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Extraction of Valuable Elements from Ash of Fuel Shale of the Aktau and Sangruntau Deposit

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ABSTRACT

Hydrometallurgical methods are aimed at leaching for the extraction of valuable components into a sulfuric acid solution, as well as selective extraction on ion-exchange resins by the method of sorption and precipitation of valuable components such as vanadium oxide, molybdenum oxide, zinc oxide and the amount of REE oxide and uranium oxide. The presented methods are not implemented on an industrial scale, since there is no processing of oil shale in the Republic. A prospective scheme for processing ash from oil shale deposits has been analyzed. It includes the addition of sodium chlorine to the oxidative roasting process, the resulting ash is leached in vats with sulfuric acid 150 g/l, at room temperature, S:L= 1:5, duration 60 minutes. The resulting metal-bearing sulfuric acid solution is filtered and sent to sorption, as well as precipitation. The extraction of metals into solution is,%: vanadium - 84.3 - 85.6, molybdenum - 95.4 - 88.1, zinc - 69.6 - 71.1, uranium - 60.3 - 64.9 and the amount of REE - 66.2 - 49.2, respectively.

For the extraction of metals from productive solutions of leaching ash from oil shale deposits of Aktau and Sangruntau, the following ion-exchange resins of the Anglia company PUROLITE were used: for vanadium grade A-109 extraction 79 - 81%, for molybdenum - grade A-100 extraction was more than 84 - 86% , uranium grade A-560 extraction 81 - 79% and for zinc S-984 extraction 75 - 71%.

Carried out precipitation to extract REE and received 18% content of the amount of REE.

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INTRODUCTION

Oil shale was formed at the bottom of the seas about 450 million years ago as a result of the simultaneous deposition of organic and inorganic silt [1-4]. In oil shales, the content of the mineral part, as a rule, prevails over the organic part. The composition of the mineral part of the shales is extremely diverse: SiO_2, Al_2O_3, CaO, Fe_2O_3, MgO, etc., there is also a number of rare and trace elements: W, Ge, Co, Cu, Mo, Pb, etc. [5].

The content of organic matter in oil shale - kerogen - is usually from 10-15 to 40-50%. The elemental composition of the organic part of oil shale is presented in table. 1. Oil shale is characterized by a very high atomic ratio H:C of 1.2 - 1.8 [6].

Table 1 Elemental composition of the organic part of oil shale

№	Significative	Shale OM, %
1	Carbon	75-78
2	Hydrogen	9-10
3	Oxygen	10
4	Sulfur	1,5-1,7

As you can see from the table. 1 organic part of oil shale mainly contains carbon from 75 to 78%, hydrogen from 9 to 10%, oxygen up to 10% and sulfur from 1.5 to 1.7%.

The composition of the mineral part of oil shale differs depending on the deposit. For example, the ash of the Leningrad shale of the Baltic basin will contain about 36% of CaO, of which up to 20% is free oxide. The main component of shale ash is SiO_2 slag glass [7].

Ash can be used as a raw material in a variety of applications, including the cement industry, due to its cementitious properties.

There are huge reserves (47.0 billion tons) of oil shale on the territory of the Republic of Uzbekistan [8-10].Only in the Kyzylkum basin there are deposits with predicted reserves of oil shale in the amount of 24.6 billion tons. At the Baysun, Sangruntau, Aktau, Uchkyr-Kulbeshkak, Urtabulak deposits, the reserves of oil shale are more than 1.0 billion tons. [11,12].

Oil shale of Uzbekistan, in addition to carbon raw materials, contains V, Mo, Au, W, Ag, Re, Cd, Se, Cu, Ni, Pb, S, U, including rare earth metals and platinum group metals.

In this regard, laboratory and technological research was carried out to extract valuable components from the ash of oil shale deposits Aktau and Sangruntau (after pyrolysis of shale) using heat treatment (pyrolysis), oxidative roasting and hydrometallurgical enrichment method.

The object of research was semi-coke after the process of heat treatment (pyrolysis) of oil shale from the Aktau and Sangruntau deposits.

Results and their discussionthe semi-coke obtained at the pyrolysis stage, according to the classical scheme, should go through the oxidative roasting stage, i.e. combustion in the presence of air. At this stage, the residual organic matter of the semi-coke burns out and the material is ashed.

For this, a special furnace was created for carrying out oxidative firing, under conditions of intensive air supply to the combustion chamber. The construction of such a furnace makes it possible to simulate the corresponding process at UTT-3000.

