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RESEARCH PAPER

Application of the surface response methodology (RSM) to optimise alkaline leaching of vanadium and molybdenum from man-made raw materials

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ABSTRACT

The growing demand for strategically important metals, coupled with the depletion of high-quality ores, has highlighted the potential of man-made waste as a secondary source of vanadium and molybdenum. This study investigates the alkaline leaching of technogenic vanadium-containing waste (filter cake) using sodium hydroxide (NaOH) and sodium hypochlorite (NaOCl) as an oxidiser. Chemical and X-ray fluorescence analyses confirmed significant contents of vanadium (3.44%), molybdenum (0.75%), and other valuable metals, indicating the feasibility of complex metal recovery. An experimental design based on the response surface methodology (RSM) and a central composite plan was employed to evaluate the effects of leaching time, reagent concentration, pH, and temperature on metal extraction. Quadratic regression models were constructed and validated using analysis of variance (ANOVA), demonstrating high adequacy (vanadium: $F = 9.55$, $p < 0.001$; molybdenum: $F = 9.84$, $p < 0.01$). Under the identified optimal conditions, vanadium and molybdenum extraction efficiencies reached 88–89% and 80–82%, respectively, and further increased to 89–93% and 82–83%, respectively, following the addition of NaOCl. X-ray phase analysis revealed the formation of stable aluminium and nickel oxide phases, which partially limited extraction and explained deviations from predicted values. The results demonstrate that combining alkaline leaching with an oxidiser and statistical modelling enables effective optimisation of multicomponent waste processing, enhancing metal recovery and reducing environmental impacts, thereby providing a basis for resource-efficient, environmentally friendly metallurgical technologies in Kazakhstan.

Keywords: vanadium; molybdenum; man-made waste; leaching; hydrometallurgy; analysis of variance (ANOVA)

INTRODUCTION

In the context of the modern development of non-ferrous metallurgy, there is a steady increase in interest in processing man-made waste as alternative sources of strategically important metals such as vanadium and molybdenum [1, 3]. The depletion of high-grade ore deposits and the increasing complexity of mining and geological conditions are driving the development of energy-efficient and environmentally sustainable technologies for the integrated processing of mineral raw materials. [4, 5].

In developed industrial countries, up to 70-80% of industrial waste is recycled [6, 7]. For Kazakhstan, whose economy is heavily dependent on mineral extraction and has significant mining potential, the efficient disposal of industrial waste is becoming a critically important task [8]. The low use of man-made raw materials in the country is due to a shortage of modern technologies and equipment for processing various types of waste [9, 10]. At the same time, products obtained from industrial waste are often cheaper than those produced from primary ores. Man-made landfills have a significant advantage: they are already ready for processing, since the raw materials have been extracted from the ground, cleaned and prepared. The processing of man-made raw materials is a priority of Kazakhstan's industrial and environmental policy. According to experts, the potential of the country's secondary mineral resources amounts to millions of tonnes of slags, clinkers, sludge and tailings accumulated at existing and mothballed industrial sites. In national strategic documents, the processing of man-made resources is considered as a key tool for rational subsurface use, aimed at reducing dependence on the extraction of primary ores, increasing the added value of products and minimising environmental risks associated with long-term waste storage. This approach is consistent with the goals of sustainable development and the "Green Economy" of Kazakhstan, suggesting the introduction of closed production cycles and the integrated use of mineral raw materials.

Man-made waste from metallurgical industries, including sludge, enrichment tailings and ash and slag materials, contains significant amounts of valuable elements, but their processing is difficult due to their complex phase and chemical composition [11]. In particular, vanadium and molybdenum are often present in refractory compounds that require modified hydrometallurgical methods, such as alkaline leaching [12].

Alkaline leaching has proven to be an effective method for selectively extracting vanadium and molybdenum from complex multicomponent systems [9, 10]. The kinetics of the process depend on several key factors, namely: temperature, reagent concentration, processing time, degree of material grinding, and the mineralogical composition of the raw material. Second-order polynomial models (quadratic approximations) are widely applied for predicting and optimising process parameters, as they account for both the main effects of individual variables and their interaction effects [13].

