

Keywords: arsenic pollution, arsenic transformation, arsenopyrite, ore mining, toxic waste, industrial waste, anthropogenic pollution

Acknowledgements: We express our sincere gratitude to the Hanoi University of Natural Resources and Environment, Hanoi University of Science, the Southern Branch of the Joint Russian-Vietnamese Tropical Research and Technology Centre and the Institute of Environmental Technology, Vietnam Academy of Science and Technology, which created the conditions for the study and helped the research team in its implementation.

For citation: Le Thu Thuy, Tran Hong Con, Nguyen Trong Hiep, Vu Thi Minh Chau, Le Minh Tuan and Do Hoang Linh, 2024. Environmental Hazard Assessment of Storage Conditions of Wastes from Mining and Processing of Arsenopyrite Minerals. *Ecological Safety of Coastal and Shelf Zones of Sea*, (2), pp. 107–121.

Оценка экологической опасности условий хранения отходов добычи и переработки арсенопиритных минералов

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Аннотация

Арсенопирит – распространенный минерал класса сульфидов, относящийся к минералам гидротермального происхождения. На техногенных отвалах арсенопирит подвергается воздействию агентов выветривания и выделяет мышьяк в окружающую среду. В районах, где разрабатываются минералы Cu, Pb, Zn, загрязнение окружающей среды мышьяком является серьезной проблемой. Результаты настоящего исследования показывают, что при выветривании на отвалах в условиях просачивания и затопления арсенопиритные руды способны выделять мышьяк и тяжелые металлы. Представлены результаты лабораторного эксперимента на разработанной имитационной модели изменения вещества в рудных отвалах шахт при двух условиях: при просачивании (моделирование открытых отвалов руды, через которые просачивается дождевая вода) и затоплении (моделирование отвалов руды, хранящихся в затопленных низинных районах). Модельные условия соответствуют реальным. Соотношение арсенопирита и песка 1:20. Продолжительность эксперимента составляет 60 сут, что позволяет определить мышьяк в различных химических веществах. В ходе эксперимента в условиях инфильтрации воды pH снижается, а окислительно-восстановительный потенциал варьирует от 5 до 50 мВ, при снижении pH выделение металлов и мышьяка в окружающую среду с течением времени увеличивается. По достижении pH значений, характеризующих кислую среду (2.0–4.5), выветривание

заметно ускоряется. В условиях избытка воды при высоком содержании растворенного кислорода металлы высвобождаются быстрее. Когда pH находится в диапазоне от 5.5 до 6.0, скорость высвобождения металлов снижается. При окислении руды железо в двухвалентной форме Fe(II) медленно окисляется до Fe(III) при pH, указанном выше. В этих условиях Fe(III) гидролизуется в колонке. Таким образом, выделяющийся мышьяк адсорбируется на Fe(III), а образующийся гидроксид железа Fe(OH)₃ покрывает частицы руды.

Ключевые слова: загрязнение мышьяком, трансформация мышьяка, арсенопирит, добыча руды, токсичные отходы, промышленные отходы, антропогенное загрязнение

Благодарности: авторы выражают искреннюю благодарность Ханойскому университету природных ресурсов и окружающей среды, Ханойскому университету естественных наук, Южному отделению Совместного российско-вьетнамского тропического научно-исследовательского и технологического центра и Институту экологических технологий, Вьетнамской академии наук и технологий, которые создали условия для проведения исследования и помогли исследовательской группе в его реализации.

Для цитирования: Оценка экологической опасности условий хранения отходов добычи и переработки арсенопиритных минералов / Ле Тху Тхуи [и др.] // Экологическая безопасность прибрежной и шельфовой зон моря. 2024. № 2. С. 107–121. EDN WCMKJB.

Introduction

Arsenopyrite is a compound of iron and arsenic sulphide (FeAsS) and one of the most common minerals among sulphides today. The chemical composition of arsenopyrite, according to microprobe analysis, is usually as follows: Fe = 34.05%; As = 43.87%; S = 21.76% [1]. Samples of waste arsenopyrite ore for this study were taken from tin ore mining and processing waste in the upper reaches of the Nam Huong River (Quy Hop district, Nghe An province, Vietnam).

