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Х А Б А Р Л А Р Ы

ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ
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КАЗАХСТАН
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NAS RK is pleased to announce that News of NAS RK. Series of geology and technical sciences scientific journal has been accepted for indexing in the Emerging Sources Citation Index, a new edition of Web of Science. Content in this index is under consideration by Clarivate Analytics to be accepted in the Science Citation Index Expanded, the Social Sciences Citation Index, and the Arts & Humanities Citation Index. The quality and depth of content Web of Science offers to researchers, authors, publishers, and institutions sets it apart from other research databases. The inclusion of News of NAS RK. Series of geology and technical sciences in the Emerging Sources Citation Index demonstrates our dedication to providing the most relevant and influential content of geology and engineering sciences to our community.

Қазақстан Республикасы Ұлттық ғылым академиясы «ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы» ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Web of Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді геология және техникалық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия геологии и технических наук» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК. Серия геологии и технических наук в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по геологии и техническим наукам для нашего сообщества.

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**DEVELOPMENT OF AN INTEGRATED TECHNOLOGY
INTENDED TO PROCESS PYRITE SLAG USING
CHEMICAL PRE-ACTIVATION**

Abstract. The existing methods intended to process pyrite slag require improvement. The novelty of the technology used in this work is the preliminary chemical pyrite slag activation in a sodium bicarbonate solution. It was found that changes of phase composition occur as a result of pyrite slag activation: disappeared phases of trisodium phosphate of zinc oxide hydrate and dolomite; the amount of sodium thiophosphate; the amount of albite and of natrozharosite phase decreased; the phase of magnesium, calcium silicate and sodium thiophosphate appeared. After activation, the content of the iron-containing phases also increases. Changes of phase composition of pyrite slag are connected with interaction reactions of sodium hydrocarbonate with trisodium phosphate zinc oxide hydrate, natrozharosite and with dolomite with formation of sodium thiophosphate, calcium silicate and magnesium aluminosilicate. At leaching of pyrite cinders after preliminary activation in 15% H_2SO_4 solution extraction in solution, wt. % was obtained: CuO 76.8; ZnO 75.9 and Fe_2O_3 26.0. Without chemical activation the degree of extraction of non-ferrous metals in sulphuric acid solution is lower by 15 – 20%. As a result of the stage neutralization of the leaching solution of pyrite slag at pH 9.7 the neutralization precipitate - concentrate of non-ferrous metals, wt. % was obtained: CuO 6.4; ZnO 12.55. Concentrate yield was 1.5% of the initial mass of pyrite slag. Smelting of leaching cakes of pyrite slag produced crude iron with chemical composition, wt %: 91.13 Fe; 3.99 Si; i/r P; 2.57 C; 0.81 Mn; 0.025 S.

Key words: pyrite slag, chemical activation, phase composition, non-ferrous metals, leaching, concentrate, blooming cast iron.

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АЛДЫН АЛА ХИМИЯЛЫҚ БЕЛСЕНДІРУ МЕН ПИРИТТІ КҮЙІКТЕРДІ ӨНДЕУДІҢ КЕШЕНДІ ТЕХНОЛОГИЯСЫН ӘЗІРЛЕУ

Аннотация. Пиритті күйіктерді өндеу қолданыстағы әдістерді жетілдіруді қажет етеді. Жұмыста пайдаланылған технологияның жаңалығы – натрий гидрокарбонатының ерітіндісіндегі пиритті күйіктерге алдынала химиялық белсендіруді жүргізу болып табылады. Белсендірудің нәтижесінде фазалық құрамда өзгерістер болатыны анықталды: үшнатрийлі фосфаты мырыш гидрат оксиді мен доломит фазалары жоғалды; натрий тиофосфаты фазасының саны артты; альбит және натрожарозит фазасының саны азайды; магний алюмосиликатының, кальций силикаты және натрий тиофосфат фазалары пайда болды. Пиритті күйіктердің фазалық құрамының өзгеруі натрий гидрокарбонатының үшнатрийлі фосфаты мырыш оксиді гидрат және, натрожарозит натрий тиофосфатын, кальций силикаты мен магний алюмосиликатының пайда болатын доломитпен өзара әрекеттесу реакцияларының жүруімен байланысты. Тиімді жағдайда 15% H_2SO_4 ерітіндісінде пирит күйіктерін шаймалау кезінде ерітіндіге өтуі алынды, салм. %: CuO 76.8; ZnO 75.9 және Fe_2O_3 26.0. Химиялық белсендірудің күкірт қышқылы ерітіндіге түсті металдарды алу дәрежесі 15-20 % төмен. Пирит күйіктерін шаймалау ерітіндісін стадиялық бейтараптандыру нәтижесінде рН 9,7 кезінде түсті металдар концентраты алынды, салм. %: CuO 6,4; ZnO 12,55. Концентрат шығымы пирит күйіктерінің бастапқы салмағының 1,5%-ын құрады. Пирит күйіктерді сілтісіздендіру кенін балқыту кезінде химиялық құрамы бар шойын түріндегі шойын алынды, салм. %: 91,13 Fe; 3.99 Si; н/о P; 2,57 C; 0,81 Mn; 0,025 S.

Түйін сөздер: пиритті күйіктер, химиялық белсендіру, фазалық құрам, түсті металдар, шаймалау, концентрат, шойын.

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РАЗРАБОТКА КОМПЛЕКСНОЙ ТЕХНОЛОГИИ ПЕРЕРАБОТКИ ПИРИТНЫХ ОГАРКОВ С ПРЕДВАРИТЕЛЬНОЙ ХИМИЧЕСКОЙ АКТИВАЦИЕЙ

Аннотация. Существующие способы переработки пиритных огарков требуют совершенствования. Новизной технологии, использованной в работе, является проведение предварительной химической активации пиритных огарков в растворе гидрокарбоната натрия. Установлено, что в результате активации происходят изменения фазового состава: исчезли фазы тринатрий фосфата цинка оксида гидрата и доломита; увеличилось количество фазы тиофосфата натрия; уменьшилось количество фазы альбита и натроजारозита; появилась фаза алюмосиликата магния, силиката кальция и тиофосфата натрия. Изменения фазового состава пиритных огарков связаны с протеканием реакций взаимодействия гидрокарбоната натрия с тринатрий фосфат цинк оксид гидратом, натроजारозитом и с доломитом с образованием тиофосфата натрия, силиката кальция и алюмосиликата магния. При выщелачивании пиритных огарков в 15% растворе H_2SO_4 получено извлечение в раствор, мас. %: CuO 76,8; ZnO 75,9 и Fe_2O_3 26,0. Без химической активации степень извлечения цветных металлов в сернокислый раствор ниже на 15 – 20%. В результате стадийной нейтрализации раствора выщелачивания пиритных огарков при pH 9,7 получен - концентрат цветных металлов, мас. %: CuO 6,4; ZnO 12,55. Выход концентрата составил 1,5% от исходной массы пиритных огарков. При плавке кека выщелачивания пиритных огарков получен кричный чугуи с химическим составом, масс %: 91,13 Fe; 3,99 Si; н/о P; 2,57 C; 0,81 Mn; 0,025 S.