Alkaline leaching extracts molybdenum (Mo) and vanadium (V) from man-made raw materials such as spent hydrotreating catalysts or boiler ash by converting them into soluble alkaline compounds. The process usually involves firing the material to oxidise low-valent compounds, followed by leaching with an alkali, usually sodium hydroxide (NaOH) or sodium carbonate (Na_2CO_3), to dissolve Mo and V. Subsequent separation is achieved by selective precipitation, solvent extraction using extractants such as Aliquat 336, or ion exchange, with controlled firing and careful pH adjustment crucial to achieve optimal results.

Among the various models used to process mineral and man-made raw materials, the quadratic model holds a special place. It is used in the context of planning experiments such as Box-Behnken, central compositional designs, etc., allowing one to account not only for main effects but also their interactions and quadratic dependencies [14]. Nevertheless, building a model requires verification of its adequacy, as the presence of statistically insignificant factors or deviations from the assumptions of regression analysis can yield unreliable results [15].

The quadratic model is a statistical tool, namely a second-order polynomial equation, used to predict the leaching efficiency of vanadium and molybdenum in alkaline solutions by correlating leaching parameters (such as temperature, time, and reagent concentration) with the leaching yield. It helps optimise these parameters by identifying their linear, quadratic, and interactive effects on the leaching process, enabling prediction of optimal conditions for maximum leaching efficiency [16]. Evaluating the adequacy of the quadratic model for predicting the rate of alkaline leaching of vanadium and molybdenum from man-made raw materials is an important stage of statistical analysis, as it allows us to determine how well the model describes the experimental data and is suitable for forecasting. Kazakhstan has a well-developed research and production base for developing and implementing technologies for processing complex, multicomponent man-made materials. Extensive expertise has been developed in the extraction of non-ferrous, rare, and precious metals from industrial waste streams, including vanadium and molybdenum, through operations at enterprises in the Karaganda, East Kazakhstan, and Pavlodar regions. The integration of modern techniques, including oxidant-assisted alkaline leaching, microwave and ultrasonic activation, and statistical modelling of process parameters (e.g., quadratic regression models), provides a basis for increasing the extraction efficiency of valuable metals, lowering production costs, and enhancing the environmental performance of processing operations [17, 18].

Assessing the adequacy of such models is a key step in ensuring the reliability of forecasting, since an insufficiently accurate model can lead to erroneous technological solutions and economic losses [19]. Statistical methods are employed for this purpose, including analysis of variance (ANOVA), determination coefficients, and significance tests for both the regression equation and individual factors [20, 21, 22].

This study aims to construct and statistically validate a second-order (quadratic) regression model that characterises the effects of process parameters on the alkaline leaching rate of vanadium and molybdenum, and to evaluate the significance and reliability of the resulting regression coefficients.

MATERIAL AND METHODS

Description of samples and equipment

The object of research is a technogenic waste of vanadium production – a filter cake. The results of chemical and X-ray fluorescence analysis show that the filter cake contains significant amounts of aluminium (42.05%), calcium (0.20%), silicon (0.46%), vanadium (more than 3.4%), and oxygen, as presented in **Table 1**.

Table 1 Chemical composition of artificial vanadium production waste

The content of the elements, %											
Na	Al	Si	P	S	K	Ca	V	Fe	Ni	Mo	O
3.12	42.05	0.46	0.42	0.46	0.06	0.20	3.44	0.60	8.82	0.75	39.62

Table 1 shows the chemical composition of man-made waste generated during vanadium production. The main component of the waste is aluminium (Al), which contains 42.05%. The vanadium(V) content is 3.44%, which confirms the prospects of these wastes as secondary raw materials for the extraction of this valuable metal. Iron (Fe) is also present in the sample in an amount of 0.6%, nickel (Ni) – 8.82%, and molybdenum (Mo) – 0.75%, which makes possible the complex extraction of several valuable components.

According to X-ray phase analysis studies (**Fig. 1**), the sample includes minerals such as corundum (Al_2O_3, 32.3%), spinel ($MgAl_2O_4$, 16.2%), γ-aluminum oxide (γ-Al_2O_3, 13.9%), nickel silicate (Ni_2SiO_4, 11.5%), iron silicon vanadium phase (Fe_2SiV, 7.4%), sodium vanadium oxides β-$Na_{0.33}V_2O_5$ (7.1%) and α-NaV_2O_5 (6.0%), as well as ε-Fe_2O_3 (5.5%). Vanadium in the sample is predominantly present in the form of vanadium bronze phases.

