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Nitric Acid Processing of Serpentenites from the Karakalpak Deposit

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Abstract

Physicochemical foundations for the technology of nitrogen acid processing of serpentinite from the Karakalpak deposit with the formation of pure magnesium nitrate and magnesium-containing compound fertiliser are developed. The chemical and mineralogical composition of serpentinite from the Karakalpak deposit is studied. As determined by X-ray diffraction analysis, microscopic and thermogravimetric studies, serpentinite sample contains clinoenstatite, pyrope, spinel, magnetites and other minerals with various crystal structures. Data on the extraction of serpentinite components in nitric acid by decomposition under different conditions (temperature, duration, nitric acid concentration) are presented. The subsequent stages of nitric acid extract processing have been carried out: removal of silica residue, ammonisation of the filtrate in order to precipitate undesirable impurities to obtain magnesium nitrate. The process of evaporation and crystallization of the filtrate to obtain pure magnesium nitrate has been studied.

Keywords: magnesium nitrate, serpentinite, processing, nitric acid treatment, fertilisers

INTRODUCTION

Magnesium nitrate is widely used in many branches of the chemical industry. Due to its properties, this compound is a compulsory component in many technological processes in various industrial areas [1].

In our opinion, chemical processing of substandard magnesium-containing materials (dolomite, talc, serpentinite, *etc.*) is of practical interest if insufficiency of magnesium-containing fertilizers provided to the agriculture of the Republic of Uzbekistan is taken into account [2].

The resources of silicate magnesium-containing rocks composed mainly of the minerals of serpentinite group, such as lizardite, chrysotile, antigorite, etc., are present in Uzbekistan. To obtain magnesium compounds from local raw material resources, their integrated processing is necessary, which will make it possible to extract all useful components from them and to manufacture commercial products [3].

During the recent years, many technological flowcharts on integrated processing of serpentinites have been developed. They are based on such chemical processes as leaching or agglom-

eration [4–12]. Each deposit of natural serpentinite is unique (because the mineral compositions of the deposits and properties vary broadly), so, the known technologies are non-applicable to processing Karakalpak serpentinite. Prospecting works aimed at evaluation of the potential of Karakalpak serpentinite mining were carried out by many geologists [13]; expected predicted serpentinite resources down to the depth of 50 m, stretching for 13.5 km, with outcrop width 150 m and bulk mass 2.57 kg, reach 270 mln t. Based on the features of any raw material, special research and technological studies are necessary to choose the optimal conditions for its processing.

Magnesium enhances the productivity of agricultural crops. As a rule, mineral fertilizers manufactured in Uzbekistan do not contain magnesium. The Republic of Uzbekistan is in need of processing the own magnesium-containing raw material, which is imported at present. Such issues as localisation of imported raw material, semi-products, and the use of natural resources provide elevated attention to serpentinite inside Uzbekistan. Thus, studies aimed at the development of physicochemical foundations and technology of processing serpentinite from the Karakalpak deposit into magnesium nitrate, with simultaneous production of magnesium-containing nitrogen-based fertilisers, are highly relevant and form one of the major tasks of the present study.

EXPEIMENTAL

Elemental composition and morphology were studied by scanning electron microscopy (SEM) with the help of SEM-EVO MA 10 scanning electron microscope (Zeiss, Germany) and energy-dispersive X-ray spectroscopy (EDX) over a local region using energy-dispersive element analyser Aztec Energy Advanced X-act SDD (Oxford Instruments, UK). The data on the elemental composition are presented by electron micrographs with selected local regions, composition table, and graphical spectrum.

The phase characteristics of the sample under investigation were measured with a powder X-ray diffractometer Empyrean (Panalytical, the Netherlands) equipped with a copper tube (K_α = 1.5406 Å). The whole process of equipment operation was computer-controlled using Data Collector software, while X-ray diffraction patterns were analysed with High Score software using the PDF 2013 database. Measurements were carried out at room temperature within 2θ angle range from 5 to 90°, in the mode of step-by-step scanning in increments of 0.013° and signal accumulation time 5 s at a point.

