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Complex Ni and Co Extraction from Leached Nontronitized Serpentinite via Hydrometallurgical Process at Atmospheric Pressure

Abstract: The growing significance of mineral processing processes in the contemporary mining and processing industry lies in their aim to augment the concentration of valuable constituents within processed natural raw materials. Predominantly, these processes encompass pyrometallurgy, hydrometallurgy, and electrometallurgy. Each method can extract nickel; however, the high energy demand of pyrometallurgy necessitates a shift toward refining the efficiency of hydrometallurgical procedures. Consequently, optimizing nickel-containing ore processing technologies to yield high-purity nickel and its compounds is a current research concern. This paper investigates the hydrometallurgical processing of nickel and cobalt via leaching, specifically targeting oxidized nickel-cobalt ore. Analytical studies reveal a promising potential for complex extraction of nickel and cobalt, achieving impressive yields of 98.4% and 90.7%, respectively. Our analysis further demonstrates that implementation of this method can notably elevate the extraction degree of targeted metals. Significant energy resource conservation is made possible by conducting extraction at a moderate temperature of 353K and at atmospheric pressure. Importantly, this approach facilitates the extraction of valuable components even from ores containing negligible concentrations. This scientific endeavor enhances our understanding of efficient, sustainable practices in the field of mineral processing, demonstrating the potential of hydrometallurgical methods for nickel and cobalt extraction.

Keywords: hydrometallurgy, nickel and cobalt extraction, leaching process, energy efficiency, mineral processing.

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Introduction

Nickel, also called the "green" metal, has a large commercial demand that is growing (Tsai & Chan, 2013; Torabi & Ahmadi, 2020; Shukla et al., 2001) because of many appealing properties, including ductility, corrosion resistance, resistance, magnetism (Morcali et al., 2017; Abikak et al., 2023), and high-temperature resistance (Li et al., 2017; Zhapbasbayev et al., 2021). Cobalt is also an equally strategic metal used in many commercial, industrial, and military applications.

Kazakhstan ranks 7th in the world regarding the total cobalt reserves. The state balance sheet records 55 deposits of cobalt[1]. The most significant reserves are held by such companies as KAZNICKEL LLP, Kyzyl Kain Mamyt LLP, and Sokolov-Sarybai Mining Production Association (SSGPO) JSC[2]. The Republic of Kazakhstan has a number of large nickel deposits: Gornostayev (173 thousand tons/nickel) in the Abay region, Shevchenko (1 million tons), Kundybay, Podolsk, Zhitikara, Akkarga, Milyutin, and Ekibastuz-Shiderti (262 thousand tons) in the Pavlodar region and others[3]. The deposits belong to the formation of iron-manganese-cobalt-nickel ores

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[1] Order of the Minister of Investment and Development of the Republic of Kazakhstan from June 28, 2018 № 478 "On approval of the Program of state subsoil fund management". URL: https://online.zakon.kz/Document/?doc_id=33233470&show_di=1&pos=5;-106#pos=5;-106 (date of reference: 02.05.2023).

[2] Best Available Techniques Information and Technology Handbook 12-2019 Nickel and Cobalt Production. URL: http://www.normacs.ru/Doclist/doc/11P04.html (date of reference: 06.06.2023).

[3] Production of nickel-cobalt products in the Republic of Kazakhstan: market research. URL: https://damu.kz/upload/iblock/51c/MarketingovoeIssledovanie_ProizvodstvoNikel_kobaltovovoyProduktsiiVKazakhstane.docx (date of reference: 20.06.2023).

and are represented by three types of ore minerals: asbolane-hydrohematites, kerolite-nontronite-ochras, and nickel-cobalt-melanites. The content in the rocks: Fe 13-33 %, Ni 0.1-0.66 % and Co 0.06-0.35 %. Developed and presented for extraction nickel-containing ores contain mainly metal oxides in the form of Fe_2O_3, NiO, and CoO. Today, about 73 % of nickel use is for the production of stainless steels, which are of high quality and long life, and nickel is also used to fabricate batteries for electric cars and other electrical applications (Olafsdottir & Sverdrup, 2021). The cobalt that accompanies nickel is also a very important component in the production of nickel-containing lithium-ion batteries; it improves their quality and service life (Li & Lu, 2020; Yukun et al., 2021; Miryuk, 2024). Oxidized (lateritic) nickel ores contain about 70 % of the world's geological reserves of nickel and cobalt. The ferronickel production stage is energy-intensive and is mainly used for oxidized ores that are transported to metallurgical plants (Selivanov & Sergeeva, 2019). Processing one ton of ore produces 1.5-2.5 kilograms of nickel, requiring the burning of coal and generating slag waste that needs to be disposed of. Currently, it is very important to develop an affordable technology for processing oxidized nickel ores that would result in minimal environmental impact.

