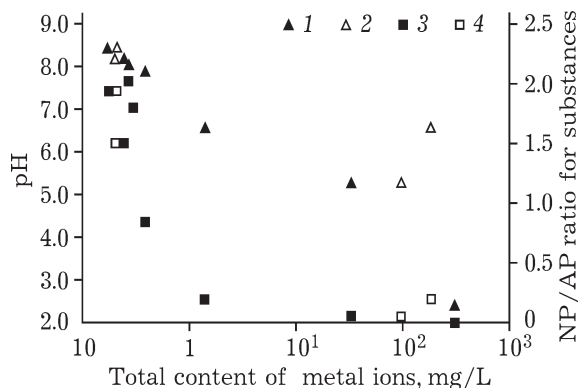


Fig. 5. Total content of metal ions (Zn, Cu, Pb, Cd, Fe) in drainage wastewaters, an aqueous part of sludge pond or in the solutions in kinetic experiments depending on the paste pH values (1, 2) and NP/AP ratio for the substance of the objects under investigation (3, 4) in the beginning (1, 3) and in the end (2, 4) of the kinetic experiment.

According to the results of kinetic experiments the samples could be divided into two groups those differing in pH value of solutions out-flowing from columns (see Fig. 4). The acid-base status of the solution media of the first group (curves 1, 2) amounted to pH 7.0–8.5 in the course of the experiments, which could be caused by NP excess comparing to AP. For the second group (curves 3, 4) the acid-base status of the solution media has changed by the end of the experiments to gain pH 3–5. For the samples of this group, a considerable AP excess comparing to NP (see Table 3) is observed, which just explains the reduction of solution pH values in kinetic experiments.

## CONCLUSIONS

Different mineral composition, the technologies of extraction processes for components required, conditions for storing the substance of waste piles under investigation determine different ratio values between AP and NP and paste pH values. There is an interrelation observed between AP, NP, paste pH value and the content of metal ions in draining flows.

Figure 5 demonstrates data of two types. For the first type of the data we measured pH paste values, the values of AP and NP for samples, and then almost immediately we determined the content of metal ions in the solutions obtained by water interaction with the

substance under investigation in the beginning of kinetic experiments (for samples taken from the Vedugino deposit), and in the solutions taken from drainage wastewaters resulting from waste piles of the Belovo Zinc Plant and aqueous part of sludge ponds (the KGRP and Salagayevov tailing pits). For the second type of the data, we measured paste pH values, the values of AP and NP for samples and investigated the solutions obtained at the end of the kinetic experiments whereat sulphides in substance were oxidized by atmospheric oxygen.

In the first case, the time interval between the determination of the paste pH value, AP, NP and the formation of drainage solutions under investigation is very short, and the transformation of the substance is insignificant; in the second case the time interval is significant, which promotes a considerable transformation of the substance. Hence, the interrelations established in the second case, could be used to make predictions for a longer time interval.

The results obtained indicate a wide scatter observed for AP and NP values, as well as for their ratio (NP : AP = 0–2), the acid-base status of paste medium corresponding to pH 2.4–8.5. The differences in physicochemical parameters of the substance result in the fact that the composition of drainage flows from different storages differ from each other to a considerable extent. The total content of ions of main technogenous elements (Zn, Cu, Cd, Fe, Pb) differs within three order of magnitude (from 0.2 to 300 mg/L). The data concerning the content of metal ions in the solution correlate well with the data concerning the paste pH value.

When the acidity level of the paste corresponds to subalkaline medium, the total content of metal ions in wastewaters is less than 1 mg/L (waste products of the KGRP, the substance from the tailing pit of the Salagayevov ravine). A low paste pH value indicates that the total content of metal ions in drainage wastewaters exceeds 100 mg/L (waste pile drainage from the BZP). The content of metal ions is also depending on the NP/AP ratio: the less is NP/AP ratio, the higher is the content of metal ions, and on the contrary.

The results of kinetic experiments allow us to draw the following conclusions. First, for the samples with high NP/AP value and alkaline

paste pH value, the change of the total content of metal ions from the beginning to the end of the experiment is insignificant (from 0.17 to 0.21 mg/L for pH 8.45 and NP/AP = 1.93; from 0.25 to 0.20 mg/L for pH 8.20 and NP/AP = 1.50). On the contrary, for the samples with a low NP/AP ratio and acidic paste pH value, the change of the content of metal ions in the course of the experiment was significant (from 1.4 to 182 mg/L for pH 6.60 and NP/AP = 0.20; from 33 to 96 mg/L for pH 5.30 and NP/AP = 0.05). In the samples with a low NP/AP ratio, much more H<sup>+</sup> ions are formed, than it is in the samples with a high NP/AP ratio, therefore much more metal ions can pass into solution. Second, the value of NP/AP correlates well with the content of metal ions in the solution at the end of kinetic experiment, which allows one to use data concerning the NP/AP ratio for long-term forecasting.

Basing on the regularities obtained one can make predictions for the drainage drain from the waste piles of the BZP. For the substance of clinkers NP/AP = 0 (see Table 3), therefore the drainage after a long time (years) would exhibit acidic pH value, whereas the total content of metal ions would be high (~10<sup>2</sup> mg/L). For the substance of the Komsomolsk tailing pit NP/AP = 2.0 (see Table 3), therefore sludge pond waters after a long time (years) would exhibit neutral pH value, whereas the total content of metal ions would be low (<1 mg/L). For the substance of the Salagayevo tailing pit NP/AP = 0.81 (see Table 3), hence, sludge pond waters after a long time (years) would exhibit acidic pH value, whereas the total content of metal ions would be high (>10 mg/L).

The prognostic estimation of the total content of elements (Zn, Cu, Cd, Fe, Pb) in drainage wastewaters resulting from ore-processing industry waste products for the near future (days, weeks) one can perform basing on the paste medium pH value, whereas a long-term prognosis (months, years) could be made basing on NP/AP ratio.

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