Mining has a negative impact on the environment and poses a danger to the ecosystem in general and to humans in particular [2]. It is a significant problem in many countries around the world, including Vietnam.

The mining and processing of natural resources increases the rate at which heavy metals enter the environment, including aquatic ecosystems. Waste arsenopyrite ore stored in dumps is exposed to the atmosphere and slowly oxidises. In the process, arsenic converts to more water-soluble oxides, which through natural transformation leads to the release of sulphur. These sulphide minerals oxidise to form acid mine discharges and alter the properties and toxicity of the metals. Despite the risk of arsenic and heavy metal pollution from mine acid wastewaters and ore dumps, little attention has been paid to this problem.

High levels of arsenic have been found in the area of ore mining, possibly due to the weathering of FeAsS arsenopyrite and its leaching in water, especially in an old tin mine in the Ron Phibun area of Thailand (5000 µg/L). In the USA, the mining district of Fairbanks County (Alaska) has an arsenic concentration of 104 µg/L, and arsenic concentrations in groundwater in Coeur d'Alene County (Idaho) are as high as 1400 µg/L [3].

G. S. Kamm et al. [4] found in their study that arsenic in the form of methylated compounds is transformed to a lesser extent than inorganic arsenic (As^{3+} and As^{5+}). Inorganic arsenic in soil can undergo various transformations including oxidation decrease, functional degradation and biotransformation. The flooded soil is dominated by arsenite with low redox potential, and at pH equal to 5–8, As^{+5} is mostly transformed as As^{3+} , while in a reducing environment and at low pH, As(III) will dominate.

Some studies on geochemical characteristics of arsenic in mining operations showed that the release of arsenic into the environment strongly depends on the pH value, redox potential and heavy metal content [5, 6].

Today, there are limited studies on arsenic transformation in Vietnam, so studying the release of heavy metals, As(III) and As(V) on ore dumps is of great importance. This will improve the environmental protection of mining operations and storage of their waste in natural conditions.

The purpose of this study is to use the results of laboratory experiments to assess the impact level of waste from arsenopyrite minerals mining and processing on the environment under different conditions of their storage (seepage and flooding).

Experimental part

Design and construction of experimental models, preparation of reagents

To study the release of arsenic and heavy metals from arsenopyrite ore dumps and the transformation of As(III) into As(V), it is first of all necessary to develop a simulation model of substance change in mine ore dumps under conditions of seepage (an open ore dump into which rainwater seeps is modelled) and flooding (ore dumps stored in flooded lowland areas are modelled) [7]. The structure of the experimental model is presented in Fig. 1, the main elemental composition in a sample of a 1:20 mixture of arsenopyrite ore and sand is given in Table.

Element composition (mg/kg) of arsenopyrite ore samples and mixture of arsenopyrite ore with sand (1:20)

Material	As	Cr	Mn	Fe	Ni	Cu	Zn	Cd
Arsenopyrite ore	164.73	9.32	1253.52	21377.45	47.96	5257.54	576.45	14.20
Mixture of arsenopyrite ore with sand	8.12	0.51	60.32	987.16	2.81	646.64	26.43	1.46

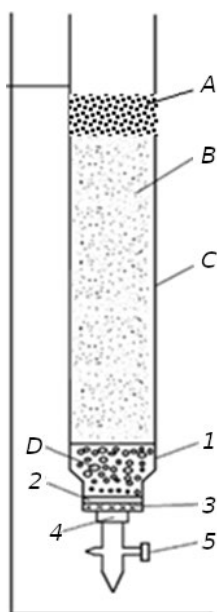


Fig. 1. Equipment scheme: 1 – plastic lid; 2 – fine mesh; 3 – plastic sheet (PE); 4 – rubber buttons; 5 – valve in the lower part; A – layer of gravel and soil taken from dump; B – sample of pyrite ore mixed with sand; C – column; D – gravel layer

The mixture of arsenopyrite ore and quartz sand in the ratio of 1:20 (this ratio is similar to that in the ore dump) is packed into a test column of 45 × 1000 mm (particle size 0.5–2.0 mm) and void volume of about 250 ml is left (Fig. 1). The order of the layers in the filled column is as follows. The first layer (A), 20 mm thick, is a mixture of soil debris and gravel removed from the mine to create conditions close to real ones (organic matter from 1.5 %) [8]. The second layer (B) is an ore–sand mixture 650 mm thick, weighing 878 g. The third layer (D) is a supporting layer of gravel with a diameter of 3–5 mm. The composition of the used aqueous phase is similar to that of natural rainwater [9].