At the installation, research was carried out on the decomposition of shale semicoke, for the oxidation of organics and sulfides in the temperature range of 850 ° C with the addition of sodium chlorine, which allows high extraction of valuable components into the metalliferous solution by leaching.

To determine the chemical composition, ashes were used, which are finely ground uniformly granular powders. Samples weighing 3g were analyzed on an ICP-MS mass spectrometer. The results are shown in table 2.

Table 2 The content of individual elements (ppm), their concentration clarkes in the original ore and in the ash of oil shale deposits according to datamass spectrometric analysis (ICP-MS)

The elements	Clark in the earth's crust	Актау		The elements	Clark in the earth's crust	Сангрунтау	
		Initial	Ash			Initial	Ash
Li	32,0	23,0	26,4	**Li**	32,0	29,0	31,9
Be	3,8	1,4	1,89	**Be**	3,8	2,2	2,6
Na	25 000	7800	12 800	**Na**	25 000	5 500	9 650
Mg	18 700	17000	14 800	**Mg**	18 700	16 000	15 200
Al	80 500	63000	66 600	**Al**	80 500	69 000	71 500
K	25 000	13000	15 400	**K**	25 000	16 000	18 600
Ca	29 600	66000	47 600	**Ca**	29 600	31 000	26 530
Sc	10,0	12,0	11,6	**Sc**	10,0	15,0	13,5
V	90,0	880,0	915,0	**V**	90,0	860,0	895,0
Cr	83,0	95,0	178,0	**Cr**	83,0	150,0	174,0
Mn	1 000	610,0	528,0	**Mn**	1 000	230,0	200,0
Fe	46 500	33000	35 200	**Fe**	46 500	42 000	44 800
Co	18,0	13,0	13,2	**Co**	18,0	15,0	15,4
Ni	58,0	220,0	131,0	**Ni**	58,0	190,0	155,0
Cu	47,0	77,0	79,8	**Cu**	47,0	100,0	104,2
Zn	83,0	130	149	**Zn**	83,0	160,0	168,4
As	1,7	7,3	0,2	**As**	1,7	16,0	0,3
Se	0,05	8,7	9,3	**Se**	0,05	15,0	16,8
Sr	340	320,0	361,0	**Sr**	340	300,0	330,5
Y	20,0	26,0	31,1	**Y**	20,0	37,0	41,2
Nb	20,0	7,3	7,18	**Nb**	20,0	9,3	8,6
Mo	1,1	360,0	397,0	**Mo**	1,1	270,0	330,0
Cd	0,13	21,0	5,94	**Cd**	0,13	32,0	14,6
Sn	2,5	2,5	0,88	**Sn**	2,5	2,6	1,3
Sb	0,5	7,2	0,8	**Sb**	0,5	5,5	2,3
Te	0,001	0,36	0,18	**Te**	0,001	1,6	1,1
Ba	650	860,0	831,0	**Ba**	650	420,0	326,0
La	29,0	21,0	34,9	**La**	29,0	28,0	35,1
Ce	70,0	32,0	56,9	**Ce**	70,0	36,0	44,6
Pr	9,0	3,7	9,4	**Pr**	9,0	4,6	8,8
Nd	37,0	16,0	33,6	**Nd**	37,0	20,0	38,5
Sm	8,0	4,7	4,6	**Sm**	8,0	6,0	6,2
Eu	1,3	1,6	1,27	**Eu**	1,3	1,7	0,9
Gd	8,0	3,4	6,29	**Gd**	8,0	4,1	7,25
Tb	4,3	0,68	0,784	**Tb**	4,3	0,79	0,84
Dy	5,0	3,6	5,07	**Dy**	5,0	4,9	6,3
Ho	1,7	0,89	1,4	**Ho**	1,7	1,2	1,8
Er	3,3	2,6	2,49	**Er**	3,3	3,0	3,1
Tm	0,27	0,4	0,341	**Tm**	0,27	0,65	0,52
Yb	0,33	3,1	3,16	**Yb**	0,33	3,9	4,0
Lu	0,8	0,27	0,291	**Lu**	0,8	0,46	0,50

∑REE	208	131,9	203,2	**∑REE**	208,0	167,3	213,11
W	1,3	1,5	3,05	**W**	1,3	0,67	1,85
Tl	1,0	12,0	33,1	**Tl**	1,0	2,3	6,4
Pb	16,0	14,0	11,0	**Pb**	16,0	16,0	14,3
Th	13,0	8,6	4,5	**Th**	13,0	9,7	5,4
U	2,5	54,0	52,0	**U**	2,5	33,0	30,0

Based on the data in table 2, in the ash of oil shale, a product of oxidative roasting at a temperature of 8500C, the content of valuable metals in the Aktau and Sangruntau deposits increases markedly compared to the original ore, ppm: vanadium from 880 to 915 and from 860 to 895, molybdenum from 360 to 397 and from 270 to 330, the sum of rare elements from 131.9 to 203.2 and from 167.3 to 213.11, as well as thallium from 12 to 33.1 and from 2.3 to 6.4, etc.