Thermal analysis of the samples (TG, DSC) was carried out with a synchronous thermoanalyser STA PT 1600 (Linseis, USA). Measurements were carried out in the oxidative environment with a heating rate of 20 °C/min.

Liquid phases were analysed by mass spectrometry with inductively coupled plasma (ICP-MS). Measurements were carried out using a highly sensitive multielement analyser ICP-MS 7700 (Agilent Technologies, USA).

RESULTS AND DISCUSSION

The mineralogical composition of serpentinite from the Karakalpak deposit was determined on the basis of results obtained by chemical, X-ray diffraction (XRD) and thermal methods of analysis. According to the data of chemical analysis, serpentinite of the Karakalpak deposit contains 33–34 wt% MgO and 41–42 wt% SiO_2 (Table 1).

According to XRD data, reflections related to the phases of magnesium silicate $Mg_3Al_2(SiO_4)_3$ [01-089-5686], SiO_2 [01-073-3412], Mg_4Zn_7 [03-065-1260], and the phases of magnetite ($(Mg_{0.198}Fe_{0.802})(Mg_{0.802}Fe_{1.198})O_4$ [01-076-2846]), birnessite ($Na_{0.58}(Mn_2O_4)(H_2O)_{1.38}$, dolomite ($CaMg(CO_3)_2$ [01-075-3697]), spinel ($MgAl_2O_4$ [00-021-1152]), clinoenstatite ($MgSiO_3$ [01-076-1931]) and pyrope ($Mg_3Al_2(SiO_4)_3$ [01-080-5007]) were identified in initial serpentinite [14].

Serpentinite from the Karakalpak deposit is inhomogeneous in its structure. Several particles

TABLE 1

Chemical analysis of serpentinite, wt%

Sample No.	MgO	FeO	Fe_2O_3	Al_2O_3	CaO	Na_2O	SiO_2	Losses on ignition
1	33.06	1.62	6.63	2.47	1.82	1.56	41.42	11.42
2	33.00	2.20	6.83	2.21	4.76	0.08	40.29	11.52

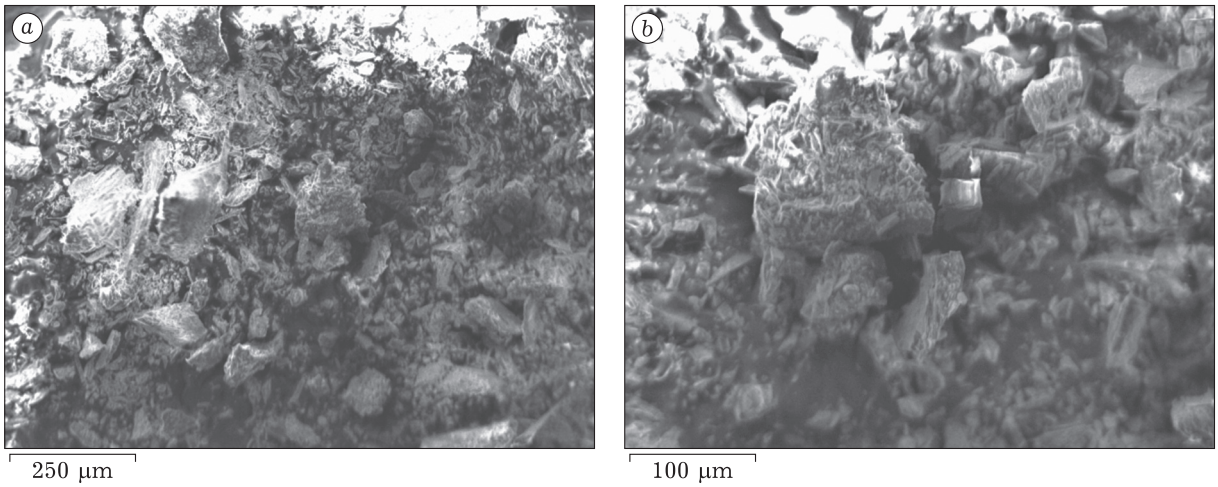

Fig. 1. SEM images of initial serpentinite with different magnification [14].

of different shapes relating to different minerals are distinguished in Fig. 1.

