Investigation of Acid-Base and Redox Regimes of Tin Ores Enrichment Wastes in Primorye and Priamurye

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Abstract. The elemental and mineral composition of enrichment and processing wastes of tin ore and possible chemical processes occurring in these wastes are analyzed. The article discusses the manifestations of acid-base and redox potentials of waste enrichment of tin ores of Primorye and Priamurye. As a result of these processes, the composition and chemical properties of the studied objects are constantly changing in time and space, so it is important to study their acid-base and redox potentials. Typically, the state of the waste deposits is estimated using indicators \(E_h\) and \(pH\), characterizing the active manifestation of the acid-base and redox properties; the hidden possibilities of the studied objects are not taken into account and are not evaluated. To evaluate these possibilities, the authors determined such indicators as total acidity and alkalinity, the number of oxidation and reduction centers (mmole equivalent per kilogram of waste). Analysis of results showed that waste processing tin ore can at the expense of the latent acidity to constantly maintain an acid-base mode, but at the expense of reduction centers to support \(pH\) less 28, i.e. to create a shortage of oxygen. Thus, when developing a system of measures to prevent the negative impact of tin ore processing wastes, it is necessary to take into account the facts of "inexhaustibility" of reserves of constantly generating acidic and reducing centers of these wastes.

1. Introduction

The study of the state of objects of technogenic origin, trends in change of this state in time and space involves the measurement and analysis of indicators that affect the quality of the environment. Such important indicators are acid-base and redox regimes of technogenic object.

The subject of this study – the sands of tailings waste of tin ore processing former mining and processing plants in the Far Eastern region of Russia. The industrial era left a lot such of tailings that not were exposed recultivation [5, 13, 15]. At present, due to the continuous negative impact on the environment and health of the population of the nearby human settlements [1-3, 16, 17] the problem of elimination of this accumulated environmental damage [4] has become urgent. In order to develop a program of economical, environmentally friendly and rational technology measures to eliminate and compensate for the negative impact of tailings on the environment, it is necessary to thoroughly study their properties, status and trends in change of this state. The leading scientists of the Far East are actively studying the physico-chemical properties of tin ores processing waste, including acid-base and oxidation-reduction regimes of the tailings dumps of Primorye and Priamurye [4-8, 13, 18-20].

To this end, as a rule, the indicators of active \(pH\) acidity and oxidation potential of \(E_h\) are determined. The authors [4-8, 13, 15] associate these indicators with the content of the sulfide...
component of the tin ore processing waste. We analysed the data they had received and, on that basis, recalculated the sulphide component to mol sulfur per kg of waste. The obtained data are presented in the form of graphs (Figure 1, 2).

**Figure 1.** The intervals of change of Eh of interstitial water in tailings Primorye and Priamurye, depending on the content of sulphide sulphur.

**Figure 2.** The intervals of change of pH of interstitial water in tailings Primorye and the Amur region, depending on the content of sulphide sulphur.

The analysis of the data presented in Fig. 1 shows significant fluctuations in pH values for different waste stores. These fluctuations may be due to various factors, such as: the mineral composition of the waste; temperature; climate; the chemical nature of the components of minerals; uniform distribution and quantity of silicate and sulphide constituents; qualitative and quantitative ion-molecular composition of pore waters, and these waters can exhibit high acidity (pH < 7) and alkalinity (pH > 7). According to the authors [5,15] the most active components of waste are sulphide minerals and they affect the exponents of Eh-pH. However, the authors clearly did not present the form of this dependence.

