

# Results of Sulfuric Acid Leaching of Ash from Oil Shale from Aktau Deposit

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## ABSTRACT

Hydrometallurgical methods are aimed at leaching to extract valuable components into a sulfuric acid solution, as well as selective extraction on ion exchange resins by sorption of valuable components such as vanadium oxide and molybdenum 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 chloride to the heat treatment process (pyrolysis), the resulting ash is leached in vats with sulfuric acid 130 g / l, at room temperature, S: W = 1: 3, 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 more than 60-70%: molybdenum and vanadium. It should be noted that with an increase in the consumption of sulfuric acid over 130 g/l, the concentration of metals in the productive solution does not increase. For the extraction of metals from the productive solutions of leaching ash from oil shale in the Aktau deposit, the following ion-exchange resins of the England Company PUROLITE were used: for the extraction of molybdenum - grade A-100 and vanadium grade A-109.

**KEYWORD:** extractant 1, hydrometallurgy 2, leaching 3, molybdenum 4, oil shale 5, sulfuric acid 6, vanadium 7

In recent years, much attention has been paid to the development of technology for oil shale [1-5]. According to 1999 data, the explored reserves of oil shale in the Republic of Uzbekistan are enormous and are estimated at 47 billion tons [6].

It should be noted that a number of Uzbek scientists have carried out research on the extraction of metals from oil shale by various chemical methods [7-11].

Today, about 20% of world production uses hydrometallurgical extraction of metals [12]. Hydrometallurgical research includes leaching (transfer of metals into solution with various alkalis and acids), sorption (transfer of metals from solutions to ion-exchange resins), desorption (extraction of metals from ion-exchange resins into solution), extraction (selective extraction of metals from a saturated solution to an extract ant).

Earlier, the State Enterprise "Institute of Mineral Resources" conducted laboratory research on the complex extraction of metals from ash of oil shale deposit of Aktau. The highest rates for the extraction of metals into solution were obtained during autoclave leaching (leaching under pressure in special expensive equipment) over 70% of Cu, Zn, Ni, Mo passed into the collective solution.

Currently, there are no proven technologies for cost-effective processing of such a "bulk" concentrate with low contents of valuable components.

**How to cite this paper:** Almatov Ilhomjon Mirzabek Ogli "Results of Sulfuric Acid Leaching of Ash from Oil Shale from Aktau Deposit" Published in International Journal of Trend in Scientific Research and Development (ijtsrd), ISSN: 2456-6470, Volume-5 | Issue-1, December 2020, pp.1187-1190, URL: www.ijtsrd.com/papers/ijtsrd38091.pdf



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In this regard, the task of developing technical regulations for the selective extraction of the most significant metals (V and Mo) in terms of content and value becomes urgent.

**The aim of this work** is the determination of the optimal parameters for extracting molybdenum by vat leaching (leaching in special acid-resistant vats) with sulfuric acid from the ash of oil shale from the Aktau deposit.

**Methods:** the research method of this work is hydrometallurgical beneficiation:

- to transfer valuable leaching components using sulfuric acid;
- for the selective extraction of molybdenum and vanadium from the metal-bearing solution, sorption on ion-exchange resins.

**The scientific novelty** of the conducted research studies is as follows:

- for the first time, a technology has been developed for the integrated processing of oil shale ash using hydrometallurgical methods that allow the extraction of molybdenum and vanadium;
- the developed technology for the integrated processing of oil shale ash is characterized by the use of local cheap acids and alkalis, which in the future will increase the economic efficiency of the integrated processing of oil shale ash from the Aktau field;
- the developed scheme for processing oil shale ash allows to reduce the cost of the resulting oil product.

**Conclusion:** chemical analysis of ash from oil shale from the Aktau deposit is presented in Table 1.

**Table 1 Chemical analysis results ash of oil shale from Aktau deposit**

The elements	Content, %	The elements	Content, %
SiO <sub>2</sub>	62,98	K <sub>2</sub> O	1,42
Fe <sub>2</sub> O <sub>3</sub> general	5,64	P <sub>2</sub> O <sub>5</sub>	1,00
TiO <sub>2</sub>	0,62	Sgeneral	2,62
MnO	0,08	WLI	23,47
Al <sub>2</sub> O <sub>3</sub>	14,9	Amount	101,86
CaO	7,76	CO <sub>2</sub>	0,35
MgO	3,02	H <sub>2</sub> O	0,44
Na <sub>2</sub> O	0,97		

As can be seen from Table 1. the main content is SiO<sub>2</sub> - 62.98% and Al<sub>2</sub>O<sub>3</sub> - 14.9%, which means that the resulting ash is in a silicate form.

Note: weight loss on ignition

The semi-coke obtained at the pyrolysis stage, according to the classical scheme, must 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, and we get ash.