This study (Ivanov et al., 2022) is devoted to solving this problem. The paper presents a study of the agglomeration, roasting, and leaching of oxidized nickel ores with the choice of optimal parameters, which include the addition of various reagents, varying the temperature, duration, and program of roasting, and creating the necessary atmosphere in the roasting furnace. Analysis shows that only a small fraction of ferrous minerals forms ferrous sulfate when sulfated with sulfuric acid, even at elevated temperatures. The loss of nickel bound to oxygen by iron compounds can be reduced after roasting at temperatures no higher than 6500C, during which the iron oxides recrystallize to form α-Fe2O3 while nickel is released, which can be leached. The sintering and roasting process can achieve higher recoveries of nickel and cobalt, which are components of the various ore minerals that are available for extraction at different stages of ore processing.

About 60 % of nickel is extracted from sulfide-nickel ores, which are depleted due to over-exploitation, and hard-to-process poor ores have now become the main raw material for nickel mining (Chen et al., 2017; Guo et al., 2009; He et al., 2013). When the ore is processed by the traditional pyrometallurgical smelting process, that is, primary ore (flotation) → nickel concentrate (high-temperature smelting) → low-grade nickel matte (converter smelting) → high-grade nickel matte (milling and flotation separation or sulfuric acid leaching or high-pressure ammonia leaching process) → nickel and copper products (Park et al., 2006), the high concentration of waste rock in the flotation process produces large amounts of sludge, making difficult the separation of the metallic minerals (Fu et al., 2010; Lu et al., 2000). Conversion of low-grade nickel matte into high-grade nickel matte results in low recovery of valuable metals and low economic benefits throughout the process (Li et al., 2017; Mu et al., 2018).

Recent research on the recovery of valuable metals from low-grade nickel matte has been conducted mainly in hydrometallurgy, namely atmospheric pressure leaching (Xiao et al., 2020), high-pressure leaching (Sinisalo et al., 2021), oxygen pressure leaching (Behnamfard et al., 2013), acidic iron chloride leaching (Cui et al., 2018) and oxidation-ammonia leaching (Chen et al., 2014). Atmospheric leaching has the advantage that the leaching of valuable metals can be significantly lower and is improved by adjusting certain process parameters such as temperature and particle size.

The hydrometallurgical process, as compared to pyrometallurgy, significantly improves the recovery of some metals, reduces energy consumption, is more environmentally friendly, and easily provides continuity and automation of equipment, but the overall recovery of valuable metals is low (Chen et al., 2017; Li et al., 2018). Sulfuric acid firing is considered to be a promising process because of its low reaction temperature, high reactivity and efficiency, and proven exhaust gas recovery technology.

There is a way of complex processing of nickel-cobalt raw materials (Nesterov et al., 2010), where autoclave leaching of raw materials is carried out by nitric acid solution (HNO2) with concentration 100-150 g/l, at temperature 200-2500C, pressure 4.0-6.0 MPa, ratio T:L = 1: (2-4) and within 1.5-2.0 hours. The extraction of cobalt and nickel from raw materials is 96.6 % and 90.3 %, respectively. Despite the high degree of extraction of target metals, this method has a number of drawbacks: (1) significant power consumption due to maintaining high temperatures during extraction; (2) the need for complex hardware design due to the use of autoclave leaching.

The method of hydrometallurgical processing of oxidized nickel-cobalt ores (Duisebayev et al., 2014), based on ore leaching with sulfuric acid with a concentration of 200 g/dm3 at 900C±50C and the use of gaseous sulfur dioxide in an amount of 1.4-3.0 vol. % of the pulp volume is also used. Treatment of ore with sulfur dioxide makes up 5-7 % of the leaching time, and the process of ore leaching takes 60 minutes. The disadvantages of the method include extremely low cobalt extraction of 58.5 % and significant energy costs due to the high-temperature maintenance on a constant basis.

Analyzing literature sources describing methods of nickel and cobalt extraction from ores, we conclude that the existing technologies have a number of serious drawbacks. In addition, there is a need to find suitable solutions related to the development of new as well as optimization and improvement of nickel-containing ore processing technologies.