Figure 1 shows the dependence of the minimum and maximum values of pH for the various contents of the sulphide component (mole of sulfur per kg of waste) and quite convincingly indicates that the range of fluctuations in pH increases with the content of sulfide sulfur. At the same time, for acidic solutions, the pH decreases with the an increase in the sulfide component, and, on the contrary, increases for the basic media. It can be assumed that the acidic or basic mediums is formed as a result of various processes.

\[
\text{MeS} \leftrightarrow \text{Me}^{2+} + \text{S}^{2-} \quad \text{(the quantity of ions will depend on the solubility of the sulphide)}.
\]
Ions $\text{Me}^{2+}$ и $\text{S}^{2-}$ – residues of weak electrolytes and undergo hydrolysis according to the scheme: 
$$
\text{Me}^{2+} + \text{H}_2\text{O} \leftrightarrow \text{H}^+ + \text{MeOH}^+ \\
\text{S}^{2-} + \text{H}_2\text{O} \leftrightarrow \text{OH}^- + \text{HS}^- + \text{H}_2\text{O} \leftrightarrow 2\text{H}^+ + \text{H}_2\text{S}^\uparrow
$$

At the same time, oxidation of sulfide sulfur by air oxygen can proceed according to the following schemes:
$$
\text{S}^{2-} + 2\text{O}_2 \rightarrow \text{SO}_4^{2-} \quad (\text{pH} > 7) \\
2\text{S}^{2-} + 2\text{O}_2 + 2\text{H}_2\text{O} \rightarrow 2\text{S}^\uparrow + 4\text{OH}^- \quad (\text{pH} > 7) \\
\text{H}_2\text{S} + 2\text{O}_2 \rightarrow 2\text{H}^+ + \text{SO}_4^{2-} \quad (\text{pH} < 7)
$$

The formed sulfate ion can precipitate poorly soluble $\text{PbSO}_4$, $\text{BaSO}_4$, $\text{CaSO}_4$, thereby increasing the solubility of the sulphide constituent of the waste. Acidification of pore solutions also promotes the dissolution of sulphide minerals, as a result of which the concentration of the sulfide-ion in the solution increases, since not all sulphide sulfur will volatilize as hydrogen sulphide. The variety and interdependence of the acid-base and redox interactions of the components of the tin ores dressing waste leads to an increase in spread of values the pH and Eh in the case of an increase in the amount of the sulphide component. In addition, a significant effect on oxidation-reduction processes can be stipulated by oxygen deficiency in the thickness of the waste, thereby information is needed on the degree of compactness of the waste and their granulometric composition. The influence of each of the properties of waste and processes on the formation of acid-base and oxidation-reduction regimes should be investigated and considered.

According to the data of the authors [5,15], the anionic composition of the pore waters of the tailings is represented by sulfates, carbonates and hydrocarbonates, hydrosilicates, chlorides, partially arsenates, plumbites, zincates, aluminates, hydroxo groups. Cationic composition, in addition to cations of non-ferrous metals, is represented by cations of potassium, sodium, magnesium, calcium, hydrogen.

These facts confirm the active participation of the silicate component and carbonization processes in the formation of pH regimes. Anions $\text{S}^{2-}$ and $\text{HS}^-$ were not found in the pore waters. Apparently part of the sulfide sulfur is oxidized to the sulfate-ion. All researchers note the strong smell of hydrogen sulfide in the area of waste storage facilities for processing tin ores. This indicates that part of the sulfide sulfur as a result of hydrolysis pick out into the atmosphere in the form of hydrogen sulfide, which leads to an increase in the concentration of non-ferrous metals in solution. The hydrolysis of these cations results in a decrease in the pH-value, and, to the formation of an acid regime.

