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INFLUENCE OF GRINDING EXTENT OF NICKEL-CONTAINING ORE RAW MATERIALS ON EXTRACTION OF METALS AT HYDROCHLORIC ACID LEACHING

Abstract. This paper presents the characteristics of ore raw materials with the application of modern methods of physical and chemical analysis, technological features of preliminary preparation of raw materials in the process of nickel leaching and the choice of the optimum degree of grinding the ore for a hydrochloric acid leaching of specified raw material.

Keywords: oxidized nickel-containing ore, ore preparation, grain-size class, leaching, solvent.

Introduction. In Kazakhstan, nickel-containing raw materials are mainly presented by the poor oxidized ore materials (for example, Kempirsay deposit) and iron-containing ore materials and concentrates (Sokolovskoye and Sarbaiskoye deposits). Low concentration of nickel in ore materials, the complexity of the mineralogical composition and intergrowth of minerals require special preparation of crude ores for metallurgical processing.

Taking into account the high demand of nickel in the global markets, as well as stocks of raw materials in Kazakhstan, the study of the preparation of nickel-containing ores to metallurgical processing is an urgent task.

Pyrometallurgical and hydrometallurgical methods are used for the processing of nickel-containing raw materials. But because of the low quality of Kazakhstani nickel-containing raw materials, pyrometallurgical methods are generally unsuitable for processing ores in Kazakhstan. Therefore, combined and hydrometallurgical methods are widely used [1, 2].

The most energy intensive and time taking processes in the production technology of dispersed materials are operations on reducing the size of the piece goods, because the costs of these processes in prime cost of mining and processing stage is 30-50%. The combination of the ore processing by a variety of methods for obtaining coarseness of grading and material composition, determined by the requirements of the process stage or regulations for finished products, is called the process of ore preparation. In general, in mining plants on processing ores of non-ferrous and ferrous metals, also in chemical production this stage precedes the stage of ore concentration [3, 4].

As Figure 1 shows, ore preparation stage comprises the following steps:

1) ore breaking involving mechanical processes, as a result, extracted massive rock is crushed to a size suitable for further size reduction by milling. Devices that crush raw materials are considered as primary crusher; roll-jaw and cone-shaped crushers are basic ones;

2) preliminary grinding of ore is a preliminary step of obtaining the required size material. Usually performed in an aqueous medium by means of machines, in which the rock is grinded by means of cobble boulders produced from solid parts of ore and enclosing rocks;

3) primary ore grinding is the final stage of size reduction of the piece goods. Generally, at this stage grinding is performed in an aqueous medium by means of machines in which the rock is grinded with the help of cast iron or steel balls. As a result, a fine pulp with a high concentration of particles of grain-size class – 0.074 mm (about 70-80% of the total weight of the fine pulp) will be formed;

4) the classification of the ore which is necessary for the preparation of a specific dimension material for concentration. As a result, a large fraction of the fine pulp, which is returned to the regrinding process, will be extracted.

Figure 1 – The structure of the ore preparation stage

Characteristics of the initial nickel raw materials. In order to perform the experiments there was used oxidized nickel and cobalt containing ore, which is represented by green gray wizened claypans with light colored impregnations. The ore was grinded to 0.2 mm minus class.

After homogenization of the whole ore mass based on a "cone-ring" method and the consistent reduction of quartering method there were taken samples to determine the material composition of the ore using mineralogical, X-ray diffractometric, spectral and chemical analysis methods.

Technological sample consists of small fragments of rocks of various compositions, with domination of metamorphically altered serpentines from green gray to a dark green color, sometimes pigmented in brown with iron hydroxides. The structure of debris is uneven – from grainy to colloformic-zoned with replacement signs of the cerpentin-chrysotile formations nontronite montmorillonite (smectite) with finely divided (0.005-0.01 mm) grains of magnetite and colloformic-zonal formations of clay material [5, 6].

X-ray diffractometric analysis is performed on a DRON-2 diffractometer with Cu-radiation, beta-filter. Diffractogram survey was performed under the following conditions: U = 35 kV; 1 = 20 mA; Scale - 2000 imp; time constant – 2 seconds; survey – 2 theta; detector – 2 °C/min. X-ray diffraction analysis on the basis of semi-quantitative determination of the proportion of crystalline phases was carried out by diffractograms of powder samples using method of equal test charge and artificial mixtures.