Sodium vanadium bronze ($Na_{0.33}V_2O_5$ and $Na_xV_2O_5$) present in the original sample, only partially pass into the solution by leaching with aqueous solutions of NaOH. To increase the completeness of vanadium extraction, the NaOCl oxidizer was introduced into the leaching solution, which ensures the oxidation of V^{4+} to V^{5+} and, as a result, an increase in the proportion of vanadium in the soluble form. During the leaching experiments, technical sodium hydroxide (NaOH), flaked (base substance content ≥ 98.5%), and grade A sodium hypochlorite (NaOCl) (active chlorine content ≥ 190 g/l) were used as reagents.

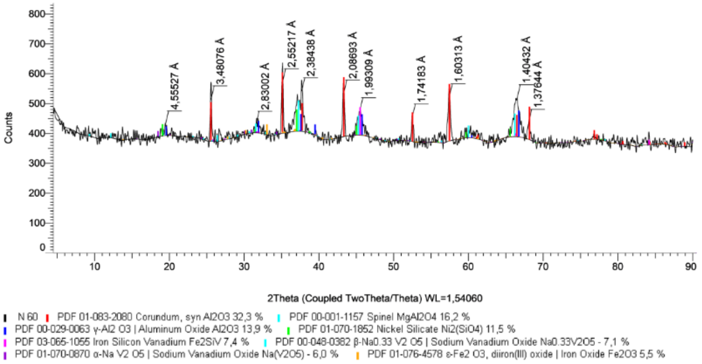


Fig. 1 - X-rays of the initial industrial product of vanadium production

The physico-chemical composition of the feedstock and processed products was determined using a set of analytical methods. X-ray fluorescence analysis (XFA) was performed on Venus 200 spectrometers (Malvern PANalytical BV, the Netherlands) and SRV-1B (Kazakhstan). The chemical composition was determined using an Optima 2000 DV inductively coupled plasma optical emission spectrometer. The vanadium and molybdenum contents were determined by titrimetric and photometric methods. X-ray diffraction analysis (XDA) was performed on a Bruker D8 ADVANCE diffractometer using copper Ka radiation at a voltage of 36 kV and a current of 25 mA.

Chemical reactions occurring during the leaching of a sample of man-made material

According to phase analysis, the compounds Al_2O_3, γ-Al_2O_3, $MgAl_2O_4$, Ni_2SiO_4, and ε-Fe_2O_3 present in the sample practically do not react under the specified conditions and remain in the cake. Thus, vanadium is present predominantly in the form of sodium vanadium bronzes in the compounds β-$Na_{0.33}V_2O_5$ and α-NaV_2O_5, which dissolve in the presence of alkali according to reactions (1) and (2) with the formation of sodium metavanadate $NaVO_3$:

For α-NaV_2O_5:

$$NaV_2O_5 + NaOH \rightarrow 2NaVO_3 + 0.5H_2O \quad (1)$$

For β-$Na_{0.33}V_2O_5$:

$$Na_{0.33}V_2O_5 + 1.67NaOH \rightarrow 2NaVO_3 + 0.83H_2O \quad (2)$$

However, in the absence of an oxidizing agent, the dissolution of vanadium bronzes is limited, since V^{4+} dissolves less well. In the NaOH + NaOCl system, vanadium is leached according to the following reaction scheme (3):

$$Na_xV_2O_5 + (2-x)NaOH + NaOCl \rightarrow 2NaVO_3 + NaCl + H_2O \quad (3)$$

Molybdenum dissolves well in alkali, forming sodium molybdate according to reaction (4):

$$MoO_3 + 2NaOH \rightarrow Na_2MoO_4 + H_2O \quad (4)$$

Methodology of the response surface (RSM)

Experimental planning (DoE) using the response surface method (RSM) is an effective approach for evaluating and optimising the parameters of hydrometallurgical processes.

The response surface methodology (RSM) is used to study the relationship between several independent variables and one or more response variables [18]. The optimal levels and ranges of values determined based on the temperature regime, the concentration of sodium hydroxide and the pH of the leaching solution as independent variables are presented in **Table 2**.

Table 2 Controlled factors and set levels of variation in DOE

Factors	Symbol	Coding level	
		−1	1
Time (h)	A	4.00	8.00
Concentration NaOH (g/l)	B	0.50	3.00
pH	C	9.00	11.00
Temperature (°C)	D	25.00	85.00

This statistical and mathematical approach allows us to build empirical models describing the dependence of the degree of metal extraction on several technological factors.