We noted in the research process the strong adhesion properties of the highly dispersed fraction of waste products of tin processing relative to paper. After contact of white paper with waste, a yellowish coating is formed on it, typical for fine-dispersed elemental sulfur (sulfuric color). The analysis of the investigated properties of waste products of processing of tin ores and possible processes in them allows suppose the following:

1. For the technogenic objects under study, a complex of various processes is characteristic, the prevalence of each of them can lead to different pH- and redox-regimes.

$$
\begin{align*}
5\text{FeS}_2_{(pyrite)} + 6\text{H}_2\text{O} + 11\text{O}_2 & = [\text{Fe}(\text{H}_2\text{O})_6]^{2+} + \text{S}_2 + 8\text{SO}_4^{2-} + 2\text{Fe}_2\text{O}_3 \\
2\text{FeS}_2_{(pyrite)} + 2\text{H}_2\text{O}_{(aq)} + 2\text{CO}_2_{(g)} & = 2\text{FeOH}^+ + 2\text{HCO}_3^- + 2\text{H}_2\text{S(g)} + 2\text{S}_{(solid)}, \Delta G_r^0 = -65,5 \text{ kJ} \\
\text{FeS}_2_{(pyrite)} + 2\text{O}_2(g) & = \text{Fe}^{2+} + \text{SO}_4^{2-} + \text{S}_{(solid)}, \Delta G_r^0 = -662,83 \text{kJ} \\
4\text{FeS}_2_{(pyrite)} + 2\text{H}_2\text{O}_{(aq)} + 15\text{O}_2(g) & = 4\text{Fe}^{3+} + 8\text{SO}_4^{2-} + 4\text{H}^+, \Delta G_r^0 = -1213 \text{ kJ} \\
\text{FeS}_2_{(pyrite)} + 3\text{O}_2(g) & = \text{Fe}^{3+} + \text{SO}_4^{2-} + \text{SO}_2_{(g)}, \Delta G_r^0 = 963,04 \text{ kJ} \\
2\text{FeS}_2_{(pyrite)} + 7\text{O}_2(g) & = \text{Fe}^{3+} + 3\text{SO}_4^{2-} + \text{SO}_2_{(g)}, \Delta G_r^0 = -1110 \text{ kJ} \\
2\text{SO}_2 + 2\text{H}_2\text{O} + \text{O}_2 & = 4\text{H}^+ + \text{SO}_4^{2-} \quad \text{(waste acts as a catalyst), } \Delta G_r^0 = -413,10 \text{ kJ} \\
\text{CuFeS}_2_{(chalcopyrite)} + 4\text{O}_2 & = \text{CuSO}_4 + \text{FeSO}_4, \Delta G_r^0 = -327,20 \text{ kJ} \\
\text{CuFeS}_2_{(chalcopyrite)} + 2\text{H}_2\text{SO}_4_{(water)} = \text{CuSO}_4_{(water)} + \text{FeSO}_4_{(water)} + 2\text{H}_2\text{S(g)} \\
2\text{FeAsS}_{(arsenopyrite)} + 7\text{O}_2(g) + 2\text{H}_2\text{O}_{(aq)} & = 2\text{H}_2\text{AsO}_4_{(water)} + \text{Fe}^{3+} + 2\text{SO}_4^{2-} \\
4\text{Cu}_2\text{FeSnS}_4_{(stannite)} + 3\text{O}_2(g) + 2\text{H}_2\text{O}_{(aq)} & = 8\text{Cu}^{2+} + 4\text{Fe}^{3+} + 4\text{SnO}_2_{(sol)}, + 16\text{SO}_4^{2-} + 4\text{H}^+ \\
2\text{PbS} + 3\text{O}_2 & = 2\text{PbO} + 2\text{SO}_2\uparrow, \Delta G_r^0 = -389,64 \text{kJ} \\
\text{PbO} + \text{CO}_2 & = \text{PbCO}_3, \Delta G_r^0 = -43,30 \text{kJ}
\end{align*}
$$
2. The acid-base regime of waste is caused both by oxidation processes of the sulphide constituent, and by the processes of dissolution and hydrolysis of the ionic components of the mineral part of the waste, the volatility of hydrogen sulfide and the formation of elementary sulfur.