For the interpretation of diffractograms we used ASTM Powder diffraction file data diffractograms of pure minerals. For the main phase we conducted contents calculation. Possible impurities, which identification cannot be valued because of the small content and the presence of only 1-2 diffraction reflections or poor crystallinity are given in diffractogram (Figure 2). The identification of mineral phases by X-ray analysis is given in Table 1.
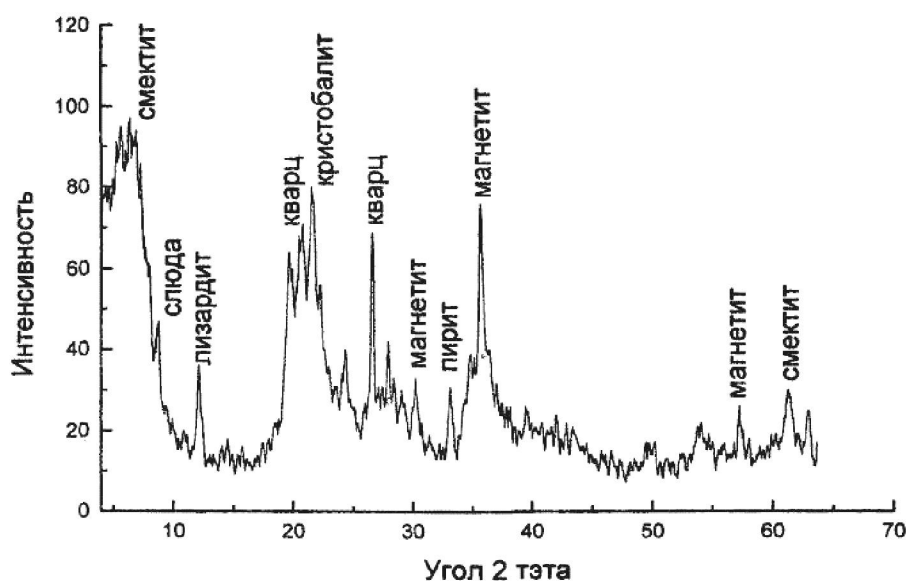

Figure 2 – Diffractogram of oxidised nickel-cobalt ore

According to the results of the physical and chemical studies of ore, mineral composition of technological sample was calculated, masses %: smectite 60-62; serpentine group 8-10; quartz 2-4; cristobalite 10-11; mica 3-5; feldspar 1-2; magnetite 12-13; pyrite 2-3.

Rock-forming minerals are presented by smectite (SMS), the serpentine group minerals (antigorite, lizardite), cristobalite (opal), quartz, in a subordinate amount – by feldspar and mica.

In the sample materials, ore minerals are represented by magnetite, less often by hematite and goethite; sulfide minerals have symbolic values they are presented by pyrite and marcasite. 0,007-0,04 mm size barrel-copper is rarely met.

Table 1 – The interplanar spacings and the phase identification of oxidized nickel-cobalt ores

2 Theta	DA	Intensity Count	Intensity, %	Phase
6,403	13,79193	98,2	100,0	Smectite
8,764	10,08180	46,9	47,8	Hydromica
12,115	7,29930	39,5	40,3	
12,421	7,12036	24,5	25,0	Chlorite
19,651	4,51392	65,9	67,1	Smectite
20,846	4,25784	68,5	69,8	Quartz
21,606	4,10969	80,4	81,9	Cristobalite
22,225	3,99658	57,5	58,6	
24,378	3,64828	41,2	42,0	Serpentine / n
26,615	3,34654	76,8	78,2	Quartz
27,925	3,19248	43,4	44,2	Pyrite middlings
28,449	3,13481	34,0	34,7	Sphalerite
30,206	2,95633	32,9	33,6	Magnetite
33,155	2,69985	31,5	32,1	Pyrite, sphalerite
34,779	2,57740	40,8	41,6	Smectite
35.657	2.51595	79.5	81.0	Magnetite
42,012	214889	25,8	26,2	Lizardite
57,192	1,60939	26,6	27,1	Magnetite
58,025	1,58825	19,9	20,2	
61,288	1,51128	30,5	31,1	Smectite
62,974	1,47481	25,3	25,8	Magnetite

Smectite and minerals of the serpentine group are in close intergrowth, forming natural inseparable mixtures with pseudomorphs of clay minerals on serpentine and small dense impregnations of magnetite. Cristobalite and quartz present in the form of individual crystals.

Magnetite and hematite present in 0.3-1.0 mm sized grains; hematite circumferentially replaces magnetite grains, forming structures of margin flanges. Larger extractions of iron oxide apparently formed through natural oxidation of iron sulfides (pyrite, marcasite).