Ключевые слова: пиритные огарки, химическая активация, фазовый состав, цветные металлы, выщелачивание, концентрат, кричный чугуи.

Introduction. Since the last century the main method of sulphuric acid production has been roasting of pyrite concentrate (Chernyshev A.K., et all 2014). Pyrite is an iron mineral of sulphide class that often contains admixtures of gold, cobalt, copper and other non-ferrous metals. The pyrite slag, formed after roasting, is deposited that creates a real hazard of water and air pollution; at the

same time, they are a valuable source of ferrous, non-ferrous and noble metals that is not used until now due to the lack of economically viable technologies. In this regard, the development of an efficient, integrated technology for the processing of pyrite slag **is relevant**.

Pyritic slag is equivalent to good iron ores in terms of iron content (50-60%), but its use for smelting cast iron is hindered by the presence of non-ferrous metals and sulphur in it. Therefore, the processing flowchart should ensure a sufficiently complete extraction of non-ferrous metals and obtain a product suitable for blast furnace smelting.

The main method intended to process pyrite slag, introduced in a number of countries, is chlorinating or sulphatizing roasting followed by leaching of non-ferrous metal compounds (Beregovsky V.I., et al 1973; Güntner, J., Hammerschmidt, J., 2012; Zabereshs I.I., 1967)

The method (Beregovsky V.I., et al, 1973) provides low-temperature (550-600 °C) chlorinating roasting of slag with table salt (or calcium chloride) introduced at Duisburg Plant in Germany. But the necessity of leaching of excessively large volumes of chlorinated slag for the extraction of non-ferrous metals is one of the drawbacks reducing its practical importance.

In Finland and Zambia, the initial concentrate is subjected to sulphate or dissolution roasting followed by hydrometallurgical processing of the slag (Güntner, J., Hammerschmidt, J., 2012). The process developed by the “Outokumpu” company includes melting of pyrite concentrates in a neutral atmosphere with sublimation of sulphur to produce a troilite matte, removal of slag, granulation of the matte in water and its oxidation roasting. The method allows to obtain a product containing up to 67% of iron, but does not provide for the extraction of non-ferrous and noble metals (Zabereshs I.I., 1967).

In more recent developments, the method involving heating of the cake and its smelting in the presence of reducing agent and flux mixtures, consisting of CaO and Al₂O₃ - containing materials, and the processing of resulting iron-based alloy with solid oxidizing agents, containing calcium sulfate (Patent 2172788 RU, 2001), in some cases SiO₂ are used as a flux (Patent 2394924 RU). The disadvantage of the methods is the low extraction of noble metals.

All the described methods are pyrometallurgical and are energy intensive. Recently great attention is given to hydrometallurgical methods intended to process of pyrite slag that require improvement for complete utilization with extraction of useful components - nonferrous, noble metals and iron.

The method (Patent 2623948 RU, 2017) includes preliminary four-stage leaching of nonferrous metals with water and sulphuric acid solution followed by leaching of noble metals with hydrochloric acid thiocarbamide solution. The method allows separation of blister copper, iron oxide pigment, zinc oxide

into separate products and extraction of noble metals from thiocarbamide hydrochloride solution.

The disadvantages of the method are the low extraction of iron and noble metals from the slag, as well as the contamination of obtained iron oxide pigments with impurities of non-ferrous metals.

The method of deep processing of pyrite slag including leaching of non-ferrous metals by bacterial complex is known (Patent 2397260 RU, 2013). Disadvantages of this method are long duration of bacterial leaching and complexity of cultivation process of acidophilic thionic bacteria.

In the conducted studies for the complex processing of pyrite slag the method of preliminary chemical activation of mineral raw materials was used that promotes the disintegration and phase changes (Patent 32333 KZ, 2017; Gladyshev S.V., Kenzhaliev B.K., et al 2018; Abdulvaliev R.A., et al 2021; Dyusenova S.B., et al 2019). The method consists in thermal processing with a solution of sodium hydrogen carbonate. Due to conditions of chemical activation modification of hard-to-recover phases and disclosure of mineral structure due to separation of non-metallic materials takes place.

In researches optimum conditions of preliminary chemical activation corresponding to features of mineral structure of pyrite slag have been determined. Positive effect of activation is confirmed by results of non-ferrous metals leaching from pyrite slag in sulphuric acid solution. The optimum leaching regime was established.

Research materials and methods. The source material for the study was a significant sample of pyrite slag from the sulphuric acid production of the Tselinny Mining and Chemical Plant in the Republic of Kazakhstan.

X-ray fluorescence, chemical and X-ray phase analyses were used in the work.

X-ray fluorescence analysis was performed on Venus 200 spectrometer with wave dispersion (PA Nalyical B.V., Holland).

Chemical analysis of samples was performed on an optical emission spectrometer with inductively coupled plasma (Optima 8300 DV, Perkinelmer, Waltham, MA, USA). The random error component is 2.0%.

X-ray phase analysis was performed using a D8 Advance (Bruker, Billerica, Massachusetts, USA) with Cu KA radiation obtained at 40 kV and 40 mA. Processing of diffraction patterns and calculation of interplanar distances were performed using EVA software, while phase identification was performed using the search/comparison program and the PDF-2 powder diffraction database.

Thermal analysis was performed using a Jupiter STA 449 F3 simultaneous thermal analysis device. Before heating, the furnace space was pumped out (achievable vacuum level ~ 92%) and then purged with inert gas for 5 minutes. The heating was performed at a rate of 100 C/min in an atmosphere of highly

purified argon. The total volume of incoming gas was maintained between 100 and 110 ml/min. Results obtained with the STA 449 F3 Jupiter were analyzed using the NETZSCHProteus software.

Microphotographs were taken using a JEOL low vacuum scanning electron microscope with thermal emission cathode (LaB6) JSM-6610LV equipped with a system of energy dispersion (ED) microanalysis, a system of wave dispersion microanalysis, a system of backscattered electron diffraction analysis, a reflected electron detector, an Everhart-Thornley secondary electron detector and a secondary electron detector for low vacuum mode.

Mössbauer spectroscopy was performed on a Mössbauer spectrometer - SM 2201. The source was cobalt 57 in a rhodium matrix, activity 100 mCi. Spectra were analyzed on PC by the method of "least squares". Values of isomeric shifts (Is) are given relative to -Fe. Temperature of spectra taking - 293 K. Imaging mode "at light".

Analysis of ICS was obtained on FT-IR spectrometer "Avatar 370CsI" in the spectral range 4000-300 cm⁻¹ from the tablets prepared by pressing 2 mg of sample and 200 mg of KBr. The spectrum of KBr was taken as a comparison spectrum. Experiment set-up: Transmission E.S.P.

Chemical pyrite slag activation was performed in a solution containing 40 to 120 g/dm³ NaHCO₃ at L:S=2-10.0 and a temperature of 90-2300, using a thermostatically controlled unit with 4 autoclaves rotating through the head and a working volume of 250 cm³.

The activation time ranged from 30 to 300 minutes. The maximum sodium hydrogen carbonate content of 120 g/dm³ in the solution was chosen taking into account its solubility limit.

Results and discussion. As a result of sieve analysis of samples of pyrite slag it was found that in the class +2.5 mm the content of useful components - noble metals, non-ferrous metals and iron is much lower (table 1).

