

## Article

# Processing of Alluvial Deposit Sands with a High Content of Copper and Nickel Using Combined Enrichment Technology

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**Abstract:** The aim of the present research was to examine the process of bioleaching and the application of a combined process for the recovery of copper and nickel from industrial sand deposits. The investigated sample of sands finer than 0.1 mm in size contained 0.32% Ni and 0.22% Cu. Industrial sands were processed by bioleaching in flasks on a thermostatically controlled shaker. In addition, sand roasting experiments were carried out with ammonium sulfate. An attempt was also made to use a combined process, including low-temperature roasting of the sands mixed with ammonium sulfate, water-leaching of the roasted mixture, and subsequent biological after-leaching of the residue. In the process of roasting the industrial sands in a mixture including ammonium sulfate at a temperature of 400 °C, more than 70% of the non-ferrous metals were recovered. We examined the possibility of recovering non-ferrous metals using a combined process including low-temperature roasting of industrial sands and the additional recovery of non-ferrous metals by bioleaching using the *Acidithiobacillus ferrooxidans* bacterial strain, which was found to increase the recovery of non-ferrous metals to up to 90%.

**Keywords:** copper; nickel; low-temperature roasting; bioleaching; industrial sands



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## 1. Introduction

Every year, in Russia, the mining and mineral processing industry produces a significant amount of mining waste (overburden and host rock dumps, sludge, concentration tailings, and other waste), which makes up approximately 95% of the total amount of waste generated from production and consumption [1,2].

Over the past 10 years, this amount of waste has increased to more than double (from 3066 to 6851 million tons), while the share that waste that is recycled is only 52%, mostly for the backfilling of mines and open pits or land reclamation. However, the accumulated waste contains valuable minerals, which, considering the depletion of mineral reserves, are of potential economic interest. In addition, industrial waste has a significant negative impact on the environment; therefore, minimizing its production is expected to not only provide economic benefits but also reduce the environmental burden in the areas where the waste is stored [1,3].

The Norilsk industrial region is a group of residential communities organized as a municipal unit—the city of Norilsk. Norilsk is located in the north of Russia's Krasnoyarsk Territory and forms a major industrial conglomerate. The region hosts the world's largest palladium and nickel production facilities, as well as one of the largest platinum and copper production facilities. The public joint stock company "Mining and Metallurgical Company Norilsk Nickel" has been operating continuously for more than 85 years, which, given the depletion of resources, is an unprecedented period at the global level. The enterprise produces heavy metallurgical products such as technical sulfur, technical selenium, cathode copper, precious metal concentrates, nickel matte, primary nickel, nickel carbonyl shot, nickel carbonyl powder, nickel concentrate, cobalt concentrate, electrolytic cobalt, technical sulfuric acid, sodium sulfate, sodium chloride, and copper matte [2–5].

The leading mining and metals operation in the area is PJSC MMC Norilsk Nickel, which mines copper–nickel ores to recover non-ferrous and platinum-group metals. The ore processing technology implemented, using, for example, ore flotation, is characterized by a significant loss of precious and non-ferrous metals with the tailings, drastically reducing process performance and leading to environmental pollution. At the moment, more than 300 million tons of industrial waste have been accumulated, which is considered a promising source of non-ferrous and precious metals [4].

Previously, the processes used did not allow for the complex recovery of valuable components from the mineral feed, given existing capabilities. However, the large-scale processing of mineral waste is currently also constrained by the need for optimized process solutions.

Biohydrometallurgy offers approaches enabling the recovery of metals from various types of residues and wastes produced by mining and industrial processes, as well as making it possible to effectively process poor and refractory ores, refractory concentrates containing gold- and platinum-group minerals, and in some cases non-ferrous metal concentrates [6–11]. Among the available biohydrometallurgical methods, the technology enabling the heap bioleaching of copper ores is the one most widely used in global practice. This technology is characterized by the simplicity of the necessary equipment, the low cost of operation, the low consumption of energy and reagents, and the reduction in gas emissions. In addition, this technology is used to process low-grade ores containing non-ferrous metals such as nickel, zinc, and uranium and is used for the processing of refractory gold-bearing ores. The use of reactor bioleaching provides greater productivity in the context of raw material processing for minerals but comes with significantly higher costs due to the more complex technological regime required; therefore, in global practice, it is used to process refractory sulfide gold-bearing concentrates and has successfully been used for processing substandard sulfide concentrates containing nickel and cobalt [12–14].