To complete the characteristics of the material composition and the identification of mineral impurities spectral semi-quantitative analysis of the sample on a diffraction spectrograph DFS – 425 was made, its results are given in Table 2.

Table 2 – Spectral analysis of the oxidized nickel-cobalt ore

Elements	Concentration, %	Elements	Concentration, %	Elements	Concentration, %
Cu	0,001	Cr	≥1,0	Fe	–
Pb	0,003	Ni	>1,0	Au	–
As	<0,005	Co	0,05	Mg	>1,0
V	0,003	Mo	0,0003	Si	>>1,0
Sb	<0,005	Zn	0,05	Ca	–
Mn	0,2	Ag	0,0005	Ge	–
Ti	0,002	Sn	0,001	Bi	–

The results of chemical analysis of the oxidized nickel-cobalt ore are given in Table 3.

Table 3 – Composition of oxidized nickel-cobalt ore sample

Title	Concentration, %								nnn, %
	Ni	Co	Cu	Fe_2O_3	SiO_2	Al_2O_3	CaO	MgO	
Ni-Co ore	1,07	0,066	0,24	18,81	47,96	3,22	0,98	1,9	12,97

Analyzing the data, we can draw a definite conclusion that the nickel presented in the sample is connected with the rock-forming clay minerals, in particular, with the smectite (nontronite) and mixed formations. Definite nickel containing minerals through mineralogical or X-ray diffraction methods of analysis have not been identified.

Influence of preparation conditions of the raw ore on hydrometallurgical extraction of target metals – nickel, cobalt and iron. For performing the experiments, the initial sample of the oxidized nickel-cobalt ore was shattered with the help of swing-hammer mill to minus 3 mm class. After averaging by means of "cone-ring" and quartering, we received four equivalent parts, three of which were grinded to minus 1 mm, minus 0.63 mm and minus 0.074 mm grain-size class. The chemical composition of samples with varying degrees of grinding remained unchanged. Screen assay was carried out for each grain-size class; the results are given in Table 4.

Table 4 – Results of screen assay for different grain-size class

Grain-size class							
3,0 mm		1,0 mm		0,63 mm		0,074 mm	
Fraction, mm	%	Fraction, mm	%	Fraction, mm	%	Fraction, mm	%
-3,0+1,0	32,5	+1	1,01	+0,63	0,41	+0,074	27,6
-1,0+0,63	13,69	-1+0,63	23,84	-0,63 +0,074	70,73	-0,074	72,4
-0,63 +0,074	35,54	-0,63 +0,074	52,73	-0,074	28,86		
-0,074	18,27	-0,074	22,42				
	100		100		100		100

Determination of the influence on the size of the ore material on metal extraction was performed by a single direct leaching of oxidized nickel-cobalt containing ore crushed to a corresponding class in a closed reactor with heating cycle and stirring device. A reactor with a volume of 0.5 dm^3 was poured with required volume of solvent (HCl) and heated to a predetermined temperature. The crushed ore was added to the heated acid. Ore loading was carried out with constant stirring. After loading process, the stirring was stopped, then we accurately fixed the amount of produced fine pulp. Then, we again started mixing for the required time. During leaching we constantly monitored the amount of fine pulp in the reactor.

Conditions in all the experiments were constant: t = 65 ° C, duration – 2 h. We used hydrochloric acid with a concentration of 250 g / dm3 as the solvent. Rotation of the stirrer was 220 rev/min. The process temperature is recorded by alcohol thermometer with an accuracy of ± 1,0 ° C.

At the end of the leaching process, the fine pulp was filtered on a nutsch filter without prethickening. The filtration of fine pulp was carried out on funnel with diameter of 13 cm through the filter of "red tape" in a vacuum of 0.9 atm.

We washed the solid phase (leaching cakes) on the filter off the filtrate residue, dried in drying box at a temperature of 105 °C to constant weight and analyzed for controlled components: Ni, Co, Fe. On the basis of difference between the concentration in the initial ore and leaching cakes, we calculated extraction rate of controlled components from the ore into liquid phase. Degree of extraction of controlled components by ore grain size is given in Table 5 and in Figures 3-5.