Table 1 - Chemical composition of classes of pyrite slag

Composition, %	Grain size class, mm					
	+2.5	-2.5+1.0	-1.0+0.25	-0.25+0.1	-0.1+0.056	-0.056
Na ₂ O	2.44	1.43	1.19	0.959	0.75	0.71
MgO	3.29	0.65	0.67	0.483	0.41	0.39
Al ₂ O ₃	10.96	6.92	6.37	4.163	3.11	3.03
SiO ₂	39.73	27.09	25.18	16.13	11.89	11.44
P ₂ O ₅	0.26	1.26	1.1	0.956	0.84	0.82
SO ₃	0.51	7.33	8.1	7.725	6.35	6.1
CaO	9.72	2.84	2.62	1.949	1.41	1.2

TiO ₂	1.26	0.42	0.37	0.25	0.178	0.16
Fe ₂ O ₃	7.63	41.63	45.63	60.94	70.187	71.16
CuO	0.02	0.2	0.22	0.26	0.261	0.28
ZnO	0.04	0.4	0.4	0.61	0.604	0.66
As ₂ O ₃	0.06	0.19	0.25	0.26	0.258	0.26
SeO ₂	0.003	0.51	1.01	0.57	0.23	0.19
BaO	0.186	2.07	2.33	2.81	2.866	3.01
HgO	-	0.09	0.19	0.14	0.055	0.04
PbO	0.005	0.15	0.17	0.2	0.185	0.21
п.п	22.266	6.29	3.61	1.237	0.146	0.08
Au, g/t	0.021	1.58	2.68	2.69	2.24	2.88
Ag, g/t	0.1	11.2	16.2	19.3	21.4	22.3
Yield, %	31.0	6.2	5.5	20.8	34.3	2.2

Magnetic separation determined that the class + 2.5 mm is not a magnetic fraction, and the class - 2.5 mm + 0 is a highly magnetic fraction, it was separated at the magnetic field strength of 200 - 400 oersted.

Chemical composition of the magnetic fraction of pyrite slag of size class - 2.5 mm + 0 wt. %: Na₂O 1.4; MgO 0.74; Al₂O₃ 5.69; SiO₂ 23.22; P₂O₅ 1.1; SO₃ 6.24; ClO.01; K₂O 0.44; CaO 2.52; TiO₂ 0.32; Fe₂O₃ 52.84; CuO 0.25; ZnO 0.53; As₂O₃ 0.24; SeO₂ 0.3; BaO 2.4; HgO 0.08; PbO 0.16; p.p. 1.82; noble metal content, g/t: Au 2.69; Ag 19.3.

The phase composition of the magnetic fraction of pyrite slag is presented, wt. %: maghemite 24.1, hematite 18.1, quartz 17.2, albite 10.2, trisodium phosphate zinc oxide hydrate 9.5, sodium aluminosilicate 6.7, barium ferrite 4.7, natrozharosite 4.2, sodium thiorphosphate 2.8 and dolomite 2.5 (Figure 1).

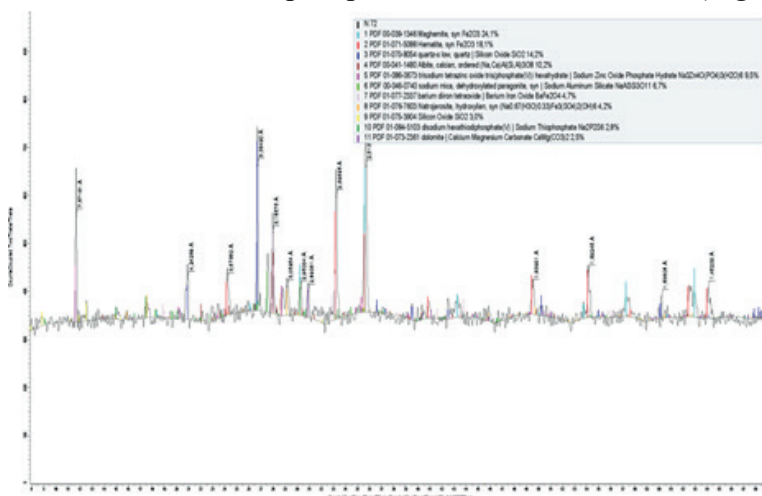


Figure 1. X-ray phase analysis of the magnetic fraction of pyrite slag of grain size class - 2.5 mm + 0

Chemical composition of the nonmagnetic fraction of pyrite slag of grain size class - - + 2.5 mm wt. %: Na₂O 2.44; MgO 0.3.29; Al₂O₃ 10.96; SiO₂ 39.73; P₂O₅ 0.26; SO₃ 0.51; Cl 0.01; K₂O 0.8; CaO 2.72; TiO₂ 1.26; Fe₂O₃ 7.63; CuO 0.02; ZnO 0.04; As₂O₃ 0.06; SeO₂ 0.003; BaO 0.186; PbO 0.005; other prod. 23.076; noble metal content, g/t: Au 0.021; Ag 0.1.

The phase composition of the non-magnetic fraction of pyrite slag is presented, wt. %: magnemite 2.1; hematite 1.4; quartz 29.9; albite 18.2; dolomite 18.2; calcite 17.3; clinoclhorite 7.7; muscovite 3.5; and gibbsite 1.4.

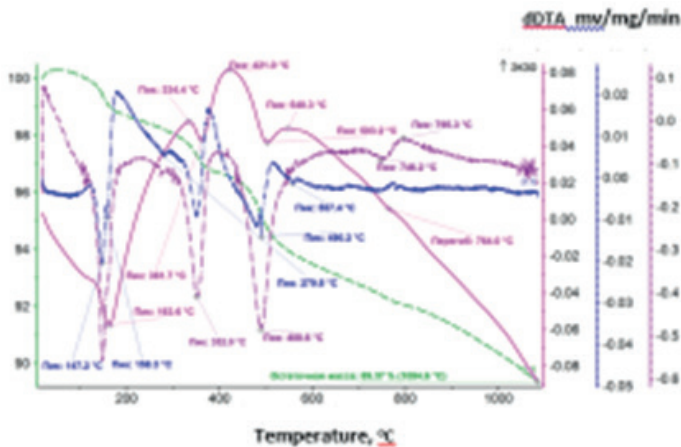


Figure 2. Thermogram of pyrite slag before activation

According to thermal analysis a peak at 334.4°C (Figure 2) reflects the oxidation of divalent iron in fine-dispersed magnetite.

Exothermic peaks (334.4°C, 421°C, 548.3°C) are associated with oxidation and decomposition of residual sulfides. Among them, arsenopyrite, chalcopyrite, realgar (AsS) and elemental sulfur.

Combination of endothermic effects with extremums at 361,7°C, 503,2°C, inflection at 7640°C and exothermic effect with the peak at 421°C on the DTA curve reflects the presence of hydrolomite (50% hydromagnesite + 50% calcite). The decomposition of calcite in this sample is in the region of minimum development at 746.2°C on the DTA curve. Combination of endothermic effects with extremums at 147.2°C, 158.3°C and 279.8°C on the DTA curve and inflection at 7640°C on the DTA curve is connected to display of metasideronatriite Na₂Fe³⁺[SO₄]₂(OH)1,5H₂O decomposition. Weak endothermic effect with extremum at 557.7°C on the DTA curve is a reflection of quartz inversion. The combination of endothermic effect with extremum at 503.2°C, kink at 764°C and exothermic effect with the peak at 548.3°C on the DTA curve presumably may be connected with occurrence of natrozharosite: NaFe₃⁺³ [SO₄]₂(OH)₆.