The purpose of this paper is to develop an effective, simplified, and accelerated method of complex extraction of nickel, cobalt, and associated metals from refractory oxidized ore that excludes high-temperature, autoclave, energy-intensive processes requiring a complex hardware design. The goal of this research is to achieve a high degree of extraction of target metals at 353 K by using available, less toxic, and efficient local reagents.

Methods

In this study, our focus was on the leached nontronitized serpentinite ore, specifically extracted from the Gornostayev deposit situated in the Abay region. The ore was subjected to meticulous preparation before the experimental procedures commenced. The primary step in the sample preparation process involved the reduction of the ore to a manageable size. This was accomplished by crushing the ore to a fraction range of 45 to 0.6 microns. The equipment utilized for this purpose was a planetary mono-mill Fritsch Pulverisette 6, originating from Germany, known for its precision and consistency in reducing the sample size while minimizing contamination.

Following the size reduction process, we subjected the samples to a detailed structural analysis by X-ray diffraction (XRD). The XRD imaging was executed with the assistance of a D8 Advance (Bruker) equipped with an α-Cu radiation source operating at a tube voltage of 40 kV and current of 40 mA. This instrument was selected for its unrivaled accuracy and superior resolution capabilities. The obtained diffractograms were analyzed, and the interplanar distances were computed with the help of EVA software, a highly regarded tool in the realm of XRD data analysis. Further steps included the generation of sample transcripts and the conduction of phase searches. For these procedures, we used the Search/match feature in combination with the comprehensive PDF-2 powder diffractometric database, ensuring a thorough examination of the structural components of the samples.

For a more nuanced understanding of the crushed ore, we also obtained high-resolution images using a Quanta 200i 3D scanning electron microscope (SEM) from FEI Company, USA. The SEM provided in-depth morphological details of the crushed ore particles, allowing us to observe the physical characteristics at a microscopic level. Moreover, the material composition of the ore was investigated via a Leica DM2500 P optical microscope. This powerful tool offered detailed insights into the mineralogical composition of the sample, paving the way for a deeper understanding of the leaching behavior.

The last stage of the analysis focused on the measurement of nickel and cobalt content in the hydrochloric acid solution post-leaching. An 8300 DV inductively coupled plasma emission spectrometer, a product of PerkinElmer Inc., was employed for this purpose. This instrument is well-recognized for its sensitivity, robustness, and high precision in trace elemental analysis, making it ideal for determining the content of nickel and cobalt in the solution.

Research experiment

The subject of our investigation was an oxidized nickel-cobalt-containing ore from the Gornostayev deposit. Amongst the variety of potential ore samples, this specific sample was selected for its challenging processing attributes by existing industrial techniques, which thereby exemplifies the pressing need for novel methodologies.

When planning efficacious solvents for the extraction of nickel and cobalt from such ores, an understanding of the compounds' forms in both the ore and their processed products is crucial. This is due to the varying reactivity of different chemical states of nickel and cobalt, which thereby affect the efficiency of their extraction. Therefore, to advance a new technology for the effective extraction of nickel and cobalt from nickel-cobalt-containing ore, a comprehensive knowledge of the phase composition of the studied material is imperative.

An X-ray diffraction (XRD) analysis was conducted to elucidate the mineralogical composition of the ore. The diffractogram of the sample, as demonstrated in Fig. 1, provides a visualization of the material's crystalline phases. Our X-ray phase analysis unveiled that the oxidized ore sample predominantly contains maghemite (77.5%), followed by hematite (12.8%), and kaolinite (9.7%), as outlined in Table 1.