The next step involved the tests aimed at choosing the optimal leaching conditions under which all valuable components are maximally extracted from serpentinite. With nitric acid norm, which accounted for 100 % with respect to the sum of metal oxides, the technological parameters of the process were varied within the following ranges: nitric acid concentration 30–50 wt%, process temperature 40–95 °C, process duration 30–240 min.

Leaching was performed using the stirred reactor, which was a flask fixed in a thermostat. Nitric acid of definite concentration was poured into the flask. The acid was supplied assuming dissolution of all metals that are present in serpentinite. Then, after achieving the prescribed

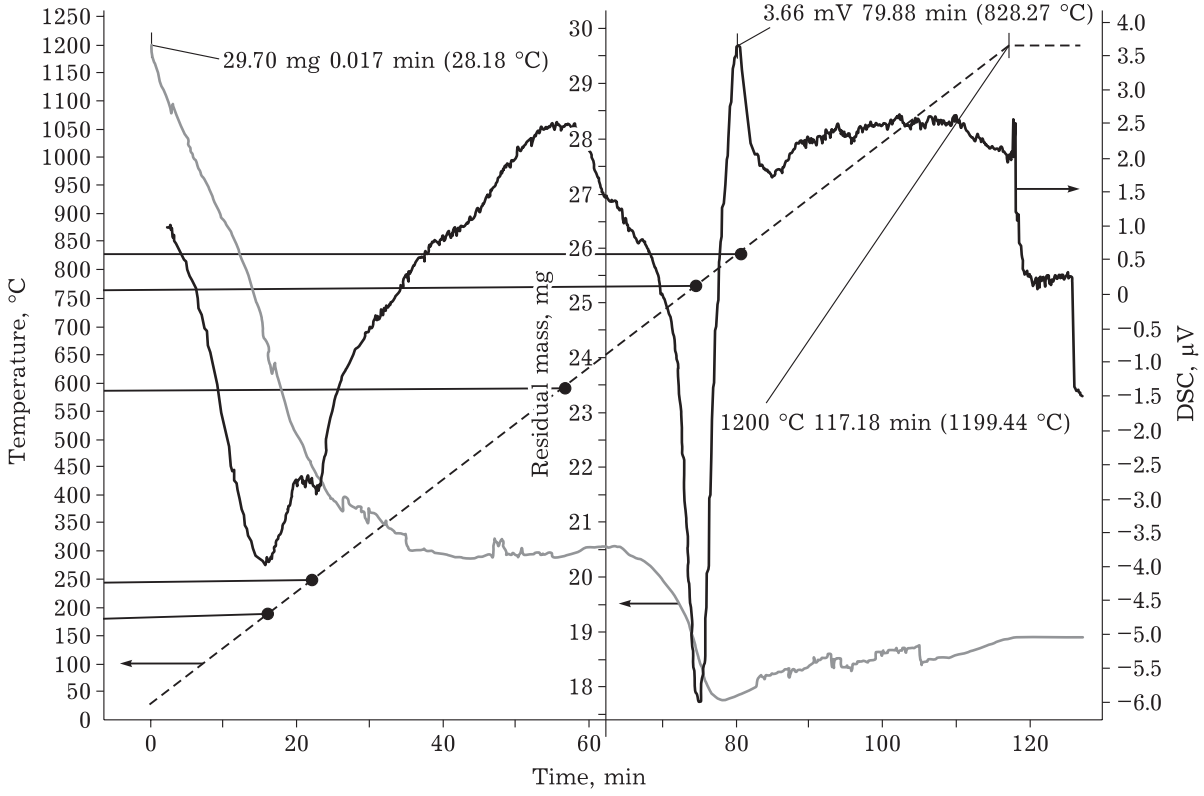

Fig. 2. Derivatogram of serpentinite [14].