Table 5 – Characteristics of the leaching process of ore raw materials, depending on the size of the ore

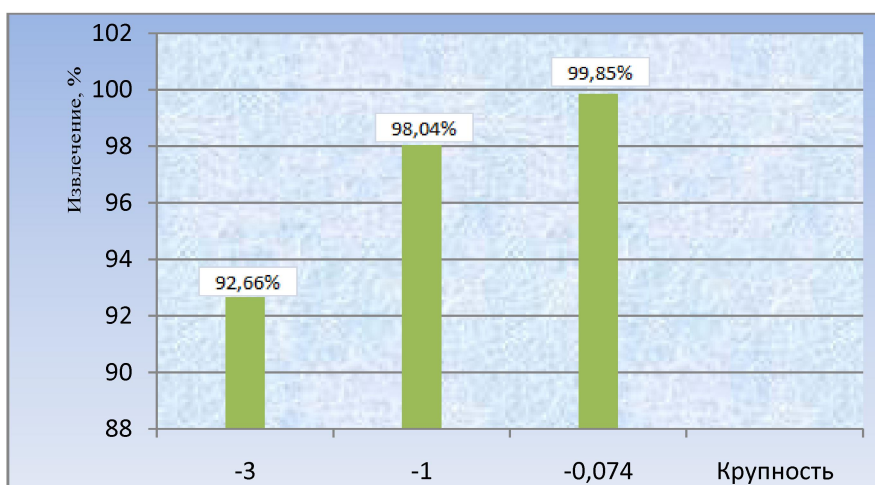| Grain-size class, mm	Loss in weight, %	Concentration in cakes, %			Extraction into liquid phase, %		
		Ni	Co	Fe	Ni	Co	Fe
-3	40,9	0,133	0,010	9,20	92,66	90,91	58,66
-1	41,7	0,036	0,004	8,28	98,04	96,37	63,29
-0,63	41,9	0,028	0,003	7,59	98,48	97,28	66,47
-0,074	44,8	0,003	0,002	0,14	99,85	98,34	99,42

Figure 3 – Nickel extraction into liquid phase depending on the size of the ore
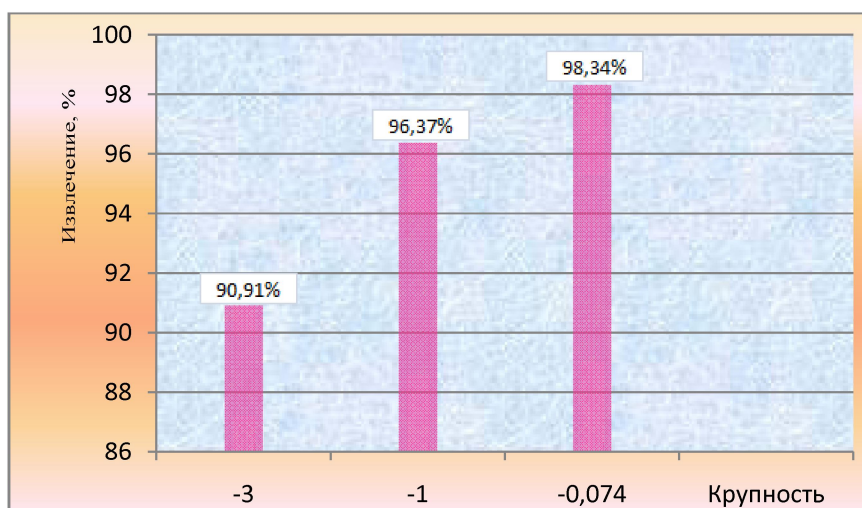

Figure 4 – Cobalt extraction into liquid phase depending on the size of the ore

Leaching solutions and washings were also analyzed for controlled components.

Composition of leaching solutions depending on the size of the ore is given in Table 6. Composition of the washings depending on the size of the ore is given in Table 7.
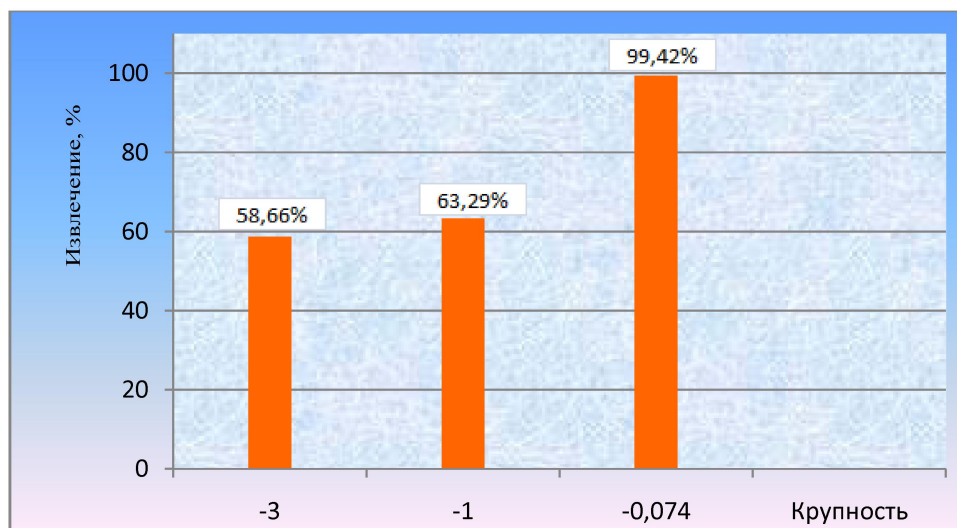
Figure 5 – Iron extraction into liquid phase depending on the size of the ore