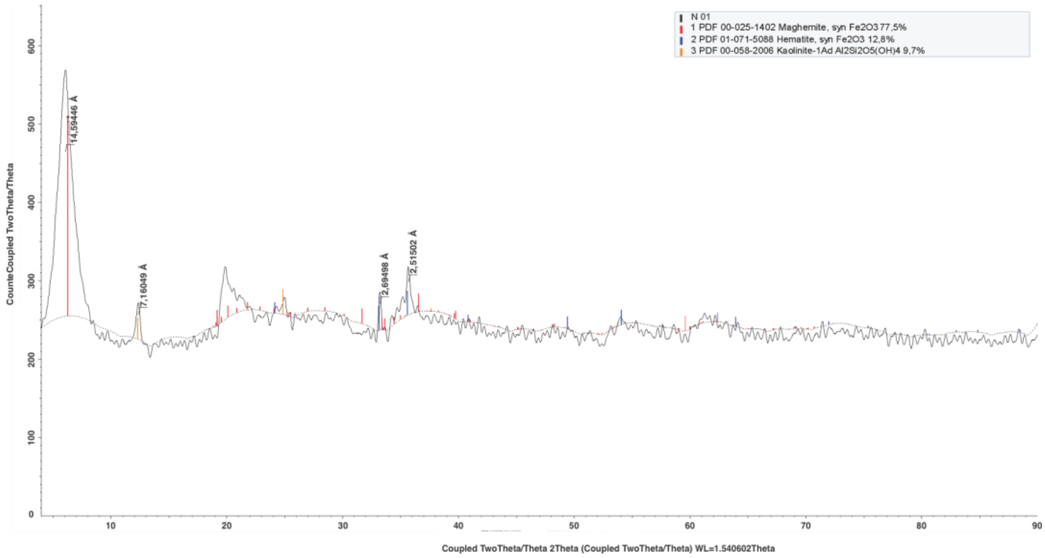

Fig. 1. Diffractogram of oxidized nickel-cobalt ore before leaching

Table 1. Phase composition of oxidized nickel-cobalt ore “before” leaching, determined on the basis of X-ray phase analysis

Sample	Name of compounds	Formula	Percentage content, %
Ore No. 1	Maghemite	Fe_2O_3	77.5
	Hematite	Fe_2O_3	12.8
	Kaolinite	$Al_2Si_2O_5(OH)_4$	9.7

Post-leaching, it was observed that the crystalline structure of the nontronite mineral, initially identified as kaolinite, was eradicated. This destruction implies that the encapsulated nickel and cobalt ions were successfully released from the solid phase and migrated to the solution, as visually represented in Fig. 2. The respective contents of maghemite and hematite post-leaching are elucidated in Table 2. The transitions of these mineral phases provide significant insights into the leaching process's effectiveness, advancing our understanding of the leaching dynamics of nickel and cobalt from such ores.

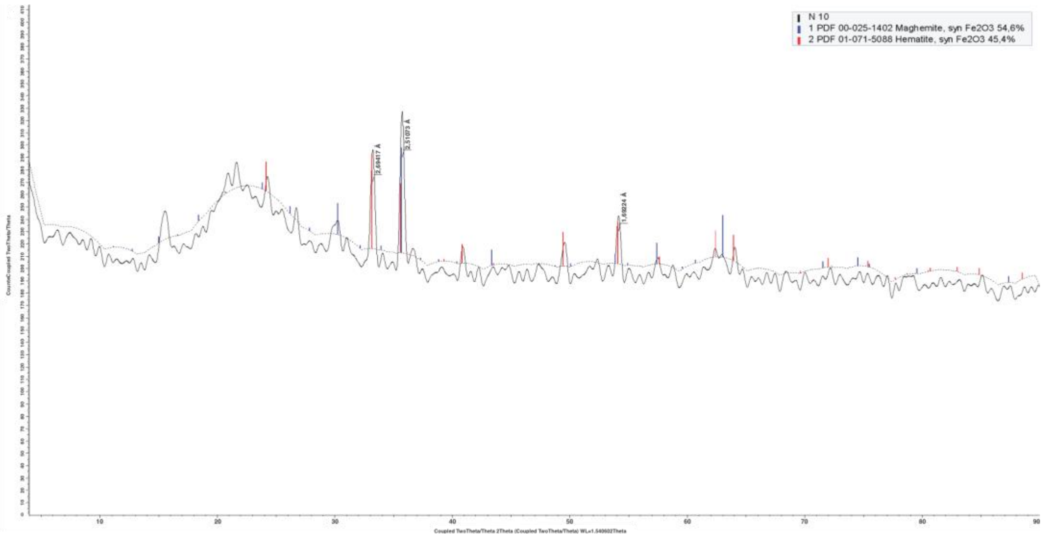

Fig. 2. Diffractogram of oxidized nickel-cobalt ore after leaching

Table 2. Phase composition of oxidized nickel-cobalt ore "after" leaching, determined from the X-ray phase analysis

Sample	Name of compounds	Formula	Percentage content, %
Ore No. 1	Maghemite	Fe_2O_3	54.6
	Hematite	Fe_2O_3	45.4

Chemical analysis of the ore showed that the refractory oxidized ore contains 0.98 % Ni and 0.05 % Co, which are bound in the oxidized ore in the form of the main mineral – kaolinite ($Al_2Si_2O_5(OH)_4$). The chemical composition of the ore is shown in Table 2. It should be noted that the above minerals in HCl are relatively difficult to dissolve with the formation of gelatinous silica. As can be seen from the analysis, nickel and cobalt are associated with hardly soluble silicates. The elemental composition of the oxidized nickel-cobalt ore is shown in Table 3.