TABLE 2

Effect of technological parameters on the analytical parameters of nitric acid decomposition of thermally treated serpentinite

Sample No.	Temperature of thermal treatment, °C	Duration of thermal treatment, min	concentration HNO_3, %	Duration of decomposition process, min	Temperature of decomposition process, °C	L : S* ratio in suspension after decomposition	Density of liquid phase, g/cm^3	Degree of MgO recovery into the liquid phase, %
1	450	30	30	30	95	4.0 : 1	1.2240	26.2
2	450	60	30	60	95	4 : 1	1.2166	28.5
3	450	90	30	90	95	3.9 : 1	1.2025	27.2
4	587	90	30	90	95	2.9 : 1	1.1010	22.2
5	765	90	30	90	95	1 : 1	1.1860	57.2
6	800	60	30	30	70	1 : 1.9	1.6160	46.3
7	800	60	30	30	80	1 : 1.3	1.1430	67.4
8	800	60	30	30	95	1 : 1.6	1.1899	58.1
9	800	60	25	30	70	1 : 2.16	1.1500	49.2
10	800	60	25	30	80	1 : 1.5	1.1130	54.4
11	800	60	25	30	95	1 : 1.7	1.2677	53.0
12	800	30	30	120	95	1 : 1.3	1.2826	66.5
13	800	60	30	120	95	1 : 1.25	1.3232	72.2
14	800	90	30	120	95	1 : 1.1	1.0394	75.2
15	800	90	30	30	95	1 : 1.05	1.6360	68.2
16	800	90	30	60	95	1 : 1.07	1.1300	68.3
17	800	90	30	90	95	1 : 1.1	1.1970	73.3
18	800	60	35	30	70	1.25 : 1	1.3220	51.4
19	800	60	35	30	80	1.3 : 1	1.3049	53.4
20	800	60	35	30	95	1.15 : 1	1.4507	63.3
21	800	60	30	30	95	1 : 0.9	1.2863	70.0

* L – liquid; S – solid.

temperature, serpentinite with particles smaller than 0.315 mm was added. After acid treatment, the resulting suspension was separated by filtering from the insoluble silicic residue. The authors of [15] have shown that the yield of magnesium oxide into the liquid phase from serpentinite decomposition is 24.4 %, while the yield from thermally treated serpentinite is more than twice as large. For this reason, we studied the effect of thermal treatment temperature on magnesium oxide yield from serpentinite.

Peaks related to the stepwise dehydration of the mineral are clearly determined in the derivatogram (Fig. 2). The first doublet endothermic effect related to the removal of adsorbed water is observed within the temperature range 200–270 °C (along the TG curves, mass loss in this region is 27.5 %). The second endothermic effect is due to the formation of Mg_2SiO_4 structure during mineral dehydration and recrystallisation (relative mass loss is 8.75 %).

As mentioned above, the content and yield of magnesium oxide in the form of magnesium nitrate from serpentinite decomposition are 2.99 and 24.4 %, respectively, while for decomposition of thermally treated serpentinite these parameters increase to 8.46 and 75 %, respectively. For instance, at 450, 587, 765 and 800 °C and identical values of other parameters in experiments 3, 4, 5 and 17 (Table 2), the yield of magnesium oxide into the liquid phase is 27.2, 22.2, 57.2 and 73.3 %, respectively.

The elemental composition of the nitric acid extract from serpentinite according to ICP-MS data is presented in Table 3. One can see than Al, Cr, Fe and Ni concentrations in the filtrate are not more than several hundredths of magnesium concentration. Aluminium and iron serve as initial substances to obtain coagulants. Chromium-nickel-manganese concentrate is the raw material used to obtain the salts of indicated metals. Results of the chemical analysis of the liquid phase of nitric acid extract are shown in Table 4.