The active development of various biohydrometallurgical approaches continues, with the use of microorganisms having been suggested for the extraction of metals from various mineral and secondary raw materials, making it possible to further develop the field of biohydrometallurgy and increase its practical significance. This work is aimed at creating new biohydrometallurgical technologies based on the use of already-known approaches for use in the processing of new types of raw material, such as ores and concentrates containing platinum-group metals, as well as the development of new principles whereby various groups of microorganisms are used that are not currently involved in biohydrometallurgical processes, but which are capable of transforming various minerals, as well as interacting with metals, such as, for example, the use of heterotrophic bacteria and fungi for the processing of gold-bearing ores, double refractory concentrates or ores containing rare earth elements, as well as the use of neutrophilic and moderately acidophilic microorganisms in the processes of biooxidation of sulfide ores and concentrates [6,8,10,15–21].

Biohydrometallurgical processes open new pathways for the recovery of metals from various types of mining and industrial waste. These technologies are characterized by simple processing equipment, low operating costs, low energy consumption, reduced consumption of reagents, and reduced gas emissions. Biological methods for the recovery of non-ferrous metals from production waste are increasingly being used globally [10].

Thermohydrochemical methods are of particular interest. A lot of attention is paid to the use of ammonium sulfate, which is due to its high reactivity when interacting with non-ferrous metal sulfides (the interaction mechanism was described in our previous reports) [22]. The low-temperature sulfide roasting process with ammonium sulfate has been successfully applied globally to various types of mineral feeds [23], such as lateritic and sulfide nickel ores [24,25], bauxite [26], oxidized zinc [27], manganese ores [28]. In general, the enrichment operations have been carried out on a laboratory scale. Ammonium sulfate has been used by the authors [29] in order to process the converter slag containing 4.03% copper, 1.98% nickel, and 0.48% cobalt. During the roasting of slag and  $(\text{NH}_4)_2\text{SO}_4$  mixture under atmospheric conditions, metal sulfates were formed. Then the resulting

roasted mixture was leached in distilled water. In the course of the research, the authors varied the roasting temperature (200–600 °C), roasting duration (15–120 min), as well as slag/ammonium sulfate ratio (1:0.5–2.5 mol/mol). The best results were achieved by the authors at a ratio of 1:2.5; the recovery of copper, nickel, and cobalt into the solution was 85%, 81%, and 85%, respectively. Similar studies were carried out with sulfuric acid, and the influence of such experimental variables as the amount of sulfuric acid (1:0.25–2 mol/mol), roasting temperature (100–300 °C), and roasting time (15–120 min) has been studied. One of the main advantages of using sulfuric acid was the reduction of the iron concentration in the pregnant solutions without reducing the recovery of other metals.

In [30], the authors studied the interaction of a concentrate of mixed oxide–sulfide nickel-containing ore with ammonium sulfate was studied when heated to 650 °C. The concentrate contained, %: nickel 8.93, cobalt 0.24, copper 6.51, iron 28.26. It has been shown that after roasting at 650 °C for two hours, the recovery of non-ferrous metals into solution during the aqueous leaching of the roasted mixture was %: Ni 70, Co 89, and Cu 90, while iron was almost completely converted into iron oxides magnetite ( $\text{FeO} \times \text{Fe}_2\text{O}_3$ ) and hematite ( $\text{Fe}_2\text{O}_3$ ).

The purpose of the research was to recover copper and nickel from industrial sands using a combined technology that includes low-temperature roasting followed by bioleaching of the residue.

## 2. Materials and Methods

### 2.1. Industrial Sands

The industrial sands are an alluvial placer deposit, which was formed by washing out the tailings of copper–nickel ore enrichment, as well as the dust of the Minor Smelter and Nickel Plants (Norilsk, Krasnoyarsk Territory, Russia). The investigated sample of sands finer than 0.1 mm in size contained 0.32% Ni and 0.22% Cu. The yield of the size class < 0.1 mm was 12.6 wt% (Table 1).

**Table 1.** Particle size distribution of the industrial sands.

Particle size, mm	−2 + 1	−1 + 0.5	−0.5 + 0.25	−0.25 + 0.1	−0.1 + 0
Content, %	4.05	14.0	32.3	37.0	12.6

### 2.2. Bioleaching Research

In order to assess the possible toxicity of this mineral feed to the bacterial strain *Acidithiobacillus ferrooxidans*, as well as the feasibility of the bioleaching method, a laboratory-scale experiment was carried out. The bioleaching experiment was carried out in flasks containing the alluvial sands sample (1 g), as well as 100 mL of 9K Silverman and Lundgren mineral solution (g/L):  $(\text{NH}_4)_2\text{SO}_4$ –3.0; KCl–0.1;  $\text{MgSO}_4 \times 7\text{H}_2\text{O}$ –0.5;  $\text{K}_2\text{HPO}_4$ –0.5;  $\text{FeSO}_4$ –44.22, the initial number of bacteria was  $6 \times 10^7$  cells/mL. The experiment was carried out in a thermostatically controlled shaker at 30 °C for 28 days with a stirring intensity of  $200 \text{ min}^{-1}$ . Bacterial cells were counted quantitatively under a light microscope equipped with a phase-contrast device (Olympus System Microscope Model CX41, Tokyo, Japan).