To plan the experiment, a central composite plan (CCD) was used, which includes a variation of four factors:

- leaching time (A, 4–8 h),
- reagent concentration (B, 0.5–3.0 g/l),
- pH of the solution (C, 9–11),
- temperature (D, 25–85 °C).

The recovery rates of vanadium (E(V)) and molybdenum (E(Mo)) in percentages were considered as responses (target functions). The experimental data obtained were approximated by a second-order polynomial regression model:

$$y = b_0 + \sum_{l=1}^{k} b_l x_l + \sum_{l=1}^{k} b_u x_l^2 + \sum_{l=1}^{k-1} \sum_{j=l+1}^{k} b_{l_j} x_l x_j \qquad (1.)$$

where y is the calculated value of the degree of metal extraction;
b_0 — free member;
b_i — coefficients for linear terms;
b_{ii} — coefficients for quadratic terms;
b_{ij} — coefficients characterizing the interaction of factors;
k — number of factors studied.

Experimental Method

The working solutions were prepared with concentrations of 0.5% NaOH and 20 g/L NaOCl. The test samples of man-made vanadium production waste were pre-homogenised in a dry grinding laboratory ball mill, LBP-78, for 10 minutes at a drum rotation speed of 68 rpm, a 4:1 ball-to-powder ratio, and a ball diameter of 20 mm.

The leaching experiments were performed by repulping the samples in water at controlled temperatures of 40, 60, and 85°C, with a liquid-to-solid ratio (L:S) of 2:1, in a thermostatically controlled reactor equipped with an OS20-S mechanical agitator operating at a fixed mixing speed.

The leaching time was 4 hours. At the end of leaching, the pulp was filtered on a laboratory press filter; the filtrate was analysed for the target components, and the solid residue was washed with distilled water (tenfold), dried, and calcined. Processing technology is shown in **Fig. 2**.

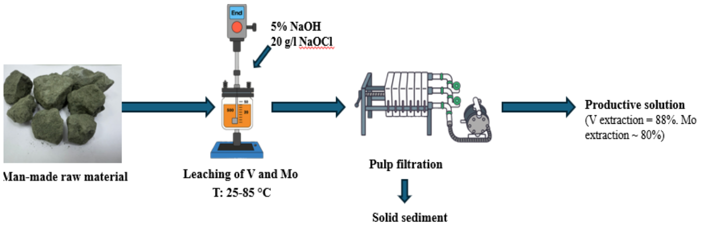


Fig. 2 - Diagram of an experimental facility for leaching man-made waste

RESULTS AND DISCUSSION

Subtitle of results and discussion

The results of the experiments on the extraction of vanadium and molybdenum by the alkaline leaching method in the presence of sodium hypochlorite (CBT) using the surface response methodology (RSM) are summarized in **Table 3**.

Table 3 Data from the experiment plan matrix (DOE)

No.	Concentration	Temperature	pH	V	Mo
1	1.75	55.00	9.00	74.90	79.60
2	2.00	65.00	9.50	53.10	79.70
3	3.00	85.00	10.00	47.00	81.90
4	3.00	85.00	11.00	49.30	81.70
5	1.75	55.00	11.50	88.50	76.90
6	3.00	25.00	11.00	88,60	78.50
7	1.75	55.00	10.00	87.40	79.30
8	0.35	55.00	10.00	88.70	77.90
9	0.50	85.00	9.00	48,30	81.40
10	3.00	25.00	9.00	77.90	78.70
11	1.75	55.00	10.00	74.80	79.50
12	3.85	55.00	10.00	71.50	80.10
13	1.75	55.00	10.00	83.80	79.40
14	0.50	25.00	9.00	89.00	77.00
15	1.75	55.00	11.68	87.90	75.80
16	0.50	25.00	11.00	88.30	76.80
17	1.75	55.00	8.32	83.50	78.40
18	1.75	105.45	10.00	47.10	82.30
19	1.75	4.55	10.00	81.2	79.9
20	0.50	85.00	11.00	80.1	80.2

Table 4-5 shows the results of the dispersion analysis (ANOVA) for the response surface model of the vanadium and molybdenum leaching process from the filter cake.