TABLE 3

Elemental composition of the nitric acid extract of serpentinite according to ICP-MS data

Sample No.*	Content, g/L								
	Mg	Al	Ca	Cr	Mn	Fe	Ni	Zn	Na
6	29	0.51	0.0016	0.28	0.068	0.2	0.41	0.0052	0.055
7	36	0.64	0.002	0.36	0.084	0.25	0.52	0.0067	0.060
8	30	0.60	0.0018	0.31	0.072	0.22	0.43	0.0085	0.079
11	34	0.60	0.002	0.35	0.079	0.25	0.49	0.023	0.058
14	33	0.69	0.0019	0.36	0.093	0.27	0.47	0.006	0.049
15	27	0.55	0.001	0.33	0.073	0.23	0.41	0.0036	0.034
16	27	0.78	0.0013	0.34	0.082	0.25	0.41	0.0042	0.10
17	29	0.59	0.0015	0.33	0.076	0.24	0.44	0.0045	0.054
20	28	0.53	0.0016	0.29	0.068	0.21	0.41	0.0039	0.054
21	24	0.47	0.0014	0.27	0.063	0.2	0.32	0.0051	0.049

* Sample number corresponds to numbering in Table 2.

The effect of technological parameters (precipitate/water ratio, washing conditions) on the efficiency of washing the insoluble residue was also studied. Washing conditions were: 1) decanting by pouring off a definite amount of water above the settled precipitate using a funnel with a filter (without mixing); 2) washing on the filter after precipitate filtration, using a definite amount of water (under mixing). Results of the studies are presented in Table 5.

It follows from the obtained data that washing extent increases with a 2-fold increase in the amount of water: in the case without mixing - from 26.4 to 59.1 %, while under mixing it increases from 51.3 to 68.2 %. Mixing during washing provides an increase in washing extent by 24.9 % (for precipitate/water = 1 : 1) and by 9.1 % (for precipitate/water = 1 : 2), and filtration rate increases in this case by a factor of 3–4 in comparison with washing without mixing.

The SEM images and EDX spectra of non-dissolved precipitate obtained after nitric acid leaching of serpentinite are shown in Fig. 3. According to the data obtained, Si content in the precipitate after washing (see Fig. 3, *b*) is 36.7–38.4, Ca – 0.1–0.2, Mg – 4.9–6.1, Al – 0.9–1.0 wt%. At the same time, according to XRD data, non-dissolved residue after washing contains 83 % calcium aluminosilicate ($CaAl_2Si_2O_8$ [01-075-1587]) and 17 % magnesium-calcium silicate ($CaMgSiO_4$ [01-075-1569]). Therefore, 70–76 % of SiO_2 is present in the amorphous form, while the rest part (24–30 % of the total mass) is present as the crystal phase composed of calcium-aluminium and calcium-magnesium silicates.

Then the process of magnesium nitrate isolation from nitric acid extracts of serpentinite was studied. To precipitate iron and aluminium oxides from the liquid phase, nitric acid extract was saturated with ammonia from pH 1 to pH 6. Ammonia-saturated solutions were filtered and separated from impurities. Results of the analysis of ammonia-saturated solutions and the effect of technological parameters on ammonation process are listed in Tables 6 and 7, respectively.

One can see in the data presented in Table 6 that the concentrations of aluminium and iron impurities decreased from 0.47 and 0.12 to 0.0001 and 0.00066 g/L, respectively, with an increase in solution pH from 0.56 to 6.03. It is shown that magnesium concentration in solution increases from 24.0 to 73.0 g/L with an increase in pH to 3.1–3.7, and impurity oxides like M_2O_3 (where M = Al, Fe, Cr, Mn) precipitate almost completely, while soluble magnesium and ammonium nitrate remain in the solution. It has been determined that some amount of magnesium gets precipitated with a further increase in pH to 6.03, and the concentration of magnesium ions in solution decreases to 31 g/L.

After saturation with ammonia and evaporation of the resulting precipitate to obtain it in the solid state, nitrogen-magnesium fertiliser with aluminium and iron impurities at a level of 0.02 and 0.012 wt%, respectively, can be obtained. As a result of ammoniation carried out at solution pH equal to 3, 4 and 5, the samples of nitrogen-magnesium fertiliser with the sum of nutrition elements (nitrogen + magnesium) 39.5; 44.2 and 41.46 wt% were obtained, in particular, nitrogen percentage was 21.86; 22.5; 23.86 wt% (among them,