### 2.3. Low-Temperature Roasting Research

In addition, research was carried out on the sands and ammonium sulfate mixture roasting. In the course of the experiment, industrial sands were mixed with chemically pure ammonium sulfate, followed by grinding in a BMU-100 ball mill (HT Machinery Co., Ltd., Harbin, China). The resulting mixture was roasted for 240 min at various temperatures in a SNOL 3/11 muffle furnace (NPF Thermix LLC, Moscow, Russia). The roasting temperature was varied between 200 and 400 °C, heating time to the set temperature was 60 min. After the roasting, the mixture was cooled for 60 min. Then, the roasted mixture was leached in

distilled water heated to  $\approx 80\text{ }^{\circ}\text{C}$  for 40 min with constant stirring at a speed of  $230\text{ min}^{-1}$  using an MV-6 overhead stirrer (LLC NV-LAB, Moscow, Russia).

#### 2.4. Combined Process of Processing Industrial Sands

An attempt was also made to implement a combined process, including low-temperature roasting of the sands and ammonium sulfate mixture, followed by water leaching of the roasted mixture and subsequent biological after-leaching of the residue. Experiments were carried out using a shaker at  $30\text{ }^{\circ}\text{C}$  for 14 days. The sample weight was 2 g, the volume of the leaching solution inoculated with bacteria was 100 mL, and the initial population was  $6 \times 10^7$  cells/mL.

#### 2.5. Sampling and Analysis

The parameters of the liquid phase were monitored throughout the experiment in order to assess the activity of the strain. The values of pH and redox potential (Eh) were measured using a pH-150MI pH meter (Izmeritelnaya Tekhnika, Moscow, Russia). The concentrations of  $\text{Fe}^{3+}$  and  $\text{Fe}^{2+}$  ions in the liquid phase were determined by the method of titration with Trilon B, while the concentrations of copper and nickel were determined by the atomic absorption spectrometry with electrothermal atomization according to PND F 14.1:2:4.140–98. The recovery intensity was estimated based on the concentration of metals in the liquid phase.

The initial sands, as well as the sample after bioleaching, were examined by the method of X-ray diffraction analysis (XRD). The diffraction patterns were recorded using a Rigaku MiniFlex-600 powder diffractometer (Rigaku Co., Ltd., Tokyo, Japan) in scanning mode at a speed of  $1^{\circ}/\text{min}$  and an increment of  $0.01^{\circ}$ . Analysis of the crystalline phases was performed using the ICDD powder diffraction database. Data processing was conducted in MS 15.0.459.1506 Excel 2013 (Microsoft Co., Ltd., Redmond, WA, USA).