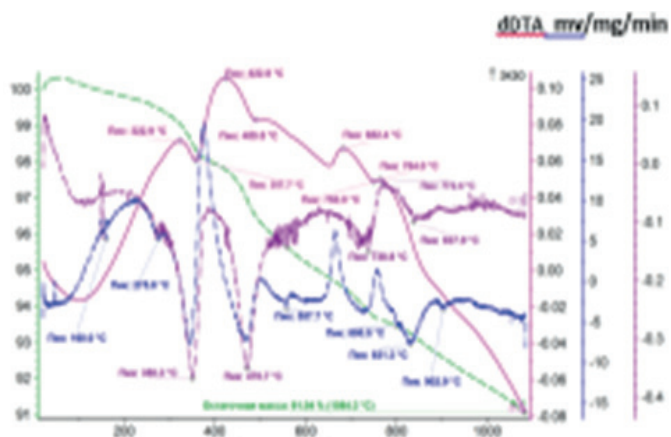


Figure 3. Thermogram of pyrite slag after activation

An increase in the number of effects as well as a shift in the temperature of individual peaks was observed at the dDTA after chemical activation (Figure 3) compared with the dDTA of the original sample. Thus, the oxidation peak of divalent iron in magnetite has shifted towards lower temperatures. The endothermic effect with the extremum at 503.2°C in the DTA of the initial sample in the considered sample appeared at 489.80°C. The inflection at 764°C turned into a full endothermic effect with extremum at 750°C.

The exothermic effect with a peak at 322.90°C may reflect the first stage of oxidation of divalent iron in magnetite. The second stage of oxidation of divalent iron in magnetite was also revealed in this sample. This is an exothermic effect with a peak at 764°C. As in the original sample, exothermic effects in the temperature range of 300 - 700°C can be associated with oxidation and decomposition of residual sulfides, as well as oxidation of elemental sulfur.

The 682.4°C peak may reflect oxidation of pyrrhotite by residual oxygen. At 700 - 950°C, carbonates such as magnesite, calcite and ankerite can decompose. The weak endothermic effect with extremum at 800.5°C on the dTAm curve may be a reflection of a polymorphic transformation of barium carbonate. The presence of hydrololomite in the superposition is not excluded. Combination of endothermic effects with extremums at 489.8°C, 750°C is manifestation of jarosite decomposition, carphosiderite – $\text{Fe}_3^{3+}[\text{SO}_4]_2(\text{OH})_5\text{H}_2\text{O}$. An impurity of metasideronatriite $\text{Na}_2\text{Fe}^{3+}[\text{SO}_4]_2(\text{OH})1,5\text{H}_2\text{O}$ (160°C, 275.8°C, 750°C) may also be present in the superposition. Weak endothermic effect with extremum at 557.7°C on the dDTA curve may be a reflection of quartz inversion.

By infrared spectroscopy an increase of CaCO_3 carbonate phase (peaks 1419, 880 cm^{-1} in the original sample (Moenke H., 1962) and peaks 1794, 1431, 876, 713 cm^{-1} in the sample after activation) was determined in pyrite slag.

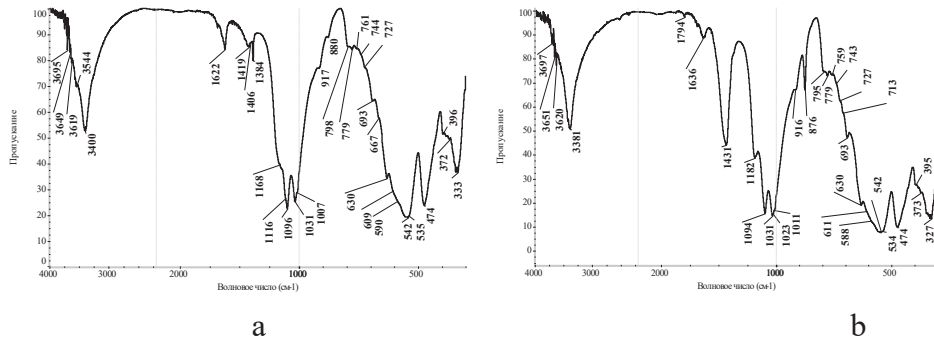


Figure 4. Infrared spectrum of pyrite slag: a - before activation; b - after activation

Mineralogical analysis of pyrite slag was performed in combination with scanning electron microscopy and X-ray microanalysis.

As a result of the analysis of magnetic fraction of initial pyrite slag was found: native iron (Figure 5), iron oxide (Figure 6), chalcopyrite (Figure 7), galena (Figure 8), quartz (Figure 9), pyrite (Figure 10), plagioclase (Figure 11), potassium barium feldspar $K(AlSi_3O_8) - Ba(Al_2Si_2O_8)$ (figure 12), barite (figure 13), forsterite (figure 14), chromospinelide (figure 15), and sphalerite ZnS (figure 16).

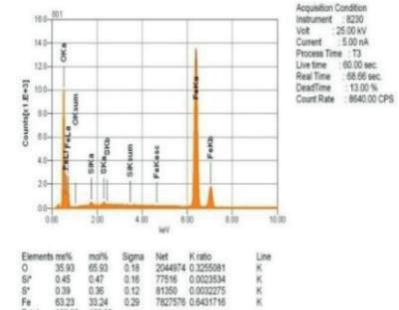
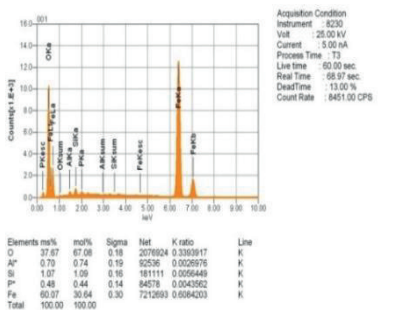
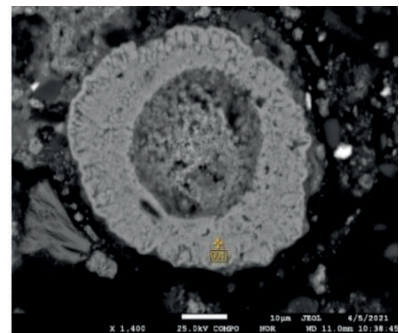
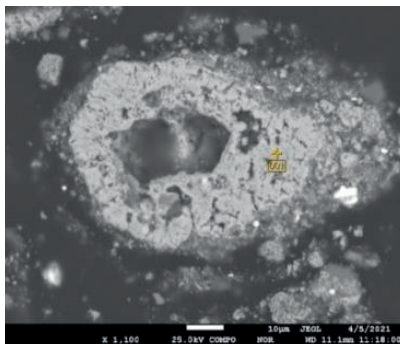