3. Silicate rocks that undergo acid hydrolysis can influence the formation of acid-base regime of waste:

\[
\text{Ca}_2\text{Al}_2\text{Fe}((\text{SiO}_3)_2)\text{OH}_{(epidote)} + 12\text{H}_2\text{O}_{(lq.)} + 10\text{CO}_2{}_{(g)} = 2\text{Ca}^{2+} + 2\text{AlOH}^{2+} + \text{FeOH}^{3+} + 10\text{HCO}_3^- + 3\text{H}_2\text{SiO}_3_{(water)}
\]

\[
2\text{NaAlSi}_3\text{O}_8{}_{(sol.)} + 11\text{H}_2\text{O} + 2\text{CO}_2 = \text{Al}_2\text{Si}_2\text{O}_5{}_{(OH)}{}_{(sol.)} + 2\text{Na}^+ + 2\text{HCO}_3^- + 4\text{H}_2\text{SiO}_4{}_{(water)}
\]

\[
\text{Al}_2\text{Si}_3\text{O}_5{}_{(OH)}{}_{(sol.)} + 3\text{H}_2\text{SO}_4{}_{(water)} = \text{Al}_2(\text{SO}_4)_3{}_{(water)} + 4\text{H}_2\text{SiO}_4{}_{(water)} + \text{H}_2\text{O}
\]

\[
\text{CaAl}_2\text{Si}_2\text{O}_8 + 2\text{H}_2\text{O} + 2\text{CO}_2 = 2\text{AlOH}{}_{(sol.)} + \text{Ca}^{2+} + 2\text{SiO}_2{}_{(sol.)} + 2\text{HCO}_3^-
\]

\[
2\text{Mg}_2\text{Fe}_2\text{Al}[(\text{AlSi}_3\text{O}_10)]{}_{(OH)}{}_{(chlorite)} + 7\text{H}_2\text{O} + 20\text{CO}_2 = 20\text{HCO}_3^- + 6\text{Mg}^{2+} + 4\text{Fe}^{2+} + 4\text{HSiO}_3^- + 2\text{AlOH}^{2+} + \text{Al}_2\text{Si}_2\text{O}_5{}_{(OH)}{}_{(sol.)}
\]

\[
\text{KAl}_3[(\text{AlSi}_3\text{O}_10)]{}_{(OH)}{}_{(sericite)} + 5\text{H}_2\text{O} + 4\text{CO}_2 = \text{Al}_2\text{Si}_2\text{O}_5{}_{(OH)}{}_{(aq.)} + \text{Al}^{3+} + \text{K}^+ + 2\text{H}_2\text{SiO}_4{}_{(water)} + 4\text{HCO}_3^-
\]

4. Acid-base regime forms the ion-molecular composition of the pore waters of waste of the processing of tin ores.

5. Oxidation-reduction regime is formed by the oxidation of sulfide sulfur, possibly by the cations of Fe[II], Mn[II], and the grade of dispersity and density of waste, that affects the access of oxygen to the zone of reactions.

6. Acid-base and redox processes are interrelated, compete with each other and have hidden reserves, which appear in the process of the weathering of the upper layers of the waste storage.

7. In order to adequately assess the regimes state of the waste, it is necessary to take into account these latent reserves of acidity and oxidizability.

For this purpose it is necessary to consider and estimate in addition indicators of the general acidity and alkalinity, oxidizability and chemical oxygen demand.

2. Experimental part

Indicators of total acidity and of alkalinity are the sum of concentrations of free ions H+ and OH - and of connected acidity and alkalinity. Connected acidity is manifested in the interaction with the bases.