Table 4 ANOVA for a quadratic model of the vanadium response surface

Source	Sum of squares	df	Mean square	F value	Source	Sum of squares	p-value prob > F	
Model	3139.82	3	1046.61	9.55	0.0008	significant	Model	significant
A-A	418.04	1	418.04	3.81	0.0686		A-A	
B-B	2530.58	1	2530.58	23.08	0.0002		B-B	
C-C	314.56	1	314.56	2.87	0.1097		C-C	
Residual	1754.25	16	109.64				Residual	
Lack of fit	1670.01	14	119.29	0.2914	0.2914	not significant	Lack of fit	not significant

According to the analysis of variance (ANOVA), the calculated F-value for vanadium is 9.55 (Table 4), indicating that the regression model is statistically significant. The probability of obtaining such an F-statistic due to random variation does not exceed 0.11%, which is substantially lower than the conventional significance level of α = 0.05. Therefore, the null hypothesis that the factors have no effect is rejected, confirming the overall adequacy and reliability of the model. The corresponding p-value of 0.0011 further supports the statistical significance of the model. As generally accepted, p-values below 0.05 indicate that the effects of the considered factors are statistically significant. According to the ANOVA data presented in Table 5, the greatest contribution to the formation of the response is made by factor B (temperature) (p=0.0002p = 0.0002p=0.0002) and, to a lesser extent, A (concentration) (p=0.0686p = 0.0686p=0.0686, the boundary value for the significance level 0.05–0.1). The factor C (pH) (p=0.097p=0.097p=0.097) is statistically insignificant at a confidence level of 95%.

Thus, when simplifying the regression equation by eliminating statistically insignificant terms, the following expression is obtained:

$$E(V) = 74.545 - 8.252A - 22.839B \qquad (2.)$$

This simplification of the model enhances its interpretability while preserving a high level of explanatory power for the experimental dataset.

Table 5 below provides an ANOVA for a quadratic model of the molybdenum response surface.

Table 5 ANOVA for a quadratic model of the molybdenum response surface

Source	Sum of squares	df	Mean square	F value	p-value prob > F	
Model	55.09	9	6.12	9.84	0.0007	significant
A-A	4.71	1	4.71	7.58	0.0204	
B-B	22.15	1	22.15	35.62	0.0001	
C-C	3.04	1	3.04	4.89	0.0514	
AB	0.37	1	0.37	0.60	0.4564	
AC	0.24	1	0.24	0.39	0.5466	
BC	0.02	1	0.02	0.03	0.8780	
A^2	8.97E-003	1	8.97E-003	0.01	0.9068	
B^2	6.80	1	6.80	10.94	0.0079	
C^2	6.18	1	6.18	9.93	0.0103	
Residual	6.22	10	0.62			
Lack of fit	6.20	8	0.77	77.47	0.0128	significant
Pure error	0.02	2	0.01			
Cor Total	61.31	19				

According to the analysis of variance (ANOVA) results presented in Tables 5 and 6, the calculated F-value for molybdenum is 9.84 ($p = 0.010$), indicating that the regression model is statistically significant overall. The probability of obtaining such an F-statistic due to random variation does not exceed 1.0%, which is well below the conventional significance level of $\alpha = 0.05$. Therefore, the null hypothesis that the studied factors have no effect on molybdenum recovery is rejected, confirming the adequacy and reliability of the developed model.

From the initial experimental data (**Table 3**), with a linear model without taking into account interactions and quadratic terms, the following regression equation in encoded variables is obtained:

$$E\ (Mo) = 79.138 + 0.152A + 1.120B + 0.348C \qquad (3.)$$

where:

- A — concentration (coded),
- B — temperature (coded),
- C — pH (coded).

A review of the p-values obtained from the ANOVA results showed that the significant factors having a statistically significant effect on molybdenum extraction at a confidence level of 95% are temperature (B) and, to a lesser extent, pH (C), while the effect of concentration (A) turned out to be statistically insignificant.

Taking into account the exclusion of statistically insignificant terms, a simplified regression equation is obtained:

$$E\ (Mo) = 79.138 + 1.120B + 0.348C \qquad (4.)$$

This equation demonstrates strong predictive capability for molybdenum extraction over the investigated technological parameter interval. A comparative analysis of the obtained models for vanadium and molybdenum showed that the temperature factor (B) is decisive in both cases; however, the pH contribution is more pronounced for molybdenum.

A set of diagnostic graphs was used to improve the accuracy of assessing the adequacy of the quadratic model when describing experimental data.