The composition of the used aqueous phase (in mg/L) is similar to natural rainwater (pH = 6.5):

Ca ²⁺	2.4	Cl ⁻	3.90	NH ₄ ⁺	1.5	NO ₃ ⁻	4.44
Na ⁺	8.5	SO ₄ ²⁻	5.35	Mg ²⁺	1.9	HCO ₃ ⁻	24.40

Reagents. As(III) 0.1 M (7500 ppm) standard solution: a mixture of 0.9902 g arsenic (III) oxide and 2.5 g NaOH (chemically pure) was placed in a 100 mL measuring flask, then 70 mL of hypoxic water was added and shaken. Then 10 mL of 2 M solution of HCl in hypoxic water was added, brought to the mark with hypoxic water and stirred. The experiments were carried out under nitrogen atmosphere.

Standard solutions of As(V) and metal ions Cu²⁺, Cd²⁺, Mn²⁺, Fe²⁺, Ni²⁺, Zn²⁺ were prepared from Merck standard solutions.

Procedure

Seepage conditions. Water with the composition given above was poured into the column (Fig. 1), with the mouth of the column and the valve at the bottom open for two days. Then 120 mL of water simulating natural rainwater was poured into the column. The valve was opened and the seepage rate in the column was maintained at 8.5 cm/h (normal rate of water seepage through a waste ore layer) [10].

The solution that had passed through the column was collected in full. Then 20 mL of the collected solution was taken to analyse the content of Fe, Mn, Ni, Cu, Zn, total As, As(III) and As(V), which are the major ions often accompanying the weathering process of arsenopyrite. The remainder of the sample was saved for the following experiments. The valve and the column mouth were opened.

In five days, 120 mL of water simulating natural rainwater was added to the stored solution, then it was passed through the column and sampled. This sampling process was repeated once a day for five days (simulation of natural processes).

Flooding conditions. The research equipment was set up as shown in Fig. 1, and the effect of horizontal flow was not considered in the experiment due to its less influence on the weathering process. The arsenopyrite ore from the dump and the used aqueous phase had the same composition as in the seepage conditions.

First, the aqueous phase was oxygenated using an aerator to ensure a dissolved oxygen concentration of about 8 mg/L. After loading the ore, the column was continuously kept filled. The water level was 25 cm above the upper boundary of the ore–sand layer. The samples were left for five days after collection. At the same time, 20 mL of the newly collected solution were carefully withdrawn to analyse the same parameters that were determined under seepage conditions. The amount of water lost during sampling was replenished.

Under both seepage and flooding conditions, the experiment was conducted for 60 days [1]. An aliquot of the sample was taken at the same time to analyse for heavy metal content. The modelling experiments were repeated three times.

To further investigate the ability to release iron and arsenic in an acidic medium under real conditions, the authors conducted experiments at pH values from 4.5 to 2.5. The course of the experiment was the same as under seepage conditions: 120 mL of aqueous phase with the above composition was added in a pre-stored solution. A 4 M HCl solution was used to adjust the pH value of the water before each wash through the column and 20 mL of sample was taken for analysis. The above procedure was repeated (pH adjustment for five days and sampling).

Methods of As(III) and As(V) separation and quality indicator in a sample

The method of As(III) and As(V) separation in a solution of ethanol and water with a ratio of 30:70 involves single filtration using Lewatit MonoPlus M 500 ion exchange resin [11]. All experiments were performed in the nitrogen atmosphere to prevent arsenic oxidation. The process of arsenic extraction is shown in Fig. 2.