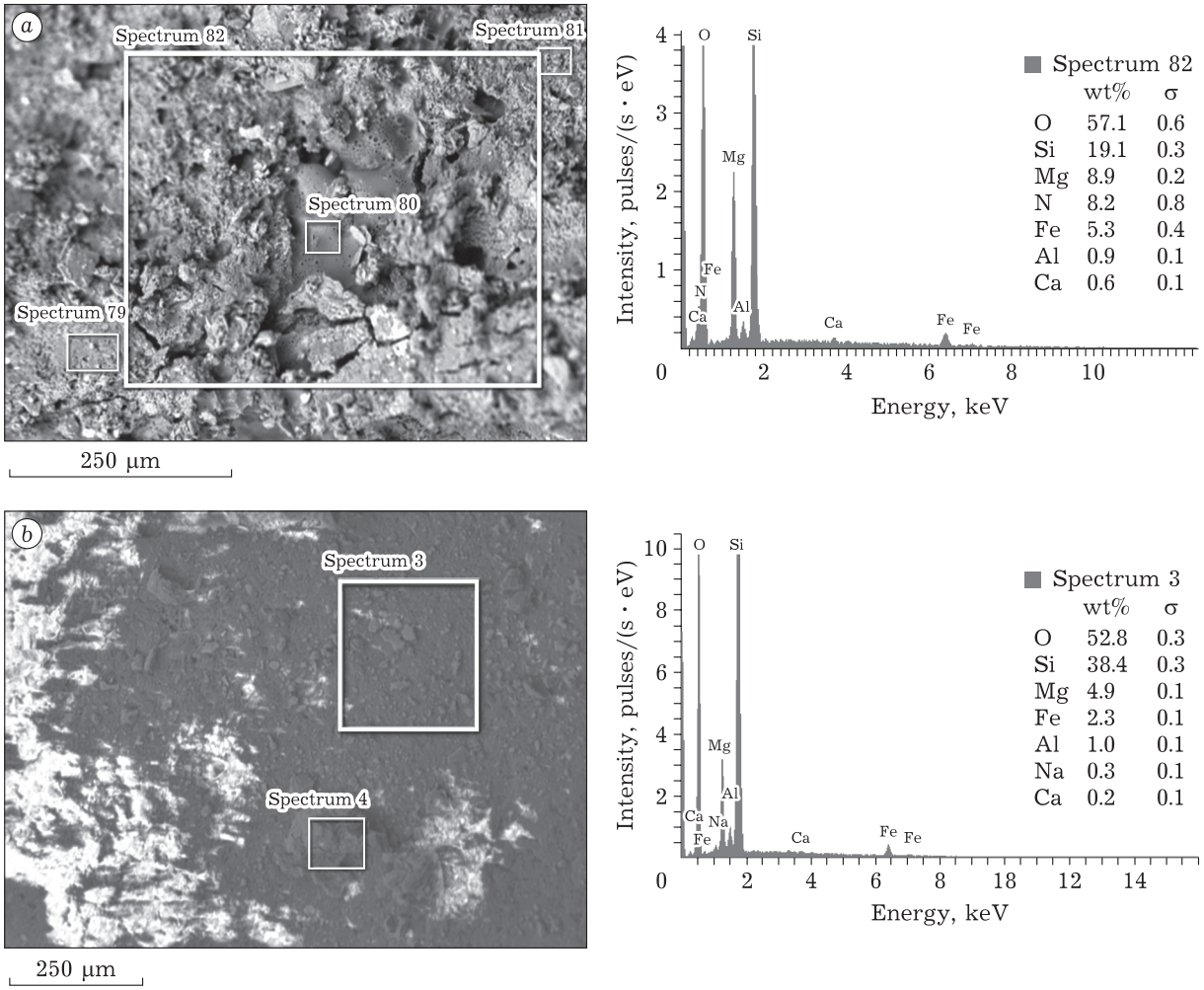

Fig. 3. Micrographs and EDX spectra of insoluble precipitate before (*a*) and after (*b*) washing.

5.44; 4.74; 6.7 wt% are in ammonium form), magnesium concentration was 18.3; 21.7; 17.4 wt%, respectively.

Pure magnesium nitrate is required in different branches of the national economy, so we studied the possibility to obtain magnesium nitrate hexahydrate from the nitric acid extract of serpentinite from Karakalpakstan.

