При подборе катиона-индикатора и растворителя была проведена серия экспрессных опытов, заключающаяся в фотографировании виалы с разделившимися фазами на белом фоне. Дрейф цветности контролировали по образцовому шаблону.

Индикаторы выбирали таким образом, чтобы интенсивность окраски органической фазы содержащего аналита была выше, чем у фона. Наиболее удовлетворительным результатам соответствовали Аурамин, Акридиновый желтый, Фуксин кислый с 1,2-дихлорэтаном как растворителем.

Отобранные индикаторы и растворитель были использованы для создания методики количественного анализа. В рамках данной методики была приготовлена серия растворов с известным количеством аналита и сняты спектры. По полученным данным построена градуировочная зависимость и определены метрологические характеристики.

LEACHING OF VANADIUM FROM THE SECOUND STAGE DUPLEX PROCESS CONVERTER SLAG

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Usually hydrometallurgical recycling of «poor» (<5 wt. %) vanadium slags, ashes of TPP, brown coal, oil sludge supposes acidic leaching. In the process of acidic leaching a lot of impurities of metals go into solution together with vanadium. So the present work is devoted to the study of processes of the selective leaching vanadium from slag and precipitation of vanadium compounds from the acidic leaching solution.

Vanadium is produced by blast melting of titanomagnetites. The first stage of this process is producing of vanadium cast iron. The cast iron is blown in the converter and get steel and vanadium slag containing up to 30 wt.% of vanadium pentoxide. This slag use for manufacturing of pure vanadium pentoxide with one of the well-known technology [1]. After the second purge blast iron in converter with adding of limestone is formed steel and «poor» vanadium slag. Now days the «poor» vanadium slag pollute the environment and there are no methods for it recycling.

Slag is the multiphase and multicomponent system, consist of the composite oxides of Ca, Mg, Ti, Fe, Al, Si. From the ratio of peak intensity on diffractogram, measured by the Bruker D8 Advance, follows that slag consist of phases rel. %: 22,12 – $Fe_{2,95}O_4$; 21,72 – $Ca_2((Fe_{1,724}Al_{0,276})O_5)$; 16,51 – $Ca_2(SiO_4)$; 14,66 – $CaMg_2Fe_{16}O_{27}$; 13,82 – $Ca_{14}Mg_2(SiO_4)_8$; 11,15 – $Mg(Mg_{0,926}Ti_{0,074})(Mg_{0,074}Ti_{0,926})O_4$. The method of X-Ray diffraction can’t determine the vanadium because it concentration is 1,87 wt.% and it’s distributed inside the phases in various oxidation states +3,+4,+5. Existence in the slag vanadium in oxidation states +3, +4 and calcium (29,5 wt.%) determine our choice the sulfuric acid instead of the alkali and soda for leaching of vanadium. The best rate of leaching 98,15 % were achieved under the follow conditions: 1:15 of liquid-solid ratio, temperature 298 K, 55 g/L sulfuric acid, 8 g/L hydrogen peroxide, 700 r/min stirring velocity, 150 min leaching time. The interesting feature of the leaching of vanadium with sulfuric acid and hydrogen peroxide is decrease rate of leaching with increase of temperature. Compositions of slag and solution after leaching are shown in table 1.

Compositions of slag and solution after leaching

Element	V	Mn	Fe	Mg	Ca	Al	S	Cr	Si	Ti	pH
«poor» vanadium slag, wt. %	1,87	1,5	26,5	7,1	29,5	1,34	0,10	0,23	4,74	0,8	-
Leaching solution, g/L	1,12	0,2	3,73	1,23	0,47	0,48	1,23	0,07	0,17	0,07	2,56

Precipitation of vanadium from solution was performed by thermohydrolysis at pH 1,6 and 2,6, at a temperature 363 K. The sediment obtained at pH 1,6 contained a mixture of vanadates and vanadium acid. After filtration, washing and annealing at 873K was obtained the vanadium containing product of the following phase composition wt. %: V_2O_5 – 46,9; $CaSO_4$ - 21,5; TiO_2 – 22,8; $Fe_2(V_4O_{13})$ – 8,8. Rate of deposition at the initial concentration of vanadium 2 g/L was 80,0 wt.%. At pH=2,6 the sediment was the mixture of vanadates. After filtration, washing and annealing at 873K was obtained the vanadium containing product containing predominantly: $CaMgV_2O_7$, $Ca_2V_2O_7$, rate of deposition was 98,21 wt.%.

Both vanadium containing product can be used for obtaining pure vanadium pentoxide by recrystallization in alkaline medium [2].

Analysis of phase composition of slag was made on the equipments of CCU «Ural-M» IMET UB RAS

1. Khalezov B.D., Vatolin H.A., et al., Processing of manganous vanadium slag converter, EPD UB RAS (2016).
2. Mizin V.G., Rabinovich E.M., et al., Complex Processing of Vanadium Raw Materials: Metallurgy, UrO RAN, (2005).