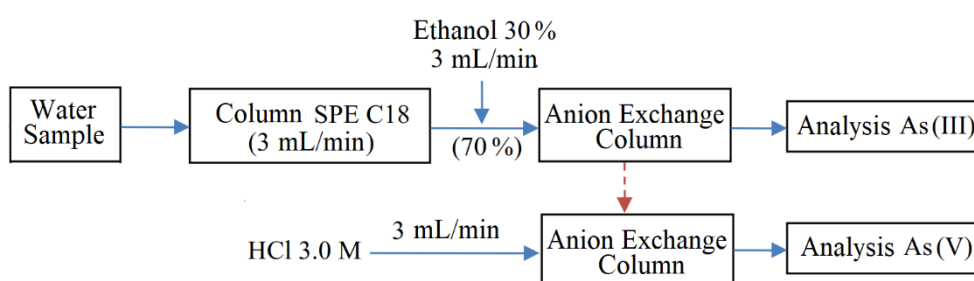


Fig. 2. Scheme of separation of As(III) and As(V) [11]

The content of Ni²⁺, Cu²⁺, Pb²⁺, Zn²⁺, Fe²⁺, As(III) and As(V) ions was analysed on an atomic absorption spectrometer, As was analysed by the cold vapour method.

Sample processing and analysis

The liquid samples were treated using HNO₃ and HCl acids at a volume ratio of HNO₃:HCl as 1:3, at a rate of 50 mL of acid mixture per 100 mL of the sample. Then, As and heavy metals were measured using an atomic absorption spectrophotometer (model iCE 3500, Thermo Scientific, USA), Fe²⁺ was analysed according to the Vietnamese standard TCVN on a DR 5000 device (HACH), whereas pH, Eh and dissolved oxygen content were measured using Hanna HI98304 electrodes (Romania).

Solutions of HCl, HNO₃ with different concentrations were prepared from 37 % concentrated perchloric acid HCl and 65 % nitric acid HNO₃ (chemically pure, Merck) in distilled water or in hypoxic distilled water, depending on the requirements of each experiment.

After separation of As(III) and As(V) by single filtration using Lewatit Mono-Plus M 500 ion exchange resin. A solution of ethanol and water (30:70 % vol.)¹⁾ was used as a reference solution [10], and the method reproducibility, the limit of detection (LOD) and the limit of quantification (LOQ) were evaluated [12].

The method reproducibility was calculated using the following formula:

$$S_r = SD = \sqrt{\frac{1}{n-1} \sum_{i=1}^n (X_i - \bar{X})^2}, \quad RSD\% = \frac{S_r}{\bar{X}} \times 100,$$

where S_r – standard deviation of repeatability; \bar{X} – average concentration of the analysed substance in the test sample; X_i – concentration of the sample of the i -th test; n – number of repetitions (10 times); RSD – relative standard deviation.

Limit of detection: LOD = 3 × SD.

Limit of quantification: LOQ = 10 × SD.

According to the experimental results, the LOD, LOQ values for As(III) were 0.06 and 0.1, respectively, and for As(V) were 0.05 and 0.08, respectively. These low values show that the analysis allows separation of As(III) and As(V) even for samples with low arsenic content.

¹⁾ Le Tu Hai, 2016. *Study of separation and determination method for anorganic arsenic(III) and arsenic(V) forms in natural water samples*. Abstract of doctoral thesis. Vietnam National University, Hanoi.

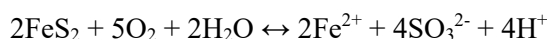
Results and discussion

Rate of metal and arsenic release in seepage conditions

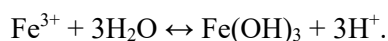
Change in the pH and redox potential Eh values in seepage conditions

The experiment was conducted as described in Section 2.2, with the thickness of the ore dump layer being 65 cm. But in fact, the exposed waste ore dumps can be tens of metres thick. Thus, the experimental model gradually accumulates weathering products. Fig. 3 shows the changes of pH and Eh after 60 days of testing.

The results of the studies, presented in Fig. 3, showed that pH tends to decrease gradually and Eh tends to increase gradually, but for 60 days of observation the change in the model was insignificant. The decrease in pH in the aqueous phase was due to the weathering of sulphur minerals (mainly iron sulphide) with the formation of H^+ ions,



and hydrolysis of metal ions (mainly Fe^{3+})



A small amount of Fe(II) was released in the first few days, and the oxidation of Fe(II) to Fe(III) consumed H^+ ions:



Thus, the pH value did not change significantly. When iron was released in larger amounts, the hydrolysis occurring in the column along with oxidative weathering led to an increase in the concentration of H^+ ions, so the pH always tended to decrease [13].