### 3. Results

#### 3.1. Bioleaching Experiments

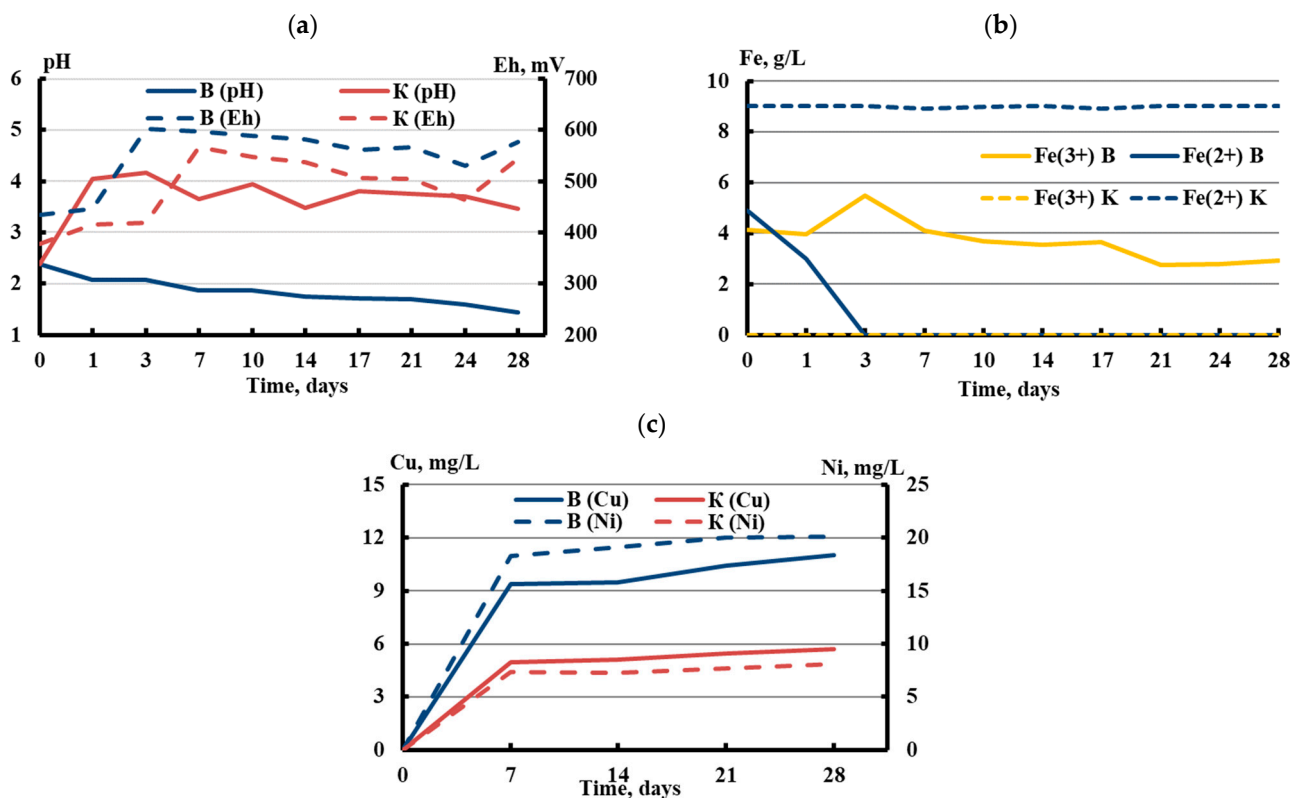
During the experiment, a gradual decrease in the pH value was noted both in the presence of bacteria and in the sterile control. At the same time, the redox potential increased significantly on the 3rd day. The change in the Eh value corresponded to the change in the concentrations of  $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$  ions in the solution; on the 3rd day, ferrous iron was completely oxidized (Figure 1a). In the sterile control, there was an increase in the pH values already on the first day, while during the following days of the experiment, it fluctuated in the range of 3.4–4.1. At the same time, the Eh value increased on the 7th day, while the concentrations of ferrous and ferric iron ions remained stable (Figure 1b). The concentrations of copper and nickel in the solution were gradually increased; however, the concentrations of copper and nickel, both in the presence of bacteria and in the sterile control, were increased most actively in the first seven days (Figure 1c). At the same time, in the case of bioleaching, the concentration of metals exceeded the concentration of metals in the sterile control. In just 28 days, 39.3% of copper and 49.1% of nickel were recovered by the bioleaching method; in the sterile control, 20.3% of copper and 19.8% of nickel were recovered.

Figure 2 shows the diffraction patterns of the industrial sand initial sample, as well as the residue after the bioleaching. The initial sample of the industrial sand (Figure 2a) was represented by the fragments and minerals of the ultramafic rocks, considerably altered (oxidized, partially broken) with a large number of intergrowths containing ore inclusions. In the residue after bioleaching (Figure 2b), the presence of ammoniojarosite reflexes was observed, which is interconnected with a decrease in the concentration of iron. Various types of jarosite (ammoniojarosite  $((\text{NH}_4)(\text{Fe}^{3+})_3(\text{SO}_4)_2(\text{OH})_6)$ , hydronium-

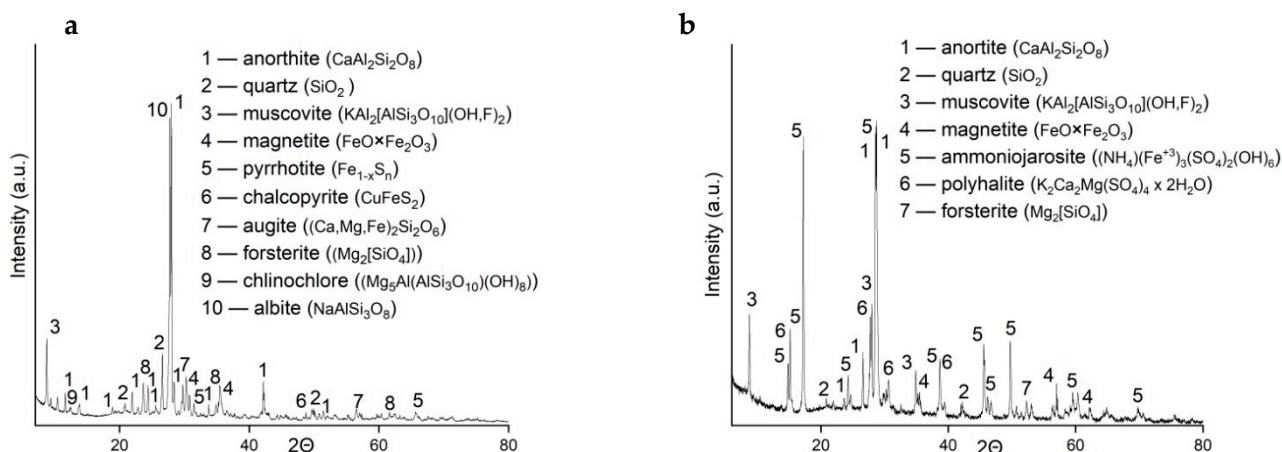
jarosite  $((\text{H}_3\text{O})\text{Fe}^{3+}_3(\text{SO}_4)_2(\text{OH})_6)$ , natrojarosite  $(\text{Na}(\text{Fe}^{3+})_3(\text{SO}_4)_2(\text{OH})_6)$  have a passivating influence on the bioleaching process, the formation of this phase occurs as follows:



where Me—ions of  $\text{K}^+$ ,  $\text{Na}^+$ ,  $\text{H}_3\text{O}^+$ ,  $\text{NH}_4^+$ .



**Figure 1.** Industrial sand leaching process parameters, where B is bioleaching, K is sterile control ((a)—pH and redox potential values, (b)—Fe ions concentrations, (c)—Cu and Ni ions concentrations).



**Figure 2.** Diffraction patterns of industrial sand samples prior to (a) and after (b) bioleaching.

In the diffraction pattern of the sand after its interaction with the bacterial solution, no reflexes of pyrrhotite  $(\text{Fe}_{1-x}\text{S}_n)$  and chalcopryrite  $(\text{CuFeS}_2)$  were found.



### 3.2. Low-Temperature Roasting

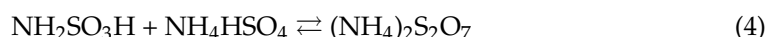
The process of thermal decomposition of ammonium sulfate occurs in the temperature range of 155–450 °C [31]. At a temperature of 326 °C, the formation of ammonium hydrosulfate begins with the release of an ammonia molecule according to the formula:



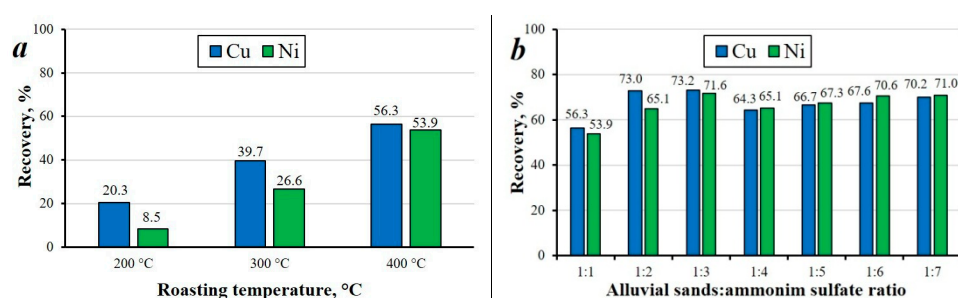
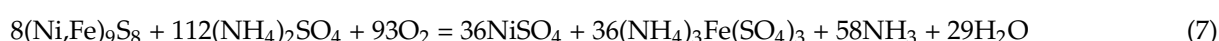
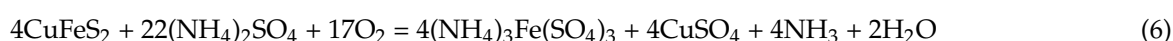
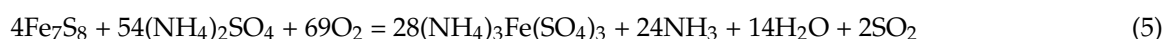
In the process of decomposition of ammonium sulfate with the formation of hydrosulfate, along with ammonia, the following compounds are released into the gas phase: water, sulfur dioxide, sulfur trioxide, sulfuric acid, hydrogen, and nitrogen. As the temperature rises, ammonium hydrosulfate begins to decompose. The first stage of decomposition is the dehydration and the formation of a small amount of sulfamic acid:



The acid, in turn, decomposes with the formation of gases—hydrogen, ammonia, and sulfur dioxide. It has been established that sulfamic acid easily reacts with ammonium hydrosulfate, forming ammonium pyrosulfate:



In the process of the industrial sands and ammonium sulfate mixture roasting, the following results were obtained. A significant increase in the recovery of metals was noted at a temperature of 400 °C, at the indicated temperature, and a ratio of feed to sulfate of 1:1, 56.3% copper and 53.9% nickel, was recovered (Figure 3a). The maximum recovery of metals was achieved at a temperature of 400 °C and a ratio of 1:3; the recovery of copper was 71.6%, while that of nickel was 73.2% (Figure 3b). The process of non-ferrous metal sulfates formation, which occurs during the low-temperature roasting, can be expressed as follows:

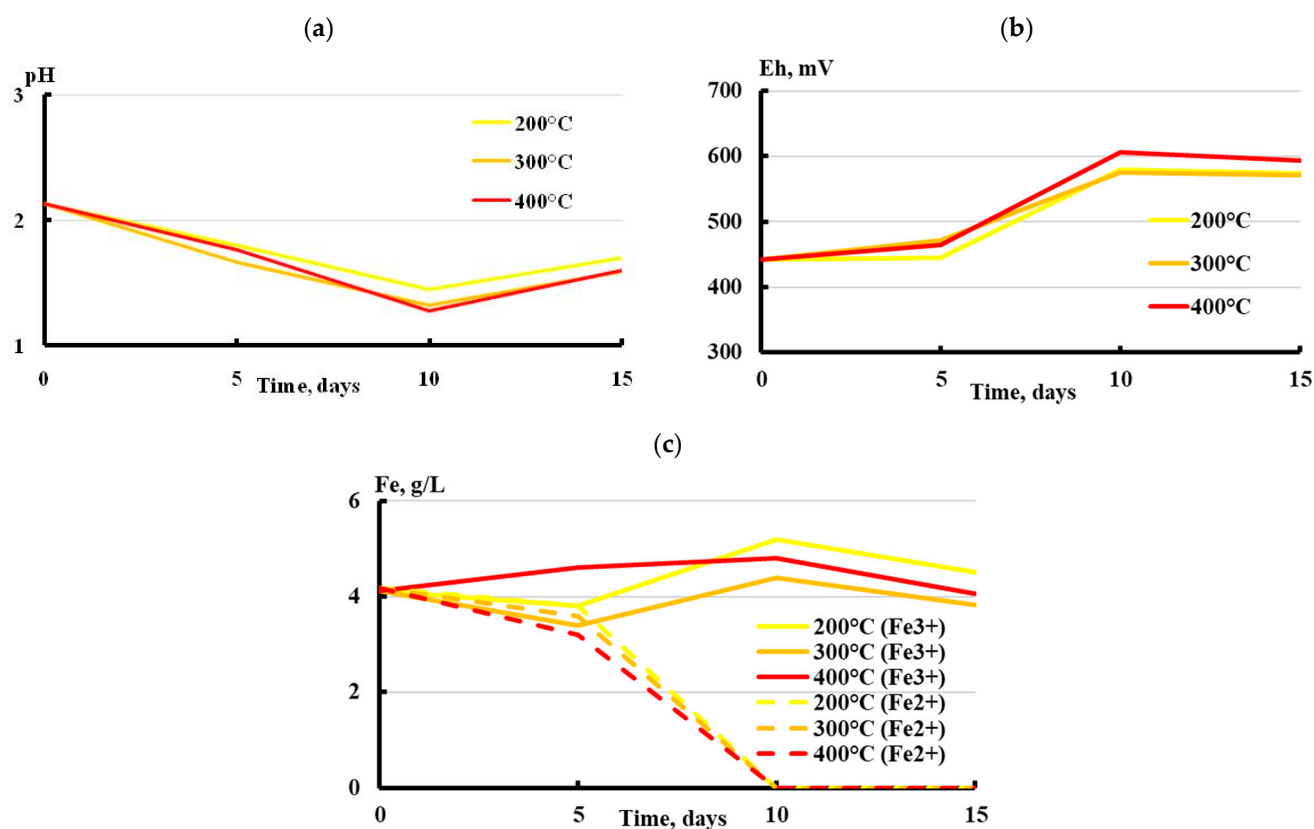


**Figure 3.** Recovery of copper and nickel during the roasting of the industrial sands and ammonium sulfate mixture, depending on the roasting temperature (a), as well as the ratio of feed to ammonium sulfate at a temperature of 400 °C (b).

The thermodynamic parameters of the roasting process of sulfide minerals and ammonium sulfate mixture, as well as the oxidation of basic copper and nickel-containing sulfides, are considered in detail in [32,33].

### 3.3. Bioleaching of the Residue after Water Leaching of the Sand and Ammonium Sulfate Roasted Mixture

In the course of the bioleaching of the residue after water leaching of the roasted mixture of alluvial sand and ammonium sulfate, a gradual decrease in the pH values was observed (Figure 4a). The lowest values of 1.28–1.32 were observed on the 12th day over the entire measurement period in the residues after roasting at temperatures of 300 and 400 °C (Figure 4b). Here, the redox potential was increased in the first 8 days, and it was in the range of 590–605 mV. On the 7th day,  $\text{Fe}^{2+}$  was completely oxidized, while  $\text{Fe}^{3+}$  remained in the same range of 3.4–4.2 g/L (Figure 4c). The total iron content in the solution was decreased, which can be caused by its active precipitation in the form of jarosite. At the same time, the population of bacterial cells was increased from  $6 \times 10^7$  cells/mL to  $12.5 \times 10^7$  cells/mL in all experiments, which indicates that a decrease in the pH values during the experiment was not an inhibitory factor for this bacterial strain. In just 14 days of the bacterial leaching, 19.2% of Cu and 23% of Ni were recovered from the residue after roasting at 200 °C, 7.54% of Cu and 5.84% of Ni from the residue after roasting at 300 °C, while 22.2% of Cu and 16.4% of Ni from the residue after roasting at 400 °C.