Figure 5. Native iron, x 1100

Figure 6. Iron oxide, x 1400

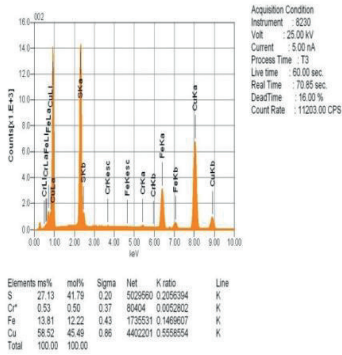
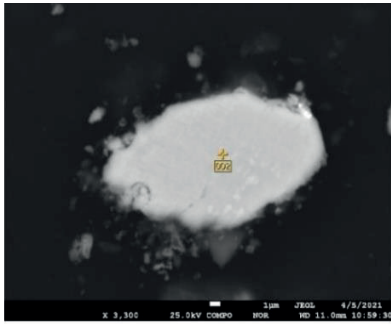


Figure 7. Chalcopyrite, x 3 300

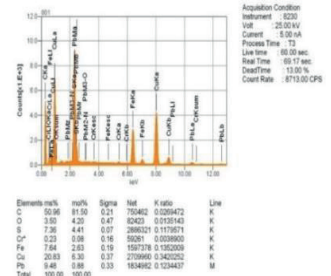
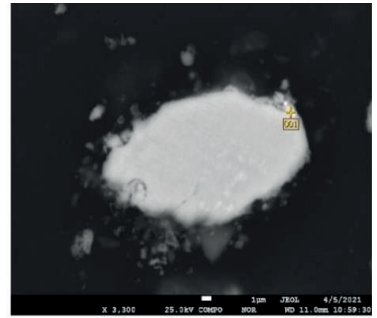


Figure 8. Galenite, x 3 300

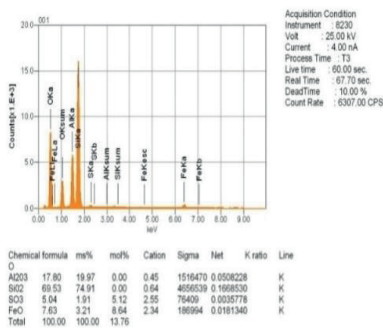
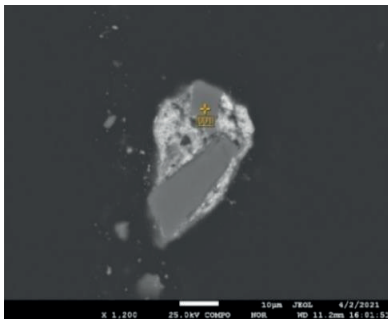


Figure 9. Quartz, x 1 200

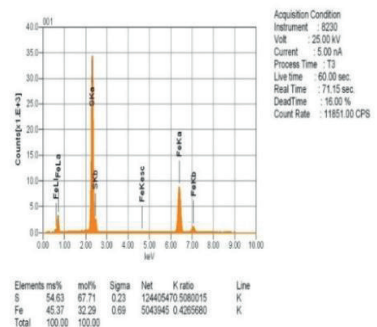
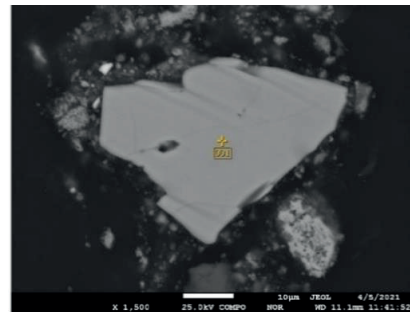
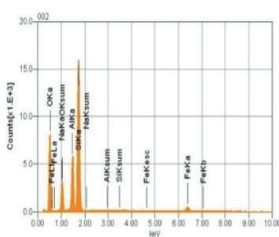
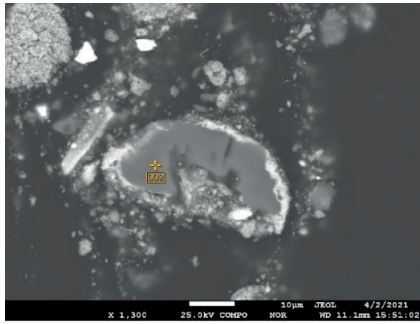
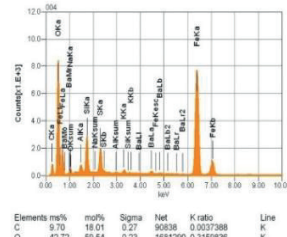
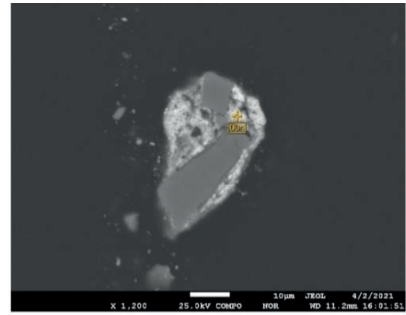


Figure 10. Pyrite, x 150



Elements	ms%	mol%	Sigma	Net	K ratio	Line
O	51.92	65.02	0.45	1831448	0.3064571	K
Na	8.66	7.55	0.32	723537	0.0481972	K
Al	9.24	6.86	0.21	1539710	0.0515963	K
Si	27.48	19.60	0.23	4666045	0.1617863	K
Fe	2.70	0.97	0.56	248135	0.0246908	K
Total	100.00	100.00				

Acquisition Condition
 Instrument : 8230
 Volt : 25.0kV
 Current : 4.00 nA
 Process Time : T3
 Live time : 60.00 sec.
 Real Time : 67.85 sec.
 DeadTime : 11.00 %
 Count Rate : 6286.00 CPS

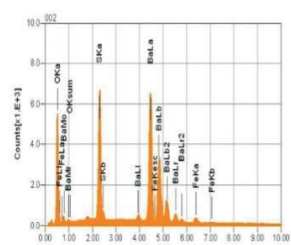
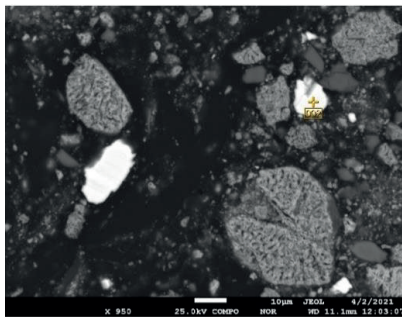


Elements	ms%	mol%	Sigma	Net	K ratio	Line
C	9.70	18.01	0.27	96838	0.0037388	K
O	42.72	59.54	0.23	1681290	0.3159836	K
Na	1.39	1.35	0.30	70263	0.0052941	K
Al	0.99	0.82	0.15	150925	0.0050002	K
Si	4.05	3.21	0.13	769677	0.0275547	K
S	2.81	1.85	0.11	597903	0.0200036	K
K	0.39	0.22	0.13	89135	0.0051612	K
Fe	36.83	14.71	0.28	4303668	0.4252903	K
Ba	1.12	0.18	0.39	137323	0.0136905	L
Total	100.00	100.00				

Acquisition Condition
 Instrument : 8230
 Volt : 25.0kV
 Current : 4.00 nA
 Process Time : T3
 Live time : 60.00 sec.
 Real Time : 67.59 sec.
 DeadTime : 11.00 %
 Count Rate : 6946.00 CPS