Table 6 – Composition of leaching solutions with distribution of controlled components depending on the size of the ore

Temperature, °C	$V_{ф\text{-}та,}$ dm³	Concentration, g/dm³			Distribution, %		
		Ni	Co	Fe	Ni	Co	Fe
-3	0,302	3,131	0,1900	24,414	88,55	86,90	56,07
-1	0,301	3,314	0,2003	26,313	93,24	91,37	60,23
-0,63	0,300	3,329	0,2023	27,628	93,34	91,97	63,03
-0,074	0,288	3,359	0,2042	41,167	90,42	89,09	90,16

Table 7 – Composition of washings with distribution of controlled components depending on the size of the ore

Temperature, °C	V dm³	Concentration, g/dm³			Distribution, %		
		Ni	Co	Fe	Ni	Co	Fe
-3	0,137	0,320	0,0190	2,486	4,11	3,94	2,59
-1	0,150	0,341	0,0220	2,683	4,79	5,0	3,06
-0,63	0,194	0,283	0,0176	2,332	5,13	5,31	3,44
-0,074	0,297	0,339	0,0205	4,100	9,42	9,25	9,26

The best result was obtained by grinding the ore to 72% minus class 0.074 mm. The degree of extraction into solution comprised Ni – 99,85%, Co – 98,34%, Fe – 99,42%.

By increasing the size of the initial material from 0.074 mm minus class to 3.0 mm minus class, degree of extraction into solution of controlled components is reduced. It depends on the degree of grinding of ore extraction in an aqueous solution of iron.

Conclusion.

– Results of the experiments showed that the degree of extraction of Ni, Co, Fe during leaching of nickel-cobalt ore depends on the grain-size class of initial material;

– On the basis of the research we found that in the leaching process it is preferable to crush nickel and cobalt ore to 0.074 mm minus class at least 72% for effective transition to the solution of nickel, cobalt, iron;

– Experimentally found that the degree of crushed ore to 0.074 mm minus class enables to transfer controlled components in the solution with maximum efficiency – Ni – 99,85%, Co – 98,34%, Fe – 99,42%.

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ТҰЗҚЫШҚЫЛДЫ ШАЙМАЛАУ КЕЗІНДЕ НИКЕЛЬҚҰРАМДЫ КЕНДІ ШИКІЗАТТЫ ҰНТАҚТАУ ДӘРЕЖЕСІНІҢ МЕТАЛДАРДЫ БӨЛІП АЛУҒА ӘСЕРІ

Аннотация. Мақалада физика-химиялық талдаудың қазіргі уақыттағы әдістерін қолданумен кенді шикізаттың сипаттамасы, никельді шаймалау процесіне шикізатты алдын ала дайындаудың технологиялық ерекшеліктері және берілген шикізатты тұзқышқылында шаймалау үшін кенді ұнтақтаудың оңтайлы дәрежесін таңдау жағдайлары ұсынылған.

Түйін сөздер: тотыққан никельқұрамды кен, кенді дайындау, ірілік класы, шаймалау, еріткіш.

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ВЛИЯНИЕ СТЕПЕНИ ИЗМЕЛЬЧЕНИЯ НИКЕЛЬСОДЕРЖАЩЕГО РУДНОГО СЫРЬЯ НА ИЗВЛЕЧЕНИЕ МЕТАЛЛОВ ПРИ СОЛЯНОКИСЛОМ ВЫЩЕЛАЧИВАНИИ

Аннотация. В статье представлены характеристика рудного сырья с применением современных методов физико-химического анализа, технологические особенности предварительной подготовки сырья к процессу выщелачивания никеля и выбор оптимальной степени измельчения руды для солянокислого выщелачивания указанного сырья.

Ключевые слова: окисленная никельсодержащая руда, рудоподготовка, класс крупности, выщелачивание, растворитель.