At the same time, the redox potential of the solution (Eh) seeping through the waste ore column tended to increase gradually. This may be due to the fact that initially arsenopyrite particles are less bound, so their contact with dissolved oxygen and air oxygen causes oxygen to diffuse through the aqueous film. Therefore,

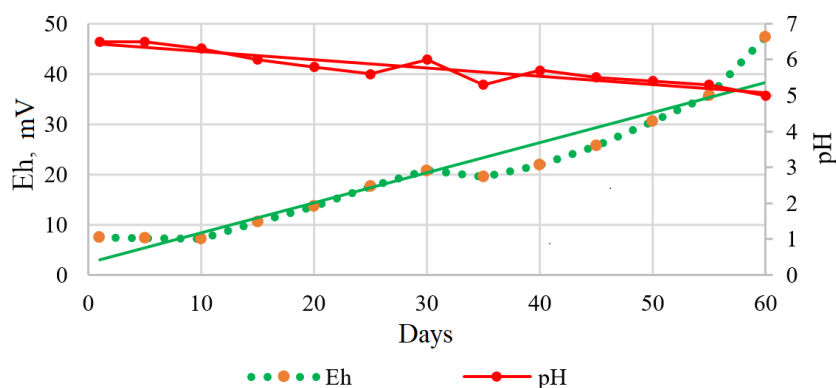


Fig. 3. Dynamics of pH и Eh under seepage conditions

more oxygen is consumed. Over time, the surface of ore grains decreases due to adhering layers of iron hydroxide, which hinder the oxidative weathering process. But under the experimental conditions, the total amount of air oxygen entering the waste ore column during sampling was almost the same.

Consequently, the concentration of dissolved oxygen increased. This led to an increase in Eh of the aqueous phase passing through the column. Throughout the experiment, the pH of the aqueous phase was always between 5 and 6, so it can be considered that the concentration of Fe^{3+} was only within the solubility product of $\text{Fe}(\text{OH})_3$ at the corresponding pH value. Consequently, Fe^{3+} has an insignificant effect on the redox potential of the aqueous phase.

Release rate of arsenic and heavy metals in the aqueous phase

Changes in the concentrations of arsenic and some major heavy metals in arsenopyrite ore accumulated in the aqueous phase during 60 days are shown in Fig. 4.

During the first 30 days of the experiment, when the waste ore was exposed to air and water oxygen, the release of iron and arsenic from the ore tended to increase gradually and pH of the aqueous phase was about 6.0. At this pH value, $\text{Fe}(\text{II})$ was oxidised to $\text{Fe}(\text{III})$ due to dissolved air oxygen in water, simultaneously $\text{Fe}(\text{III})$ was hydrolysed to form a sparingly soluble $\text{Fe}(\text{OH})_3$ precipitate and remained on the waste ore column. The released arsenic is also adsorbed on $\text{Fe}(\text{OH})_3$, so the arsenic concentration in the aqueous phase increased slowly.