Table 3. Elemental composition of oxidized nickel-cobalt ore

Component	**Na**	**Mg**	**Al**	**Si**	**P**	**S**	**K**	**Y**	**Ca**
Content, %	0.49	8.45	1.45	19.50	0.02	0.05	0.04	0.004	0.65
Component	**Ti**	**V**	**Cr**	**Mn**	**Fe**	**Co**	**Ni**	**Sr**	**Zn**
Content, %	0.01	0.02	0.58	0.20	13.14	0.05	0.98	0.01	0.01

The material composition was determined by a Leica DM2500 P microscope. The sample was studied in immersion liquids with different refractive indices. Liquids with refractive indices characteristic of nontronite (kaolinite) from N=1.550 to N=1.600 were selected. The refractive index of nontronite (kaolinite) is about 1560. The content of nontronite, defined as kaolinite, according to X-ray diffraction analysis is 9.7 %. Kaolinite containing Fe belongs to the nontronite group.

Nontronite is a clayey material, a sheet silicate of the montmorillonite group (smectite group), with the formula $(Fe,Al)_2[Si_4O_{10}][OH]_2 \times nH_2O$. Usually contains significant amounts of Al_2O_3 (up to 14 %) and Mg (up to 8 %), also CaO (up to 2 %), in small amounts of K_2O, Na_2O, sometimes NiO, Cr_2O_3. Cobalt and nickel in this ore are isomorphically related to the mineral badellite $Al_2[Si_4O_{10}][OH]*nH_2O$. The mineral has a brownish-green color. The presence of iron oxides gives it a brownish hue. In the immersion preparation, the product has a leafy form (see Fig. 3).

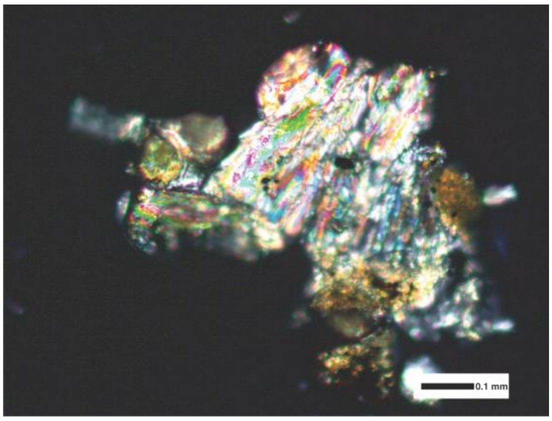

Fig. 3. Nontronite in the immersion preparation. Magnification 100, Nicoli +

Serpentine forms nests in nontronite rock. Serpentine as a radiating aggregate of grains, nontronite is present as a cryptocrystalline aggregate.

According to the refractive index, serpentine belongs to group IV, so the relief and shagreen surface are weak. In thin sections the mineral is colorless or yellowish-greenish, but in colored varieties the pleochroism is absent or hardly noticeable.

Serpentine is similar to chlorite in optical properties, and differs from it in mineral association. Serpentine develops mainly from olivine, rhombic pyroxene, and chlorite by monoclinic pyroxene, hornblende, biotite (see Fig. 4).

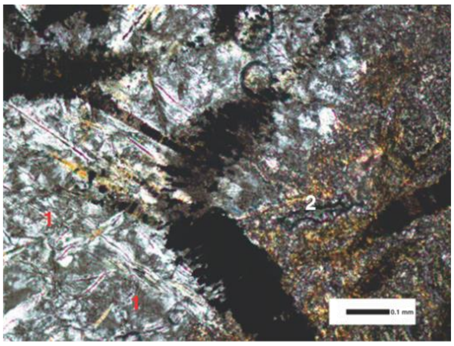

Fig. 4. Serpentine (1)-nontronite rock (2). Transparent thin section, magnification 100, Nicoli +

Scanning Electron microscope images at different scales of crushed particles of oxidized nickel-cobalt ore are shown in Figure 5. As can be seen from Figure 5, the size of most of the particles is not bigger than 45 microns and are mostly aggregates of ultrafine particles, but there are also smaller particles with an average diameter in the range of 100 to 620 nm.