Fig. 3 shows the diagnostic dependencies for the extraction of vanadium (V).

Figure 3 presents an assessment of the model adequacy for vanadium based on diagnostic plots. The normal probability plot of the internally studentized residuals (a) demonstrates their close alignment with a straight line, confirming the assumption of normality and the absence of systematic deviations. The plot of residuals versus predicted values (b) shows a random and uniform scatter of points around the zero line, indicating that the residuals are independent of the fitted responses and supporting the validity of the model. The residuals plotted against the experimental run order (c) exhibit a random distribution without any discernible trend, suggesting the absence of systematic error associated with the sequence of experiments. Comparison of the predicted and experimentally observed response values (d) demonstrates their close line fit, confirming the high accuracy of the model prediction and the agreement between the calculated and empirical data.

Next, **Fig. 4** shows the diagnostic dependencies for molybdenum (Mo) extraction.

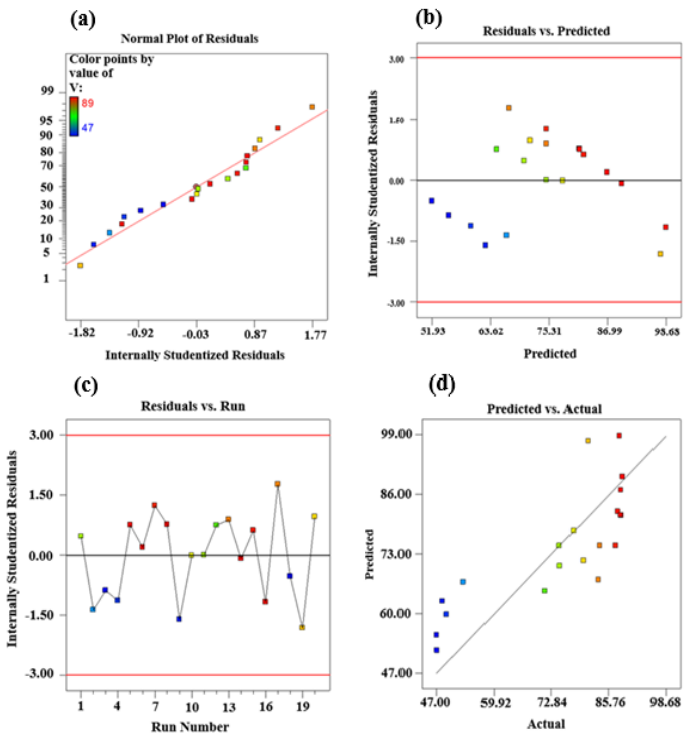


Fig. 3 The analysis of the variances of the adequacy of the vanadium model was carried out by constructing diagnostic graphs: (a) normal probabilistic dependence of internal studentised residuals; (b) distribution of internal studentised residuals relative to predicted responses; (c) dependence of the internal studentised residues on the sequence of experiments; (d) comparison of calculated and actual response values

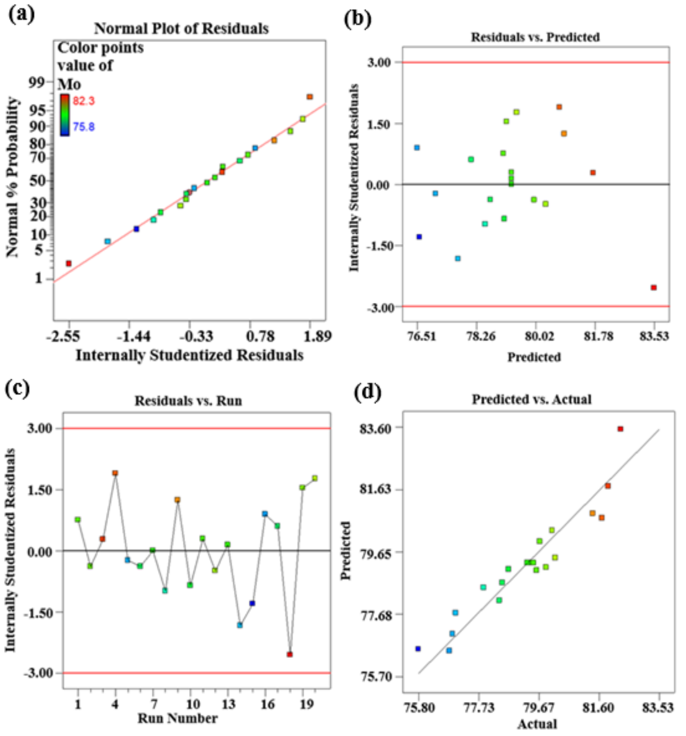


Fig. 4 - The analysis of model adequacy variances for molybdenum was carried out by constructing diagnostic graphs: (a) normal probabilistic dependence of

internal studentised residuals; (b) distribution of internal studentised residuals relative to predicted responses; (c) dependence of the internal studentised residues on the sequence of experiments; (d) comparison of calculated and actual response values