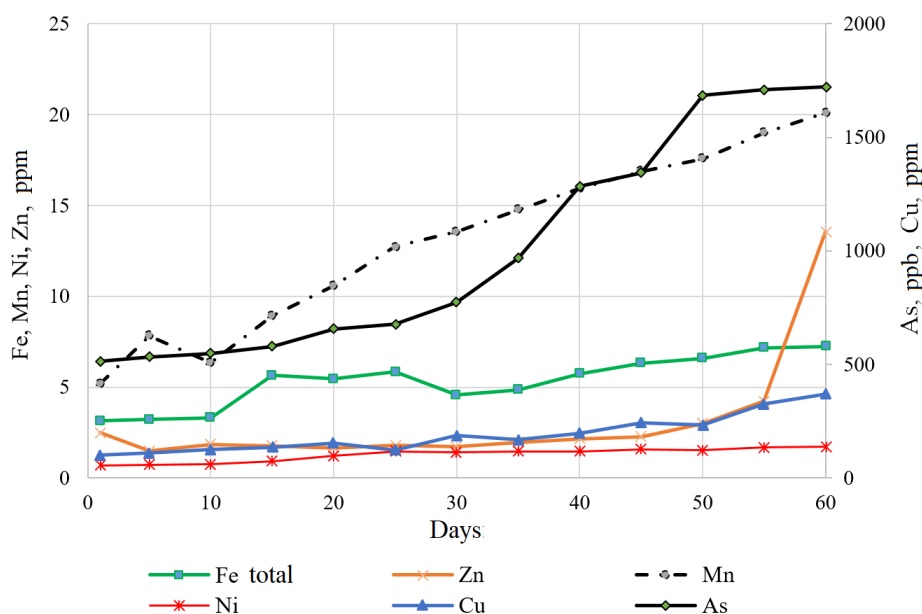


Fig. 4. Release of arsenic and some metals into the water phase

After 30 days, when pH of the aqueous phase decreased below 6 and continued to decrease below 5, the concentration of total iron in the aqueous phase increased markedly due to a decrease in the oxidising capacity of air oxygen and the precipitation of $\text{Fe}(\text{OH})_3$ decreased. In parallel with this process, the total arsenic concentration was increasing rapidly as the percentage of this element, adsorbed on $\text{Fe}(\text{OH})_3$, was decreasing.

The content of iron and arsenic in the aqueous phase at this stage is also affected by the increased rate of ore weathering along with an increase in H^+ concentration in the aqueous phase. The content of other metals increased markedly only during the first week of weathering; thereafter, it remained almost unchanged. At the same time, arsenic was also adsorbed on the formed $\text{Fe}(\text{OH})_3$ until pH of the water decreased to about 5.0. The mechanism of heavy metals (Mn, Ni, Cu and Zn) release from the ore is the same as for arsenopyrite. When the pH value falls below 5.0, their concentration noticeably increases and is: Mn from 5.21 to 20.11 ppm, Ni from 0.67 to 1.74 ppm, Cu from 99.64 to 370.42 ppm and Zn from 2.51 to 13.56 ppm. The above results showed that pH is a secondary factor, while it has a decisive influence on the release of arsenic and other heavy metals from exposed dumps of arsenopyrite ore.

The study results also showed that the release of arsenic and heavy metals in the waste ore increases over time and their concentration in the filtrate stream will increase (especially Zn and Mn) (Fig. 4). Consequently, they pose a higher risk to the environment.

Transformation of arsenic during arsenopyrite weathering

The experiment under the above conditions shows that in parallel with oxidative weathering of arsenopyrite, As(III) is oxidised and released from the ore, as well as sulphur is. The results showed that Eh of the aqueous phase in the column varied from 5 to 50 mV (Fig. 5). Over time, as the concentration of As(III) increased, the concentration of As(V) also increased. This suggests that the oxidation of As(III) to As(V) in the waste ore occurs almost continuously, at pH values between 5 and 6. There is a balance between adsorption and desorption of pentavalent arsenic on precipitated $\text{Fe}(\text{OH})_3$ in the column as well as between As(III) and As(V) content in the aqueous phase, which is also shown by [1].

In dumps of arsenopyrite ores or sulphur-containing minerals, the actual measured pH of the filtrate has very low values (from 2.5 to 4.5) [13, 14]. This pH favours the dissolution of minerals with the formation of dissolved metals, which are toxic particles that then enter the water.

During the experiment, the pH value of the solution in the waste ore column may not have reached the actual pH values in the aqueous phase (Fig. 5).

Fig. 5 shows that pH decreased from 4.5 to 3.5 and the total iron concentration in the aqueous medium increased sevenfold. This is due to the solubility of iron in water, mainly Fe(III), by precipitation of hydroxide. At the same time weathering was stronger in the acidic environment. At pH from 3.5 to 2.5, the total iron concentration increased more slowly (about 2.2 times), since the hydroxide forms of iron are almost completely dissolved in the aqueous phase in the form of