To determine the chemical composition, ashes were used, which are finely ground uniformly granular powders. Samples weighing 3 g were analyzed using 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	Aktau		The elements	Clark in the earth's crust	Aktau	
		Initial	Ash			Initial	Ash
Li	32,0	23,0	26,4	Sb	0,5	7,2	0,8
Be	3,8	1,4	1,89	Te	0,001	0,36	0,18
Na	25 000	7800	12 800	Ba	650	860,0	831,0
Mg	18 700	17000	14 800	La	29,0	21,0	34,9
Al	80 500	63000	66 600	Ce	70,0	32,0	56,9
K	25 000	13000	15 400	Pr	9,0	3,7	9,4
Ca	29 600	66000	47 600	Nd	37,0	16,0	33,6
Sc	10,0	12,0	11,6	Sm	8,0	4,7	4,6
V	90,0	880,0	915,0	Eu	1,3	1,6	1,27
Cr	83,0	95,0	178,0	Gd	8,0	3,4	6,29
Mn	1 000	610,0	528,0	Tb	4,3	0,68	0,784
Fe	46 500	33000	35 200	Dy	5,0	3,6	5,07
Co	18,0	13,0	13,2	Ho	1,7	0,89	1,4
Ni	58,0	220,0	131,0	Er	3,3	2,6	2,49
Cu	47,0	77,0	79,8	Tm	0,27	0,4	0,341
Zn	83,0	130	149	Yb	0,33	3,1	3,16
As	1,7	7,3	0,2	Lu	0,8	0,27	0,291
Se	0,05	8,7	9,3	ΣREE	208	131,9	203,2
Sr	340	320,0	361,0	W	1,3	1,5	3,05
Y	20,0	26,0	31,1	Tl	1,0	12,0	33,1
Nb	20,0	7,3	7,18	Pb	16,0	14,0	11,0
Mo	1,1	360,0	397,0	Th	13,0	8,6	4,5
Cd	0,13	21,0	5,94	U	2,5	54,0	52,0
Sn	2,5	2,5	0,88				

Based on the data in Table 2, in the ash of oil shale, a product of oxidative roasting at a temperature of 850°C, the content of valuable metals in the Aktau deposit increases markedly compared to the original ore, ppm: vanadium from 880 to 915, molybdenum from 360 to 397, the amount of rare elements from 131.9 to 203.2, and thallium from 12 to 33.1.

To optimize the degree of extraction of molybdenum and vanadium into the solution in vat leaching with sulfuric acid, the influence of the size class of ash grinding and the ratio S: L (solid: liquid) of the degree of extraction was determined.

#### Ore size class

The following size classes were investigated: original ash; -1; -0.5 and -0.315 at leaching conditions S:L= 1:3, H<sub>2</sub>SO<sub>4</sub> - 15%, leaching time - 60 min, room temperature.

Extraction of Mo and V into solution at different particle sizes

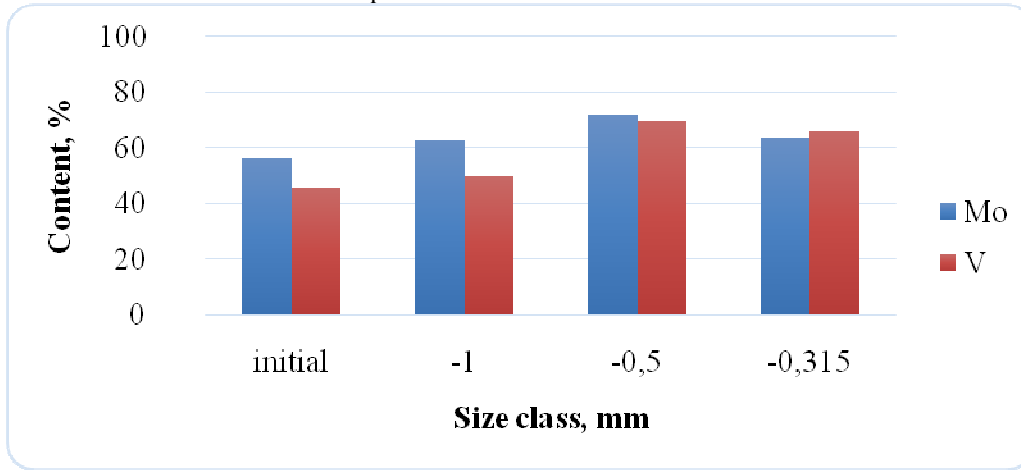


Fig.1.

Fig. 1.The results showed that the degree of ash grinding in the studied classes affects the extraction of molybdenum and vanadium into the sulfuric acid solution; it should be noted that the difference in the increase is around 15%. The degree of extraction into the solution is in%: initial 56.36, -1mm - 62.72, -0.5mm - 71.81 and -0.315mm 63.63.

**S: L ratio (solid: liquid)**

To determine the influence of S:L on the degree of extraction of metals into the solution, leaching was carried out at different S:L and sulfuric acid concentration. Leaching conditions: size class -0.5 mm, time - 60 min, room temperature.