Connected acidity due to the presence of weak acids, hydrolysis of cations of weak bases, their ability to form insoluble hydroxides:

\[
\text{H}_2\text{CO}_3 + \text{OH}^- \leftrightarrow \text{HCO}_3^- + \text{H}_2\text{O} \quad \quad \quad \quad \text{HCO}_3^- + \text{OH}^- \leftrightarrow \text{CO}_3^{2-} + \text{H}_2\text{O}
\]

\[
\text{H}_2\text{SiO}_4 + \text{OH}^- \leftrightarrow \text{HSiO}_3^- + 2\text{H}_2\text{O} \quad \quad \quad \quad \text{HSiO}_3^- + \text{OH}^- \leftrightarrow \text{SiO}_3^{2-} + \text{H}_2\text{O}
\]

\[
\text{AlOH}^{3+} + \text{OH}^- \leftrightarrow \text{Al(OH)}_2^{3+} + \text{H}_2\text{O} \quad \quad \quad \quad \text{Mg}^{2+} + \text{OH}^- \leftrightarrow \text{Mg(OH)}^+ + \text{H}_2\text{O} \quad \leftrightarrow \text{Mg(OH)}_2
\]

In some way, the connected acidity forms the pH-buffer effect, that maintains a constant pH value despite the effects of alkaline agents.

The connected alkalinity is manifested in the interaction with the acids and this is due to the presence of insoluble basic hydroxides and basic cations of these weak bases, as well as of hydrolysis of anions of weak acids.

\[
\text{CaCO}_3 + \text{H}^+ \leftrightarrow \text{Ca}^{2+} + \text{HCO}_3^- + \text{H}^+ \leftrightarrow \text{H}_2\text{O} + \text{CO}_2
\]

\[
\text{MgOH}^+ + \text{H}^+ \leftrightarrow \text{Mg}^{2+} + \text{H}_2\text{O} \quad \quad \quad \quad \text{Fe(OH)}_2 + \text{H}^+ \leftrightarrow \text{FeOH}^+ + \text{H}_2\text{O}
\]

The connected alkalinity forms pH-buffer of the system and maintains the pH constant at the action of strong acids. In the process of weathering, hyper - and technogenesis with chemical reagents of the environment reacts new and new layers of waste. The pH value does not take into account the potential protolytic potential of the mineral components of the waste. More reliably characterize the acid-alkaline regime of wastes of tin ores processing indicators of total acidity and total alkalinity.
3. Method of research
Evaluation of the protolytic capabilities of the studied wastes of tin ore processing at the "Solnechny" ore mining and processing enterprise was carried out according to standard methods [14], our help in the chemistry of environmental media.

Determination of total alkalinity was carried out by the method of reverse titration of a known amount of H\textsubscript{2}SO\textsubscript{4} after its 30-minute contact with 1 g of waste with a working solution NaOH. Determination of the total acidity was carried out by the method of reverse titration of a known amount of NaOH after its 30-minute contact with 1 g of waste working solution H\textsubscript{2}SO\textsubscript{4}. In both cases, was used the methyl orange indicator. Active acidity (pH) was determined in single, double and triple 10-day water extracts from 1 g of waste after contact with 10 cm\textsuperscript{3} of water on the pH-meter-conductometer «Anion 4154/5».

4. Results and discussions
4.1. Accordingly, pH values were obtained a pH of 4.99; of 5.23; 5, 69. The obtained pH values indicate the presence of acidic centers that generate free hydrogen cations. These centers probably act from the surface, since the pH values increase with the multiplicity of treatments with water. It is estimated that approximately are washed 0.04 mmol of ions H\textsuperscript{+} out of 1 kg of waste. As a result of a 30-minute contact of 1 g of waste with 4 cm\textsuperscript{3} of 0.1 n NaOH was established adsorption of groups OH\textsuperscript{-} in the amount of 0.16 mmol/g or 160 mmol/kg of waste. In the end, associated (potential) acidity amounted to 159,96 mmol/kg.

Thus, the experiment showed a huge reserve of potential acidity of tin ores processing wastes, which is not taken into account by the pH indicator, but affects the acid-base mode of man-made object. Reverse titration of sulfuric acid after contact with the waste did not reveal changes in its concentration, indicating the absence on the surface of waste donors hydroxide groups. It can be assumed that centers that absorb free hydroxyl groups, are the molecules weak acids, their acidic anions, basic cations and weak insoluble bases of the surface. Thus, the acid-base regime of mineral waste is formed both by ion-molecular composition of pore waters (pH index) and by mineral component with its potential protolytic capabilities. Therefore, in addition to the pH value and for a more objective assessment of the acid-base regime of mineral wastes, an additional assessment of the total acidity and alkalinity is required.