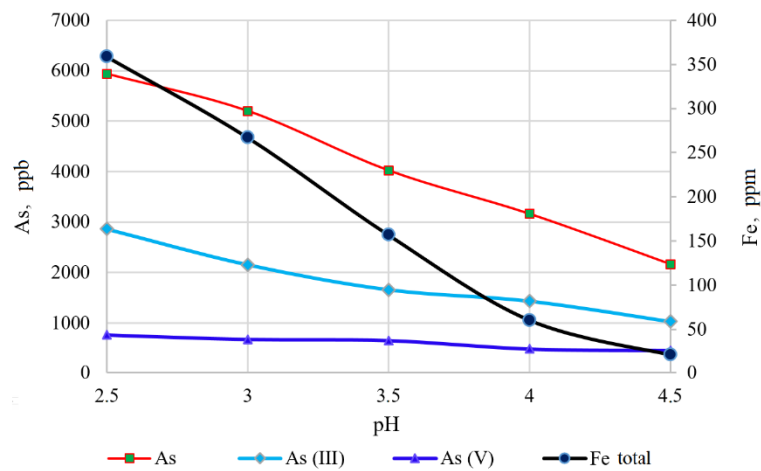


Fig. 5. Release of iron under low pH

$\text{Fe}(\text{OH})^{2+}$ and $\text{Fe}(\text{OH})^{2+}$ hydroxocations, as well as Fe^{3+} . As can be seen in Fig. 5, As(V) is an ion with strong adsorption capacity on Fe(III) hydroxide, so in the region of low pH values, the concentration of As(V) increased less than that of As(III). This indicates that As(V) was not adsorbed (whereas $\text{Fe}(\text{OH})_3$ was almost absent). When pH decreased from 3.5 to 2.5, the concentrations of Fe and As(III) increased, possibly due to an increase in the weathering rate in the acidic media, resulting in greater release of these elements into the aqueous phase.

Release rates of weathering products and some heavy metals in flooding conditions

The experiments were carried out as described in Section 2.2. The changes in pH, Eh and concentration of some typical heavy metals during the experiment are presented in Fig. 6, which shows that the pH value decreased markedly (from 6.5 to 5.0) when the ore was loaded into the column. The decrease in pH is due to the fact that dissolved oxygen in the initial aqueous phase provides the oxidative weathering of the ore. As a result, a certain amount of H^+ ions is released and pH decreases. In this condition, the Eh value decreased sharply, possibly due to the oxidation processes in the ore, which resulted in the formation of alternative forms of reduced compounds. By the end of the experiment, the Eh value decreased sharply due to the predominance of reduced forms, and no more dissolved oxygen was introduced into the column.

In flooding conditions with low water content, the pH and Eh dynamics were completely different from those under seepage conditions, and the weathering processes with release of ions into the aqueous phase also varied completely in different conditions. Concentrations of heavy metals in the water increased significantly in the first three weeks and then decreased slowly (Fig. 6, b). This is fully consistent with the evolution of the pH value in the aqueous phase. As mentioned above,

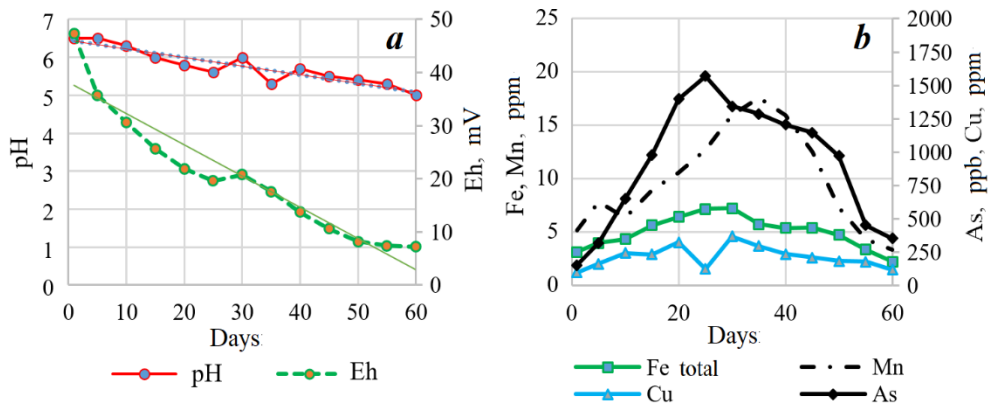


Fig. 6. Dynamics of pH и Eh (a) and concentrations of some elements (b) under flooding conditions

oxidative weathering occurs at this stage under the influence of initially dissolved oxygen. However, with a gradual decrease in oxygen content, the pH increased, Eh decreased, the concentration was almost constant, as with other metals, and then gradually decreased. This reduction process occurs because the amount of generated metal ions decreases while pH increases, which favours the adsorption of metal ions and their stronger binding to the newly formed $\text{Fe}(\text{OH})_3$. This is also in agreement with the results of similar studies [5, 13].