Extraction of Mo and V into solution

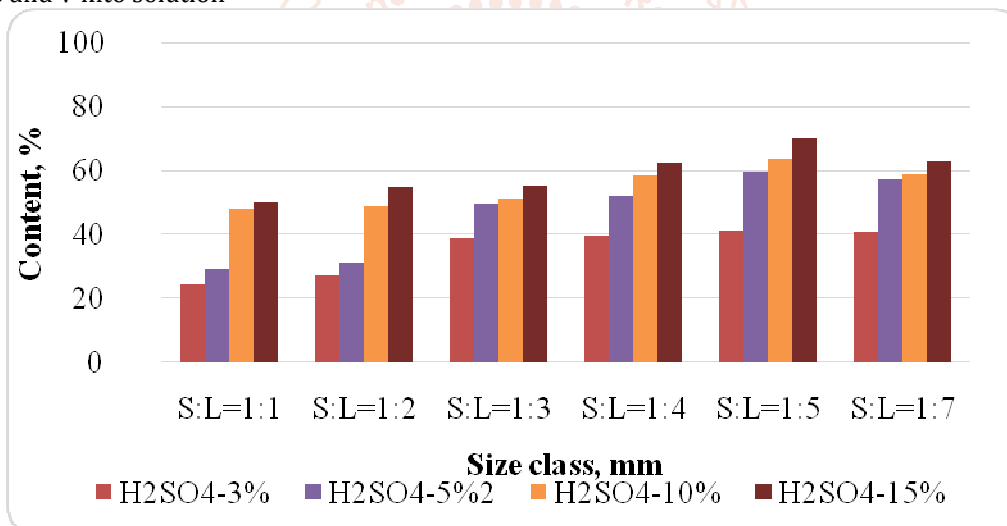


Fig.2.

Extraction of Mo and V into solution

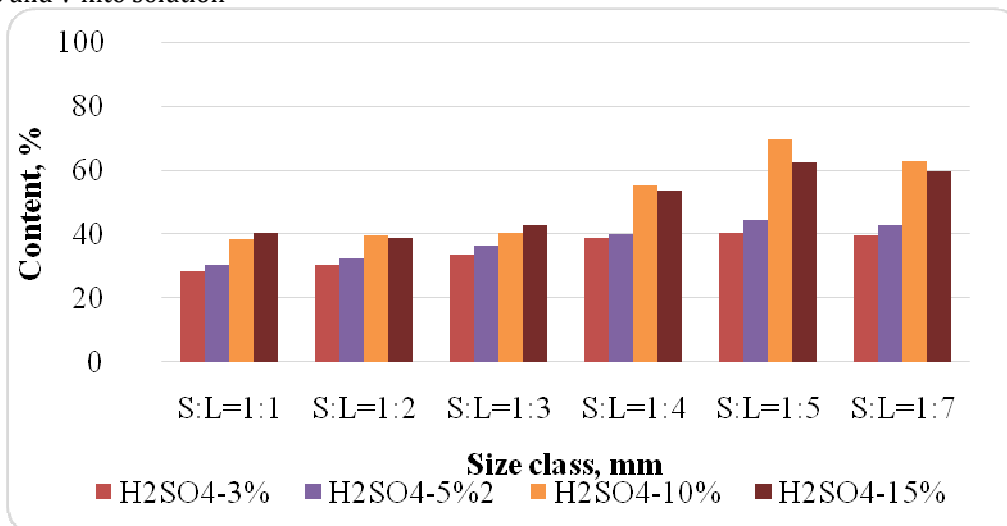


Fig.3.

Fig. 2.-3. With an increase in the liquid phase to the solid, the extraction of molybdenum and vanadium into solution increases. It should be noted that, in general, the increase in the extraction of molybdenum and vanadium occurs up to S:L= 1:3 (more than 60%), a further increase in the liquid phase does not affect the extraction of molybdenum and vanadium.

The studies carried out in general have shown that in vat leaching of ash from oil shale from the Aktau deposit with a solution of sulfuric acid, up to 60-70% of molybdenum and vanadium, respectively, are extracted into the solution.

According to the results of the study in laboratory conditions, the optimal parameters of vat leaching with a solution of sulfuric acid of ash from combustible shale's from the Aktau deposit were determined, which were: sulfuric acid concentration - 130 g/l, ore size class - 0.5 mm, time - 60 min, temperature - room temperature and ratio S:L (solid:liquid) = 1:3, preliminary sintering of ash with sodium chloride.

To extract useful components from a metalliferous solution of sulfuric acid solution, ion-exchange resins of the England Company PUROLITE were used: for the extraction of molybdenum - grade A-100 and vanadium grade A-109, the extraction is more than 90%.

#### Because of the research, the following conclusions were formulated:

- determined the parameters for the transfer of valuable components into a metalliferous sulfuric acid solution;
- the parameters of the extraction of molybdenum and vanadium by the method of sorption on ion-exchange resins have been determined;
- ash from oil shale from the Aktau deposit may well be used as a ready-made useful material for extracting valuable components (vanadium and molybdenum);
- Also in the developed scheme of ash processing technology obtained from oil shale from the Aktau deposit, it is envisaged to utilize residues (cakes) for their use as cement production and road surfaces.

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