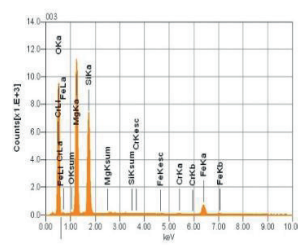
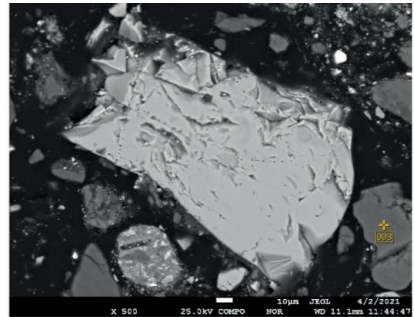
Figure 11. Plagioclase, x 1300

Figure 12. Potassium barium feldspar, x 1200



Elements	ms%	mol%	Sigma	Net	K ratio	Line
O	31.39	69.91	0.22	1040898	0.1961091	K
S	13.65	15.16	0.11	2269441	0.1055125	K
Fe	1.78	1.13	0.30	158598	0.0154246	K
Ba	53.19	13.60	0.39	5280042	0.5392572	L
Total	100.00	100.00				

Acquisition Condition
 Instrument : 8230
 Volt : 25.0kV
 Current : 4.00 nA
 Process Time : T3
 Live time : 60.00 sec.
 Real Time : 66.44 sec.
 DeadTime : 14.00 %
 Count Rate : 8416.00 CPS



Elements	ms%	mol%	Sigma	Net	K ratio	Line
O	56.34	68.76	0.37	1905982	0.3598005	K
Mg	23.31	19.72	0.21	2695074	0.0369821	K
Si	15.61	10.85	0.24	2185087	0.0786881	K
C	0.33	0.12	0.43	35882	0.0027121	K
Fe	4.41	1.54	0.53	373059	0.0363602	K
Total	100.00	100.00				

Acquisition Condition
 Instrument : 8230
 Volt : 25.0kV
 Current : 4.00 nA
 Process Time : T3
 Live time : 60.00 sec.
 Real Time : 66.56 sec.
 DeadTime : 9.00 %
 Count Rate : 5231.00 CPS

Figure 13. Barite, x 950

Figure 14. Forsterite, x 500

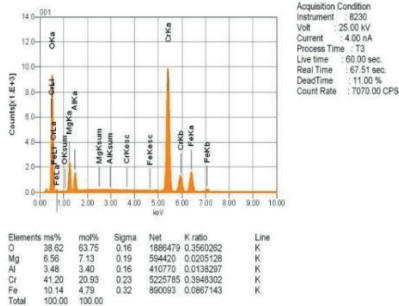
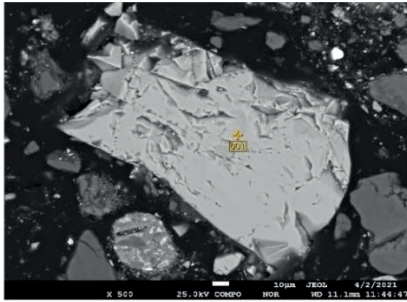


Figure 15. Chromospinelid, x 500

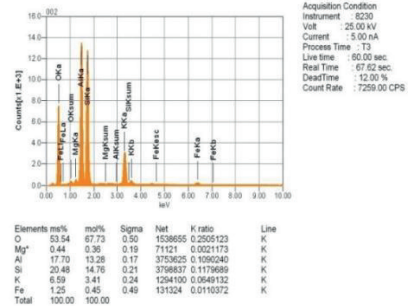
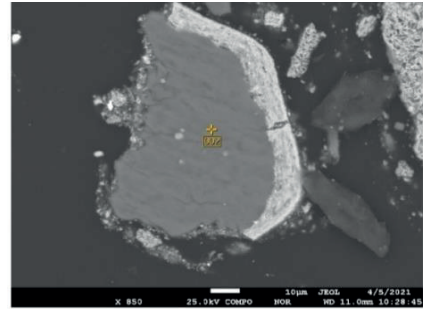


Figure 16. Sphalerite, x 1100

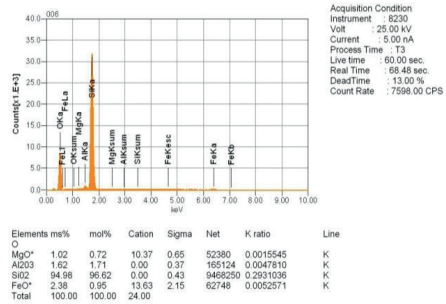
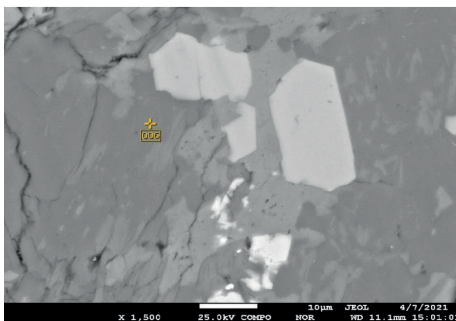


Figure 17. Apatite, x 1500

Analysis has shown that in the initial sample of pyrite slag iron minerals, pyrite, titanomagnetite and others are often connected with non-metallic material that causes difficulties in processing during extraction of nonferrous, noble metals and iron.

By results of the electron-microscopic analysis of a magnetic fraction of pyrite slag after chemical activation in optimum conditions are found: iron oxide (figure 18), sphalerite (figure 19), barite (figure 20), native iron (figure 21), tin - bismuth (figure 22), gallite - bismuth (figure 23).