As shown by the chemical analysis of the studied ore, the silicate content in the ore is quite high (19.5%, Table 2), which in turn complicates the dissolution process. Therefore, a cheap but effective reagent - ammonium fluoride (NH_4F) – was chosen for the effective destruction of silicate groups.

It should be noted that in order to achieve optimal extraction of the target metals it is necessary to consider the optimal parameters affecting the dissolution process, such as the concentration of dissolving reagents, solid-to-liquid ratio, stirring rate, and leaching duration, and temperature (Ospanov et al., 2020; Ospanov et al., 2021). In addition, orthogonal scheduling of the experiment of the second order with a "star shoulder" ($\alpha = \pm 1.215$), where it is necessary to change only one particular factor while keeping the others strictly constant, was used to select the optimal conditions for the complex extraction of nickel and cobalt from the oxidized ore (Smailov et al., 2022).

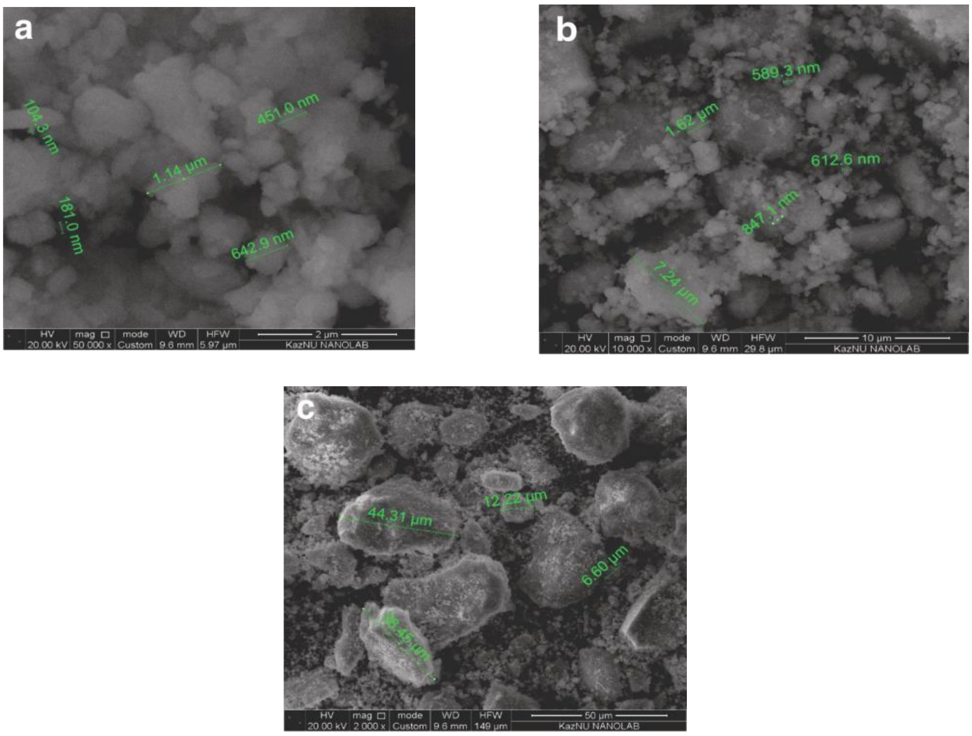

Fig. 5. Scanning electron microscope images of oxidized nickel-cobalt ore at magnifications of (a) 2 µm, (b) 10 µm, (c) 50 µm.

During the experiment, a sample (5.0 g) of oxidized nickel-cobalt ore was placed in a sealed reaction vessel of 250 cm^3, then added 100 cm^3 of aqueous solution containing 15 cm^3 of concentrated hydrochloric acid. After every 60 minutes, 1 g of ammonium fluoride was added under constant stirring at 250 rpm with a

T:L ratio of 1:20 for 300 minutes. The temperature was gradually raised from 273 K to 353 K and maintained at this temperature until the end of the dissolution process. The resulting hydrochloric acid solution was filtered through a medium-density filter and the precipitate was washed 5-6 times with 1 mol/dm^3 hydrochloric acid, and the filter with the cake was discarded. Then, the filtrate was analyzed to determine the content of valuable components by using a PerkinElmer Inc. 8300 DV inductively coupled plasma emission spectrometer.