Fig. 4 shows the results of the model adequacy variance analysis for molybdenum based on the diagnostic graphs constructed. On the normal probability dependence of the residuals (a), the points are located along a straight line, which confirms that the distribution corresponds to the normal law and the model is correct. The distribution of the residuals relative to the predicted values (b) shows that all data are within acceptable limits. The dependence of the residuals on the sequence of experiments (c) is characterised by a random distribution of points around the zero line, confirming the independence of errors and the absence of systematic bias.

A comparison between the predicted and observed response values (d) demonstrates their close alignment with the line of perfect agreement, indicating the high predictive accuracy of the model and strong concordance between the calculated and experimental data.

The resulting parameter interactions, which significantly affected the responses, were then used to construct three-dimensional (3D) graphs (**Figs 4 and 5**) for a more accurate assessment of the parameters involved. All fixed parameters were based on the average value.

Fig. 5 shows 3D surfaces and contour diagrams illustrating the effects of reagent concentration (A), temperature (B), and medium pH (C) on the degree of vanadium recovery.

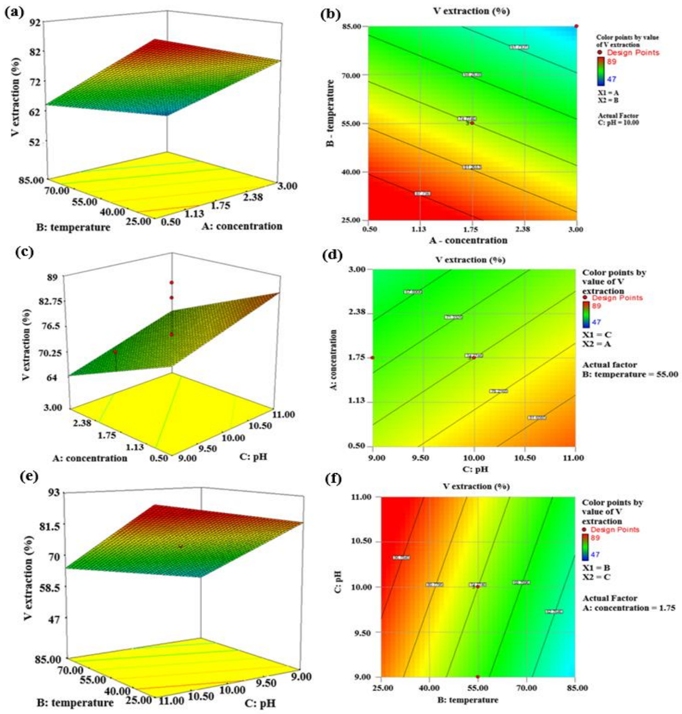


Fig. 5 – Three-dimensional and contour graph of the variable interaction of leaching parameters V; a), b) the interaction between temperature and alkali concentration, c), d) the interaction between the concentration of alkali and the pH of the solution, e), f) the interaction between time temperature and pH of the solution

It can be seen from **Figs 4a) and 4b)** that increasing concentration increases extraction, whereas increasing temperature has the opposite effect, reducing it. **Figs 4c) and 4d)** show that the combination of a high concentration and a more alkaline medium (pH 9-11) contributes to an increase in the degree of vanadium extraction. Graphs (e, f) show the combined effect of temperature and pH: at low temperatures and high pH, the best results are achieved. The maximum recovery values (89-93%) are observed in regions of high pH, at concentrations near 3.0 and at relatively low temperatures (25-40°C). The experimental points are in good agreement with the calculated surfaces, which confirms the reliability of the model. Thus, the main factors determining the process efficiency are the reagent concentration and the medium pH, while temperature has a negative effect. Optimising these parameters enables achieving a high degree of vanadium recovery.