The effect of technological parameters of ammonation, evaporation, and crystallisation of the nitric acid extract of serpentinite on the purity of resulting magnesium nitrate hexahydrate was investigated. Experiments show that the formation of magnesium nitrate hexahydrate crystals during ammonation and crystallisation is accompanied by the formation of ammonium nitrate in the concentration from 6 to 21 %, depending on evaporation degree and the amount of nitric acid.

For this reason, to obtain purer magnesium nitrate hexahydrate, crystallisation products were washed with saturated magnesium nitrate solution at 10 °C. The concentration of ammonium nitrogen decreased to 0.012 %, which corresponds to the pure reagent grade of magnesium nitrate hexahydrate according to GOST 11088-75. The micrographs of magnesium nitrate hexahydrate

TABLE 4

Chemical composition of the liquid phase of the nitric acid extract

Sample No.*	Component content, %				
	MgO	CaO	Fe_2O_3	SiO_2	Al_2O_3
1	1.88	0.129	0.20	0.04	0.01
2	1.87	0.122	0.24	–	0.01
3	1.50	0.124	0.24	–	0.01
12	6.78	0.133	0.24	–	0.01
13	7.28	0.129	0.24	–	0.01
14	7.95	0.136	0.30	–	0.01

* Sample number corresponds to numbering in Table 2.

TABLE 5

Effect of technological parameters (precipitate/water ratio, mixing) on the efficiency of precipitate washing

Mass, g		MgO content, wt%		Washing degree, %
Precipitate	Water	Liquid phase	Solid phase	
Without mixing				
10	10	1.71	8.86	26.4
10	15	1.47	8.36	36.4
10	20	1.84	7.51	59.1
Under mixing				
10	10	1.81	6.06	51.3
10	15	1.54	5.34	59.7
10	20	1.33	4.50	68.2
10	40	1.22	2.30	95.6

TABLE 6

Results of the analysis of ammonia-saturated solution composition by ICP-MS

No.	pH	Element content, g/L						
		Na	Mg	Al	Fe	Ca	Cr	Mn
Initial solution	0.56	0.043	24.0	0.47	0.12	0.0014	0.37	0.067
1	0.96	0.12	67.0	0.0013	0.18	0.0035	0.9	0.18
2	1.49	0.11	64.0	0.0012	0.026	0.001392	0.41	0.16
3	2.17	0.14	72.0	0.0014	0.0071	0.001393	0.098	0.22
4	2.48	0.13	71.5	0.84	0.036	0.001394	0.13	0.2
5	3.10	0.13	73.0	0.05	0.00083	0.001395	0.005	0.18
6	3.51	0.10	71.0	0.029	0.00059	0.001396	0.0015	0.16
7	4.70	0.091	59.0	0.0018	0.00064	0.001397	0.00034	0.13
8	5.08	0.076	46.0	0.0019	0.00069	0.001398	0.00063	0.091
9	6.03	0.058	31.0	0.0001	0.00066	0.001399	0.00014	0.01

TABLE 7

Effect of technological parameters on ammonation of the nitric acid extract

No.	pH	L : S* ratio in the system after ammonation	Rheological characteristics of liquid phases at 20 °C	
			Density, g/cm^3	Viscosity, cm^2/s
1	0.96	19.7 : 1	1.255	1.559
2	1.49	9.4 : 1	1.258	1.456
3	2.17	6.9 : 1	1.288	1.628
4	2.48	7.2 : 1	1.302	1.662
5	3.10	4.0 : 1	1.276	1.459
6	3.51	3.6 : 1	1.245	1.313
7	4.70	4.2 : 1	1.275	1.335
8	5.08	4.5 : 1	1.278	1.418
9	6.03	1.7 : 1	1.249	1.461

* L – liquid; S – solid.