In conditions of oxygen deficiency in water, the concentration of As(V) in the aqueous phase tended to decrease gradually with decreasing dissolved oxygen, while the concentration of As(III) increased gradually (Fig. 7). In this case,

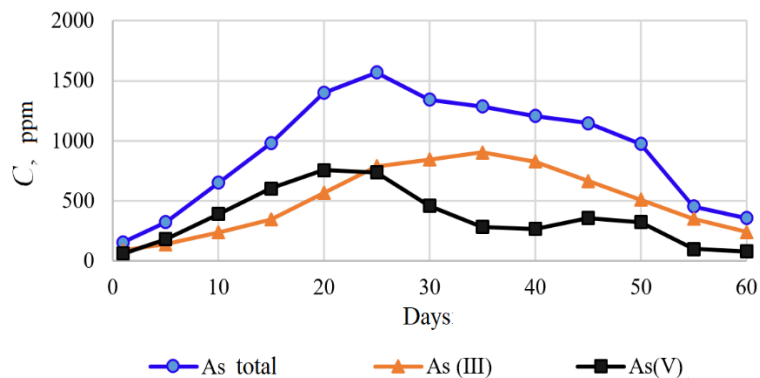


Fig. 7. Transformation of arsenic under flooding conditions

it can be clearly seen that the total concentration increased only in the first days when oxidative weathering process mainly occurred. Consequently, the concentration of metals increased in the aqueous environment. Then, with decreasing oxygen content, the total concentration hardly changed. Only the transformation of As(V) to As(III) in the aqueous phase took place with the predominance of reducing ions such as sulphite ions.

The above experimental results show that in the open air the dumped arsenopyrite ore is able to oxidise with the release of iron and other heavy metals. However, at pH value > 5 heavy metals are easily adsorbed and bound to Fe(III) hydroxide, which results in reduction of their ability to enter the environment. In the pH range from 2.5 to 5.0, large amounts of iron and other heavy metals are released.

Under flooding conditions, due to oxygen deficiency, the weathering process only occurs initially, when there is still enough oxygen in the aqueous phase as well as in the dumped ore mass. After that, the weathering process almost stops, instead reactions between the previously formed products take place, with the ion concentration in the aqueous phase almost unchanged.

If tailings of arsenopyrite or other sulphide minerals are left submerged, the potential for environmental contamination from weathering will be greatly reduced.

Conclusion

In order to assess the environmental safety of storage conditions of arsenopyrite waste ore dumps, the paper presents the results of laboratory experiment in two conditions: under seepage (modelling of an exposed ore dump with rainwater seepage) and flooding (modelling of ore dumps stored in flooded lowland areas). The obtained data allowed assessment of the influence of storage conditions on the release of arsenic and other heavy metals into environmental objects.

Laboratory modelling of weathering of arsenopyrite waste ore in seepage conditions shows that the pH value gradually decreases with time; while the content of released elements gradually increases. At lower pH values (4.5–2.5), the concentration of elements in the aqueous phase noticeably increases. This is explained by the effects of adsorption, desorption, dissolution of Fe(III) hydroxide formed due to weathering of ore, and formation of acid in the process of weathering.

In flooding conditions, the elements are released from arsenopyrite ore only in the initial period, then weathering almost does not occur and the concentration of the elements in the aqueous phase almost does not increase.

Weathering of arsenopyrite ore is one of the pathways for metals to enter the aquatic environment and is a cause of environmental pollution. Thus, limiting the As release process by storing waste ore and ore tailings in a flooded form will significantly reduce environmental pollution during mining of arsenopyrite and sulphur-containing ores in general.

Thus, As(III) dominates the weathering process. It is toxic to the environment, so further research is needed to propose solutions for its conversion to As(V).

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Submitted 20.04.2023; accepted after review 28.10.2023;
revised 27.12.2023; published 25.06.2024

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All the authors have read and approved the final manuscript.