4.2. The redox regime in the solid waste is sum of Eh of the water of pores and the redox capabilities of the mineral component, which are manifested in time. To assess the potential redox-function of the mineral components of the waste, we have identified indicators of waste, such as: oxidability and chemical consumption of oxygen. These indicators were determined using standard titrimetric methods [12].

To determine the amount of reducing centers, we titrated the KMnO\textsubscript{4} residue after its 30-minute contact under mild conditions with 1 g of waste. As a titrant was used a 0.01 N solution of salt of Mohr.

As a result of the titration, it was found that the content of recovery centers in waste, which for 30 minutes in contact with the oxidizer (KMnO\textsubscript{4}), amounted to 21.8 mmol-EQ/kg waste. In order to estimate the maximum number of waste reducing centers, we used the standard methodology for accelerated determination chemical consumption oxygen [12]. As a result of titration, it was found that under extremal conditions the chemical consumption of oxygen of the mineral component of the waste is within 1120 – 1280 mg / g of waste or 140 – 160 mmol-EQ of reducing-centers for 1 g of waste.

After a 10-day contact of waste with water we received once flushed the waste and re-purposed them for research chemical consumption of oxygen, which amounted to the value of 1040 mg / g or 130 mmol-EQ of reducing-centers for 1 g of waste.

In parallel, we determined the chemical consumption of oxygen in water extracts from waste. In primary, secondary and tertiary extract it was respectively 120, 72 and 40 mg. oxygen in 1 dm\textsuperscript{3}. To
determine the probability of the presence of oxidation centers in solid waste of tin ores processing, the inverse titration of the Mora salt solution was carried out after its 30-minute contact with 1 g of waste. Served as the titrant is 0.01 n KMnO4, titration was carried out in sulfuric acid medium. The results of the analysis indicate that the concentration of the Mohr salt after contact with the waste has not changed. Consequently, the centers of oxidation on the surface of waste absence.

5. Conclusion

The following conclusions can be drawn from the assessment of the redox properties of the mineral components of tin ores processing wastes:

5.1. Tin ore processing waste stores are large reservoirs of reducing-reagents that can continuously consume oxygen from the environment for a long time. As a result, formates anaerobic zones with a constant deficiency of oxygen. When planning and carrying out measures for reclamation with the help of biotechnologies of such technogenic formations, specialists should take this fact into account.

5.2. The potential possibilities for absorbing oxygen out of the environment in waste storage facilities is enormous and in the near future we can not hope to replace the restorative (dead) mode on oxidative (life-giving).

5.3. However, as wastes is washed by water, the chemical consumption of oxygen by the surface layers have tendency to the decrease.

5.4. Unfortunately, extracted restored forms migrate to groundwater, shifting their redox regime towards the more reducing one.

5.5. Despite the fact that the content of reducing agents in solutions decreases as washing out, the dynamics of this process should not be considered promising, since in the processes of weathering include more and more deep layers of waste.

5.6. The formation of anaerobic zones and maintenance of oxygen deficiency is undoubtedly an important element of environmental damage. Having defined indicators of oxidability and chemical consumption of oxygen it is possible to predict the nearest and prolonged ecological damage from a reducing-regime of a mineral component of waste of processing of pewter ores.

5.7. And if liming is used to reduce pH during land reclamation, it is logical to find suitable, environmentally acceptable and affordable oxidizing reagents for elimination of reducing-centers of wastes, that be simultaneously perform the role of microelements, stimulants, herbicides. However, this aspect of the problem is not the subject of this study.

6. References

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