This experiment was conducted in 4 parallels and their error was less than ±5%. Table 4 below shows the results of the search for optimal conditions of nickel and cobalt extraction, taking into account the various parameters mentioned above.

Table 4. Degree of Ni and Co extraction depending on various factors

NH_4F concentration, %	**Ni extraction rate, %**	**Co extraction rate, %**
2.0	84.2	82.7
3.0	87.7	84.2
4.0	91.5	87.6
5.0	**98.4**	**90.7**
6.0	96.2	91.5
Temperature, K		
298	58.2	52.7
323	71.7	64.2
343	84.5	83.6
353	**98.4**	**90.7**
363	96.5	91.8
HCl concentration, %		
5	51.4	47.5
10	71.1	63.3
15	**98.4**	**90.7**
Duration of the experiment, min		
120	49.0	46.3
180	68.5	54.2
240	88.2	82.7
300	**98.4**	**90.7**
360	96.4	91.5
Ratio of solid to liquid (S:L)		
1:10	64.6	54.0
1:15	83.2	77.4
1:20	**98.4**	**90.7**
1:25	96.7	91.5
Stirring speed, rpm		
200	88.7	83.5
250	**98.4**	**90.7**
300	96.6	91.4

Research Results and Discussion

From the results tabulated in Table 4, it is deducible that optimal conditions for the recovery of nickel and cobalt have been determined. These include the following parameters:

- a concentration of 5% and 15% for the dissolving reagents NH_4F and HCl respectively;
- an experimental duration of 300 minutes;
- a solid-to-liquid ratio (S: L) set at 1:20;
- a stirring speed maintained at 250 rpm.

Under these stringent yet necessary conditions, the extraction efficacy of nickel was observed to be 98.4% while that of cobalt reached 90.7%. It is worth mentioning that deviations below these critical values resulted in diminished extraction efficiency for both target metals.

These findings underscore several salient advantages of the proposed method over existing approaches. Firstly, the method utilizes cost-effective and readily available reagents of less toxicity that are sourced locally. Secondly, the technique significantly enhances the extraction efficiency of nickel and cobalt from refractory oxidized ores. Thirdly, the incorporation of NH_4F in HCl enables extraction to be undertaken at 353K under normal atmospheric pressure, thus accommodating ores of diverse compositions. Additionally, this method not only achieves extraction of nickel and cobalt but also facilitates the recovery of other valuable associated metals in a comprehensive manner.

Based on the results obtained, a novel scheme of hydrometallurgical processing of oxidized nickel ores has been proposed. This includes the following steps: the ores are first crushed and ground from 45 microns down to 0.6 microns, then subjected to flotation for enrichment. Subsequently, the ore undergoes leaching at 353K with an NH_4F-based composition in HCl. The dissolved ore is then filtered before precipitation of nickel, cobalt, and associated metals occurs. The filtrate is then cycled back to the start of the process. The final steps encompass the decontamination and neutralization of filtrate, and the resultant cake is either disposed of securely or repurposed within the construction industry. Figure 6 provides a graphical illustration of this proposed technological scheme of complex extraction of nickel and cobalt from oxidized nickel-cobalt-containing raw materials.