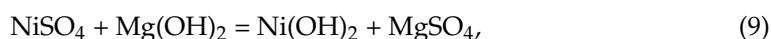
**Figure 4.** Bioleaching parameters after roasting with ammonium sulfate at 200, 300, and 400 °C ((a)—pH values, (b)—redox potential values, (c)—Fe ions concentrations).

For the processing of pregnant solutions, a technological scheme was proposed consisting of three stages. The first stage includes the extraction of copper from the pregnant solution by its cementation on iron. Cementation on iron is the most common method of extracting copper from low-concentrated sulfate solutions, which is important for the processing of waste and low-grade ores. The main reaction of copper cementation is the following process:



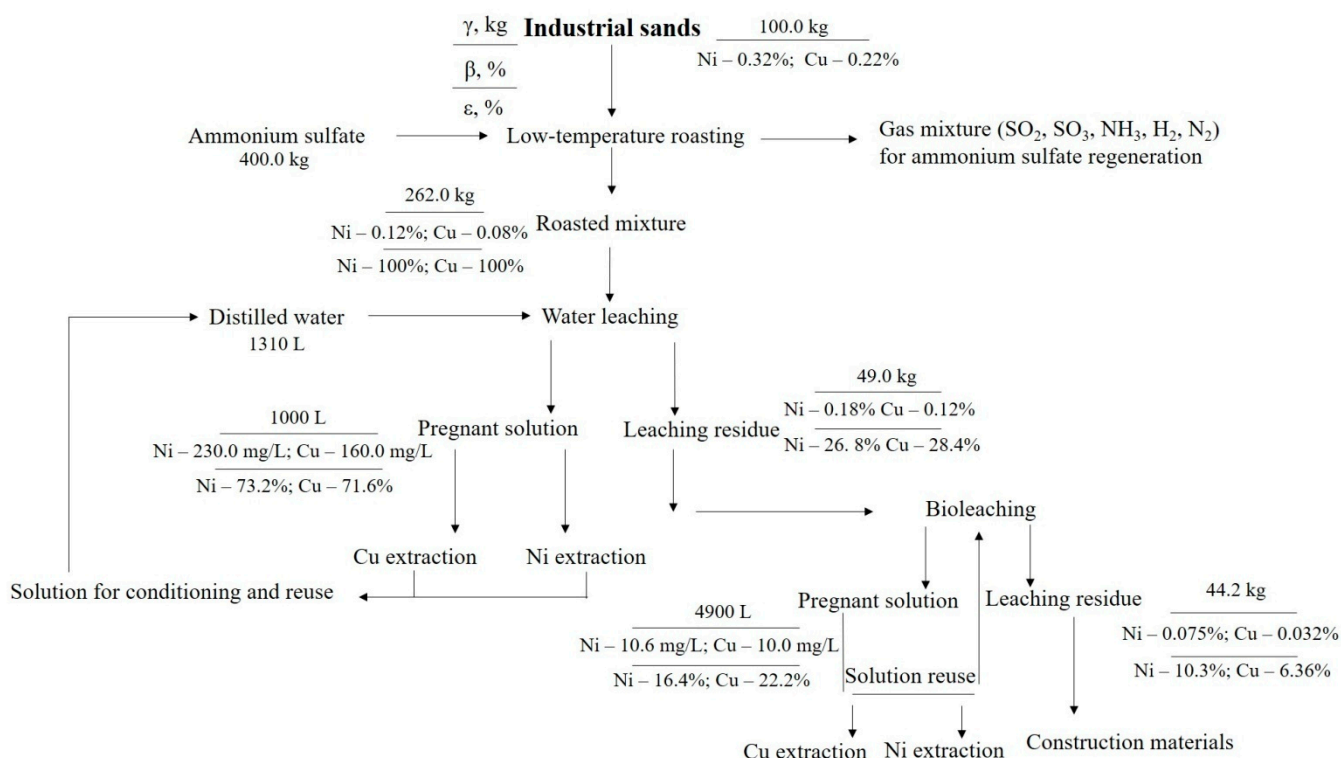
The use of the cementation method is especially important for small-scale production (up to 5 thousand tons/year) when the use of extraction methods for concentrating solutions

is not economically feasible. With the full mechanization of the cementing plant, the solution is sent for further processing, while cement copper is sent to a vacuum filter or filter press. In this case, the final product is obtained in the form of cement copper powder. The second stage consists of the precipitation of iron from the solution after the cementation. In the final third stage, it is expedient to precipitate nickel from the solution by raising the pH value using magnesium hydroxide.