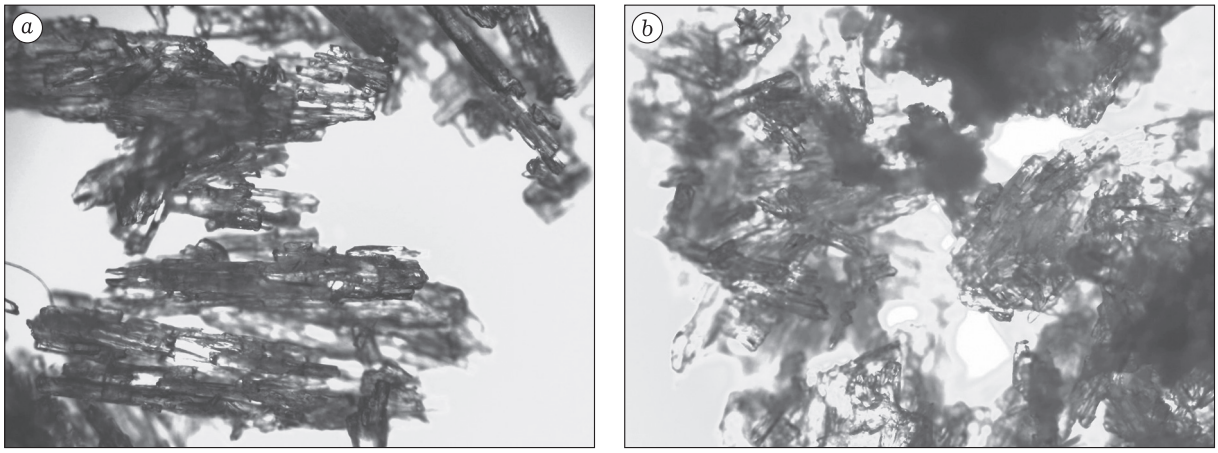

Fig. 4. Micrograph of magnesium nitrate hexahydrate crystals obtained from serpentinite of the Karakalpak deposit.

crystals obtained from serpentinite of the Karakalpak deposit are shown in Fig. 4.

CONCLUSION

Mineralogical composition of serpentinite from the Karakalpak deposit was determined on the basis of results obtained by chemical, X-ray diffraction and thermal analysis. The composition of serpentinite ore is as follows, wt%: SiO_2 40.29, CaO 4.76, Al_2O_3 2.21, Fe_2O_3 8.83, MgO 33.0, TiO_2 0.08, CaO 0.86, SO_3 0.10, losses on ignition 11.52. The mechanism of transformations taking place in magnesium silicate components of the ore under the action of temperature and nitric acid has been investigated. Experiments show that the maximal extent of leaching the soluble components from serpentinite (75.2 %) was achieved by decomposition with nitric acid at 95 °C for 120 min after preliminary thermal treatment (800 °C, 60 min). The following optimal conditions are recommended for the process: acid concentration 30 %, total norm of acid consumption for decomposition of acid-soluble components of the raw material 100 % of the stoichiometric amount. After decomposition, the pulp was separated into liquid and solid phases by vacuum filtration. Analysis of the dried and washed precipitate shows that under the indicated conditions the major ore components are extracted into solution almost completely, while the degree of MgO recovery reaches 95 %. It has been established that about 9.8–10.0 % MgO remains in the precipitate, mostly because of sludge humidity. At the next stage of the process, the filtrate (containing magnesium nitrate in nitric acid) was subjected to ammonation. The salts of aluminium and iron were separated in the form of precipitate by filtration, and then the residual solution containing mainly magnesium and ammonium nitrates was evaporated; mass loss was 30–60 %. The major part of magnesium remains in the nitric acid solution. It is recommended to process the filtrate into complex nitrogen-sodium fertilisers. The saturated solution was cooled to 20 °C in a crystallisation vessel. The formed crystals were separated by filtering, washed with the saturated solution of magnesium nitrate. The results of chemical analysis show that, depending on ammoniation degree and washing, magnesium nitrate hexahydrate contains: $Mg(NO_3)_2 \cdot 6H_2O$ (up to 98 wt%), NH_4NO_3 (0.2–1 wt%), Fe_2O_3 (<0.001 wt%), CaO (<0.001 wt%), which corresponds to the pure reagent grade according to GOST 11088-75.

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