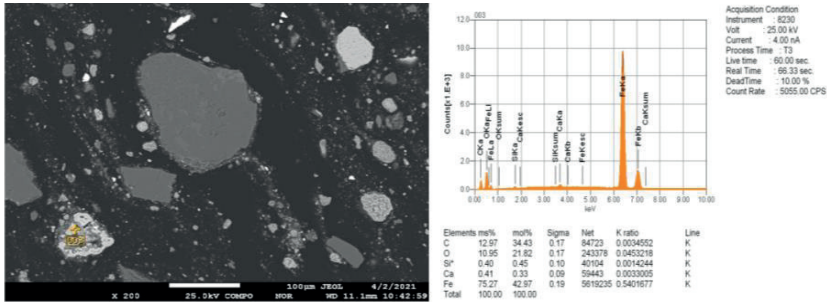


Figure 18. Iron oxide, x 200

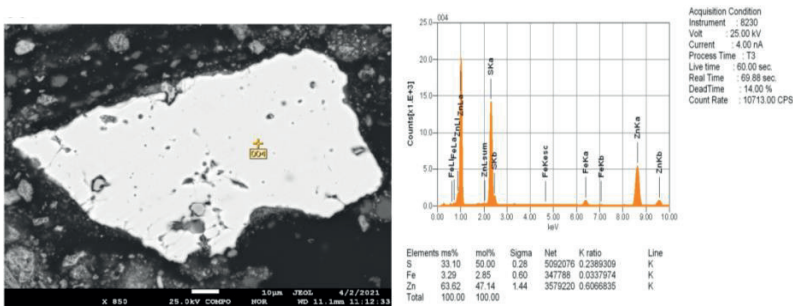


Figure 19. Sphalerite, x 850

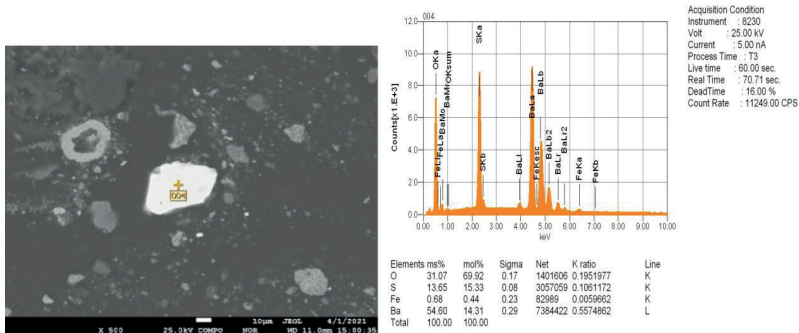


Figure 20. Barite, x 500

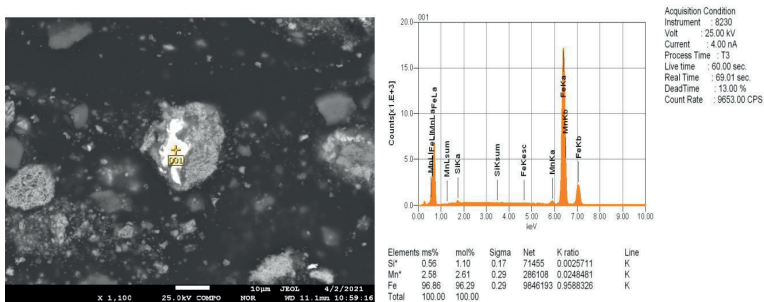


Figure 21. Native iron, x 1 100

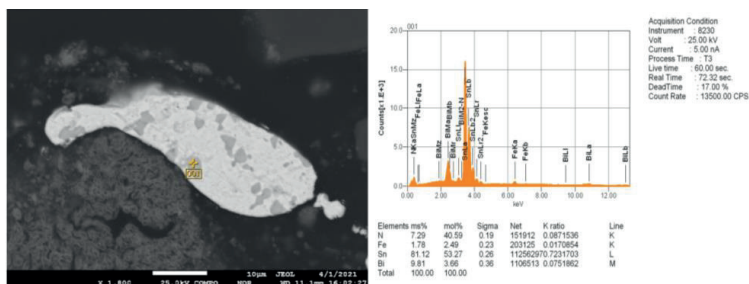


Figure 22 . Tin - bismuth, x 1,800

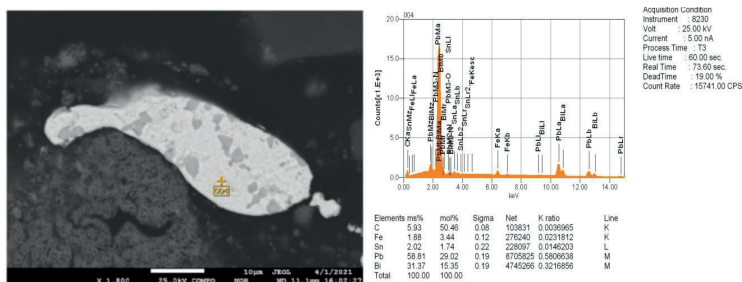


Figure 23. Gallite - bismuth, x 1,800

The microphotographs of the slag after chemical activation show that grains of ore minerals are more free from non-metallic minerals (feldspar, plagioclase, forsterite, chromitespinelide).

X-ray phase analysis of samples of pyrite slag after chemical activation in a solution of sodium hydrogen carbonate depending on temperature (90 – 230°C), duration (30 – 300 minutes), L:S ratio (2÷10: 1) and NaHCO₃ concentration (40-120 g/dm³) showed that what is in optimal conditions the maximum changes in the phase and chemical composition of cinders occur at temperature 120°C, duration 30 - 60 min, ratio L:S=4:1 and concentration of NaHCO₃ solution 60 g/dm³.

As a result of chemical activation of pyrite slag under optimal conditions, there were changes in the phase composition: the phases of zinc phosphate trisodium hydrate and dolomite disappeared; the amount of sodium thiophosphate phase increased; the amount of albite and natroharosite phases decreased; the phase of magnesium aluminosilicate, calcium silicate, carbonate and ferrite of sodium appeared.

In these conditions the chemical composition of pyrite slag after activation under optimum conditions is presented by wt. %: Na₂O 1.59; MgO 0.73; Al₂O₃ 5.63; SiO₂ 22.3; P₂O₅ 0.59; SO₃ 3.45; Cl0.01; K₂O 0.43; CaO 2.64; TiO₂ 0.31; Fe₂O₃ 51.89; CuO 0.24; ZnO 0.52; As₂O₃ 0.16; SeO₂ 0.25; BaO 2.32; HgO 0.09; PbO 0.16; other prod. 6.69.

After activation, the content of P_2O_5 , SO_3 and As_2O_3 in pyrite slag decreased by 46.36%, 44.31% and 33.3%, respectively.

The phase composition of pyrite cinder samples after chemical activation under optimal conditions is shown in Table 2 and in the Figure 24.

Table 2 - Phase composition of pyrite slag samples after chemical activation under optimum conditions

Name	Composition, %
Magemite Fe_2O_3	28.4
Hematite Fe_2O_3	21.8
Quartz SiO_2	14.7
Albite $Na(AlSi_3O_8)$	8.7
Trinatrium phosphate zinc oxide hydrate $Na_3Zn_4O(PO_4)3(H_2O)_6$	-
Sodium aluminosilicate $NaAl_3Si_3O_{11}$	6.0
Barium ferrite $BaFe_2O_4$	6.2
Natrozharosite $(Na_{0.67}(H_3O)_{0.33})Fe_3(SO_4)_2(OH)_6$	4.3
Sodium thiophosphate $Na_2P_2S_6$	5.7
Dolomite $CaMg(CO_3)_2$	-
Magnesium aluminosilicate $(MgAl_2Si_3O_{10})_{0.6}$	2.5
Calcium silicate $CaSiO_3$	1.7