Hydrometallurgical research.
In order to extract valuable metals from the ash of oil shales, leaching was carried out at the deposit.

Leaching was carried out in heat-resistant glasses and flasks with a capacity of 0.5 and 1.0 liters under draft. Pulp agitation was carried out by mechanical and pneumatic methods.

The recommended scheme of leaching was carried out at different grades of grain size (initial, -0.5, -0.08 mm), as well as under different conditions and acids. The optimal developed technological scheme for the processing of oil shale ash is presented under the following conditions, the result is shown in Fig. 1: ash size class -0.5 mm, duration 60 min, ratio S:L = 1:5, room temperature, starting material weight 100 g.

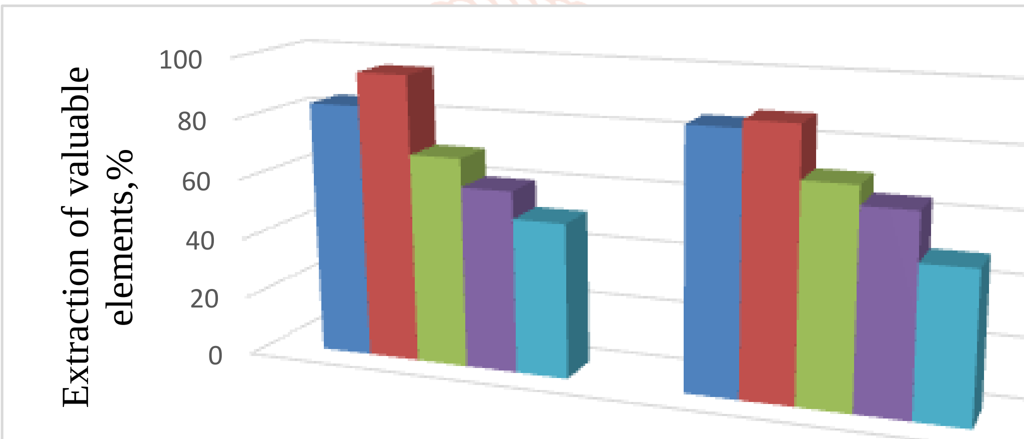

Fig 1. Diagram of the extraction of valuable components into a sulfuric acid solution

As can be seen from Fig. 1, the leaching of ash from oil shale from the Aktau and Sangruntau deposits with the use of sodium chlorine in the roasting process increases the extraction of valuable metals into the sulfuric acid solution without any effort, including the use of expensive autoclave equipment. The results obtained for the extraction of%: vanadium - 84.3 - 85.6, molybdenum - 95.4 - 88.1, zinc - 69.6 - 71.1, uranium - 60.3 - 64.9 and the amount of REE - 66 , 2 - 49.2, respectively, with an acid consumption of 150 g / l. However, a further increase in the consumption of sulfuric acid above 150 g / l does not lead to an increase in recovery.

It should, be noted that the obtained metal-bearing solutions were investigated by the selective separation of valuable metals by the sorption method and more than 70% separation were obtained.

For the extraction of metals from productive solutions of leaching ash from oil shale deposits of Aktau and Sangruntau, the following ion-exchange resins of the Anglia company PUROLITE were used: for vanadium grade A-109 extraction 79 - 81%, for molybdenum - grade A-100 extraction was more than 84 - 86% , uranium grade A-560 extraction 81 - 79% and for zinc S-984 extraction 75 - 71%.

Carried out precipitation to extract REE and received 18% content of the amount of REE.

Main conclusions
- determined the chemical composition of ash from oil shale deposits Aktau and Sangruntau, analyzed the samples on a mass spectrometric device ICP-MS;
- adding sodium chlorine to the oxidative roasting process, which shows the reaction of valuable metals (vanadium, molybdenum, etc.) in the ash of oil shale and facilitates the leaching process with the extraction of valuable metals into a sulfuric acid solution and selective extraction with ion exchange sorbents;
- ash from oil shale deposits Aktau and Sangruntau may well be used as a ready-made useful material for the extraction of valuable metals (vanadium, molybdenum, uranium, zinc and REE). In addition, the recommended scheme provides for the disposal of residues (cakes) on road surfaces and cement production.

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