### 3.4. Technology Flowsheet for the Industrial Sands Processing

According to the research results, a basic technological flowsheet for the processing of technogenic sands containing copper and nickel was proposed. The flowsheet included the following technological steps (Figure 5). The first stage is the roasting of the industrial sands and ammonium sulfate mixture at a mass ratio of 1:4 and at a temperature of 400 °C for 240 min. Subsequent aqueous leaching leads to the formation of pregnant solutions saturated with nickel and copper ions. Pregnant solutions can be further processed by traditional methods in order to obtain copper and nickel as commercial products. The off-gases at the roasting stage can be captured, and the composition of the gases indicates the possibility of ammonium sulfate regeneration; this measure will reduce the cost of reagent purchasing and minimize the impact on atmospheric air. After the water-leaching stage of the roasted mixture, a residue is obtained containing 0.18% of nickel and 0.12% of copper. The second stage of the technological process is the bioleaching of this residue in order to obtain pregnant solutions. The residue after bioleaching can be further used as an additive in the building mixture production, the metals from the pregnant solution are selectively extracted into the commercial products, while the solutions after conditioning are proposed to be returned to the leaching stage.



**Figure 5.** Technology flowsheet for the industrial sands processing (γ—product weight, β—metal content, ε—recovery).



#### 4. Conclusions

The results obtained in this study showed the possibility of using a combined method for industrial sand processing.

The development and selection of combined technological pathways for the extraction of metals are caused by a number of problems, such as a decrease in the quality of ores, the inability to use the traditional pyrometallurgical processing methods, increasing demand for metals, the environmental impact of the mining industry, etc. [9]. A large amount of literature indicates that significant efforts have been made in order to involve the various types of low-grade ores and industrial waste in processing [11,13,15,22,34–36].

The research [17] on sustainable biological leaching of low-grade chalcopyrite ore showed that the use of low-grade chalcopyrite ore was improved by increasing the number of microbes. The shorter dormant cycle contributed to the increase in the redox potential value during irrigation. Intermittent irrigation has been shown to enhance the dissolution of chalcopyrite ore, but the excessively long periods of dormancy adversely affect the chemical and bacterial activity due to a lack of acid and/or microbial nutrients [17].

The authors [37] considered the bioleaching of gold from the mine tailings using the *Alcaligenes faecalis*. It was shown that using only bacteria as a treatment, the Au yield was quite similar to that of NaCN, indicating that the use of *A. faecalis* is a promising pathway for the gold leaching from tailings.

In a study of static and percolation leaching with alkaline pre-treatment, alkaline bacteria easily removed oxidized minerals such as copper oxide and sulfates, as well as metals that coated the mineral sulfides surface. Subsequent acidic bioleaching made it possible to decompose the remaining sulfide minerals. The highest copper yield of 67% with alkaline bioleaching was achieved after 9 days, while the subsequent addition of nutrients in the acid medium led to an increase in the copper yield to 74% [38].

The authors [39] proposed the sequential biological leaching of pyrite wastes, as well as the leaching of non-ferrous iron-containing slags, as a promising method for mining waste processing. After the acid leaching stage, followed by 12 days of bioleaching, the total recovery of metals from pyrite tailings into the liquid phase was 68% for copper, 77% for zinc, and 75% for iron; recovery of gold by the cyanidation method has reached 92%.

The research on the industrial sand's bioleaching feasibility showed the effectiveness of this pathway. An increase in the bacterial population during the experiment was observed, while the recovery of copper and nickel with the assistance of bacteria exceeded the recovery from the sterile control by two times. In the process of the industrial sands and ammonium sulfate mixture roasting at a temperature of 400 °C, more than 70% of non-ferrous metals were recovered. A distinctive feature of the technology is the possibility of reagent regeneration. The composition of the off-gases during the roasting process will allow for the regeneration of ammonium sulfate, not only reducing the cost of the reagent purchasing but also minimizing the negative environmental impact.

The possibility was examined of recovering non-ferrous metals by a combined process, including the low-temperature roasting of industrial sands, followed by the additional recovery of non-ferrous metals by the bioleaching using the *Acidithiobacillus ferrovorans* bacterial strain. The recovery of non-ferrous metals was increased up to 90%. Further optimization of the bioleaching process conditions will make it possible to avoid the precipitation of iron in solution in the form of jarosite, which can increase the recovery of non-ferrous metals.

The results obtained demonstrate the prospects of processing the industrial sands based on an additional economic benefit due to the absence of grinding costs. The implementation of the proposed concentration process can significantly increase the mineral resource base of non-ferrous metals. The considered processing methods, subject to optimization, can also be considered promising for processing low-grade ores due to the high selectivity, the relatively low energy consumption, and the low cost.

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**Conflicts of Interest:** The authors declare no conflict of interest.

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