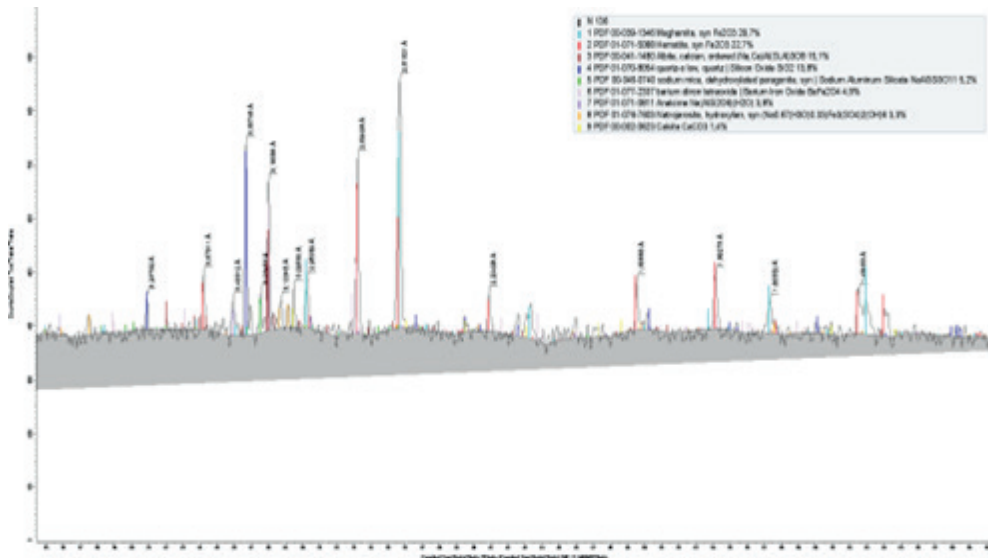
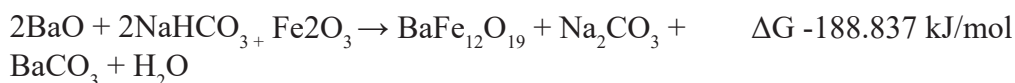
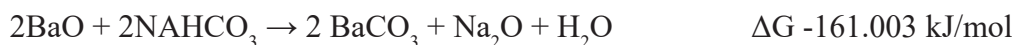
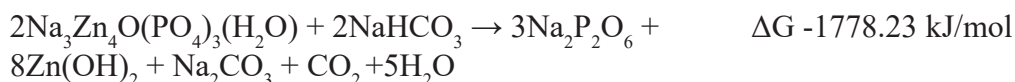
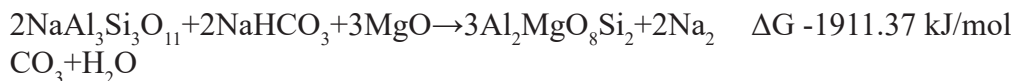


Figure 24. X-ray of pyrite slag after chemical activation under optimum conditions

Changes in the phase composition of pyrite slag during chemical activation can be attributed to the course interaction reactions with sodium bicarbonate:



The influence of preliminary chemical pyrite slag activation on extraction of non-ferrous metals and iron during leaching in sulphuric acid solutions was studied.

Leaching of pyrite slag after activation was performed in H_2SO_4 solutions containing 5-20% at temperature 60°C and duration of 30 minutes (Figure 25).

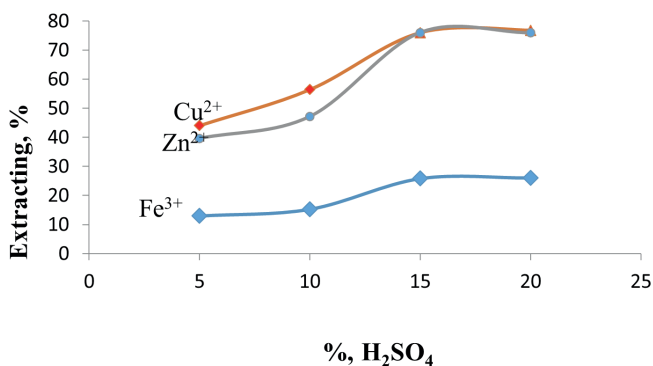


Figure 25. Extraction of non-ferrous metals and iron from pyrite stubs into solution of H_2SO_4

According to the data obtained, optimum is the use of sulphuric acid solution with concentration of 15% H_2SO_4 for leaching. Under these conditions the extraction into sulphuric acid solution was, %: Cu^{2+} 76.8; Zn^{2+} 75.9 and Fe^{3+} 26.0.

To compare the results obtained, pyrite stubs were leached without activation, and the degree of extraction of non-ferrous metals into a sulfuric acid solution was 15-20% lower.

To obtain non-ferrous metals concentrate the sulphuric acid leaching solution was neutralized with potash in several stages (Table 3).

Table 3 - Chemical composition of neutralisation deposits as a function of solution pH

Composition, %	pH		
	3.7	5.46	9.7
K ₂ O	0.11	0.08	13.8
MgO	-	-	1.2
Al ₂ O ₃	0.12	3.0	7.5
SiO ₂	0.12	1.2	2.9
P ₂ O ₅	25.27	3.5	0.02
SO ₃	8.3	11.08	15.9
Fe ₂ O ₃	53.37	19.3	3.8
CuO	-	0.04	6.4
ZnO	-	0.01	12.54
As ₂ O ₃	3.08	0.39	-
SeO ₂	0.1	0.01	-

Neutralisation to pH 3.7 was performed to deposit trivalent iron from the solution. Under these conditions phosphorus, arsenic and selenium partly settled out of solution together with iron.

At pH 5.46 iron, phosphorus and arsenic remnants were separated into a neutralisation deposit from the solution.

Neutralisation to pH 9.7 was performed taking into account the pH of complete precipitation of zinc and copper. As a result, the concentrate of non-ferrous metals with content, wt. % was received: CuO 6.4; ZnO 12.55. The concentrate yield was 8.5% of the total amount of neutralization deposits or 1.5% of the initial mass of pyrite slag. The yield of neutralization residue at pH 5.46 was 2.1%, at pH 9.7 – 14.2%.

From the obtained results it follows, that neutralization for separation of Fe₂O₃, P₂O₅ and As₂O₃ impurities and for production of concentrate of non-ferrous metals should be performed in two stages up to pH 5.46 and 9.7.

For complex processing of pyrite slag, cast iron was obtained from iron-containing cakes leaching pyrite slag in sulphuric acid solution.

Smelting of cast iron was performed based on obtaining metallic iron in the form of blooming cast iron.

The quality of the bloom depends on the composition of the slag produced. For good quality, slags containing 50 – 60% SiO₂, 10 – 20% Al₂O₃, 3 and 15 –

25% CaO+MgO are needed, so 20% CaO is added to the charge. The required Al_2O_3 and SiO_2 content in the charge is sufficient.

The charge for smelting cast iron consisted of cake, calcium oxide and 10% anthracite as a reducing agent.

The charge was briquetted before smelting. Smelting was performed in graphite crucibles in a muffle furnace at 1350°C for 2 hours.

After magnetic separation the unmetallized slag and cast iron in the form of blooming metal with a particle size of 1 - 3 mm were obtained. (Figure 26).



Figure 26. Cast iron

Conclusion. The phase composition has changed during chemical pyrite slag activation in a solution of sodium bicarbonate at temperature 120°C: zinc oxide phosphate hydrate and dolomite phases disappeared; sodium thiophosphate phase increased; albite and natrojarosite phase decreased; magnesium aluminosilicate phase appeared; calcium silicate and sodium thiophosphate phase;

At leaching of pyrite slag in 15% H_2SO_4 solution extraction in solution, wt.% was received: CuO 76.8; ZnO 75.9 and Fe_2O_3 26.0 that is 15 – 20% higher than the recovery without activation.

Staged neutralization of the leaching solution of pyrite slag at pH 9.7 resulted in non-ferrous metal concentrate, wt. %: CuO 6.4; ZnO 12.55.

The method of reduction smelting obtained nodular cast iron.

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