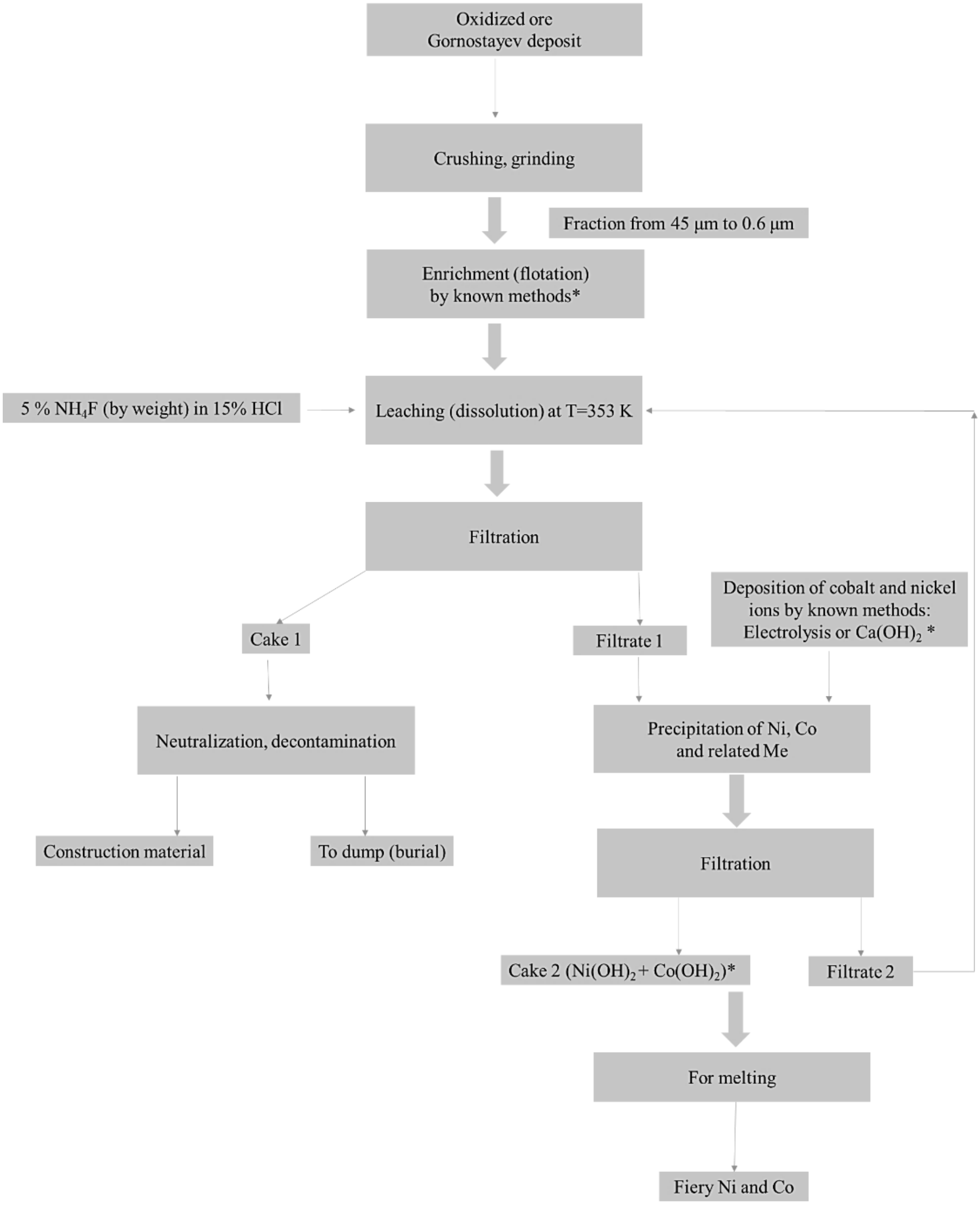

Fig. 6. Basic technological scheme of complex extraction of nickel and cobalt from oxidized ore of Gornostayev deposit

In summary, this research paper proffers an innovative, environmentally friendly, and cost-effective scheme for the comprehensive extraction of nickel and cobalt from oxidized nickel-cobalt-containing ores, using local reagents of less toxicity. The optimal conditions for the dissolution of nickel and cobalt-bearing minerals have also been precisely determined.

Conclusions

In the contemporary economic climate, characterized by acute oscillations in global market prices of nickel and cobalt, an exigent issue arises regarding the economically viable processing of low-grade oxidized nickel ores indigenous to Kazakhstan. In an endeavor to address this, we proposed a novel and more promising hydrometallurgical methodology as a feasible alternative to the extant ore processing techniques being implemented within the nation.

The novelty of our technology revolves around the comprehensive extraction of nickel and cobalt from oxidized nickel-cobalt-bearing resources. This innovative approach is distinctive in its obviation of the high-temperature process. Conventionally, this process is notorious for the emission of noxious gases and has a proclivity for energy profligacy. Our method, thus, not only minimizes environmental hazards but also underscores energy conservation - features that are instrumental in driving the transition towards sustainable mining practices.

Upon meticulous analysis of the experimental data gleaned from our research, we ascertain that our groundbreaking technology facilitates enhanced extraction efficacy of nickel and cobalt from refractory oxidized ores. We therefore postulate that the deployment of this technology in industrial-scale operations can potentially revolutionize the nickel and cobalt mining landscape by augmenting metal recovery rates, thereby maximizing resource utilization and industrial efficiency.

In summary, our work is not only responsive to the immediate challenges posed by fluctuating commodity prices, but it also sets a benchmark for future endeavors in the field, thus pushing the frontier of sustainable mining practices. Further research will be instrumental in understanding the scalability of this innovative technology and its broader implications for the industry.

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