Fig. 6 shows three-dimensional response surfaces and contour diagrams showing the effect of concentration (A), temperature (B), and pH (C) on the degree of molybdenum recovery.

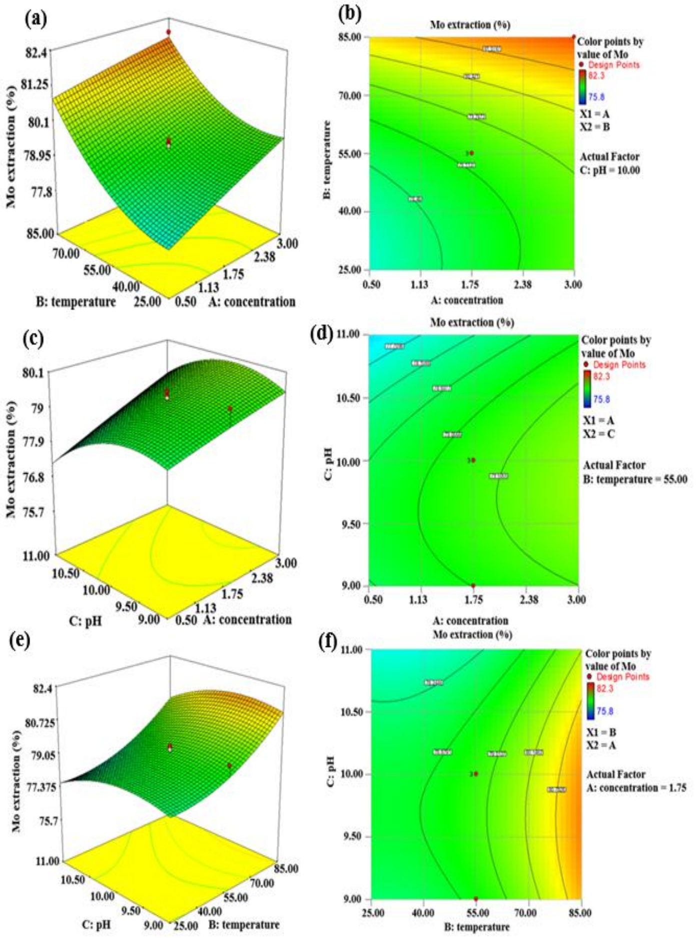


Fig. 6 – Three-dimensional and contour graph of the variable interaction of Mo leaching parameters; a), b) the interaction between temperature and alkali concentration, c), d) the interaction between the concentration of alkali and the pH of the solution, e), f) the interaction between time temperature and pH of the solution

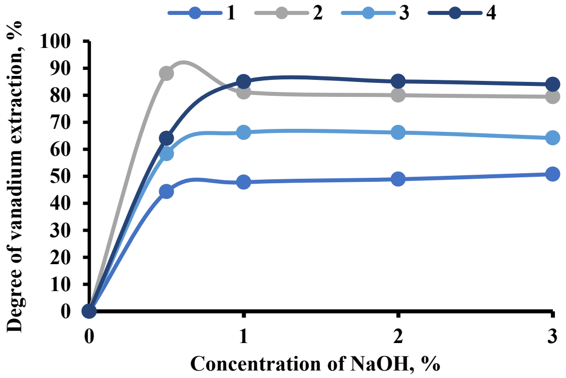
Analysis of **Figs 6a) and 6b)** indicates that a simultaneous increase in temperature and concentration at pH 10 results in increased Mo extraction, reaching a maximum of about 82%. **Figs 6c) and 6d)** show that an increase in pH in the range of 9.0–11.0 and an increase in concentration also contribute to an increase in the degree of extraction, but the influence of these factors is less pronounced compared to temperature. **Figs 6e) and 6f)** show that increasing temperature and pH at a fixed concentration enhances Mo extraction, with maximum values observed at high temperatures (70-85°C) and pH ~10-10.5. In general, it has been established that the main factors determining the process's effectiveness are temperature and concentration, with pH also exerting an additional influence. The optimal conditions for achieving the highest degree of molybdenum extraction (82-83%) correspond to an elevated temperature, a concentration of 1.75–2.5, and a pH of about 10-10.5.

Effective leaching of man-made waste using sodium hydroxide and sodium hydroxide in the presence of sodium hypochlorite.

After determining the optimal parameters of traditional leaching by mathematical modelling, at which the recovery rates were ~88% for vanadium and ~80% for molybdenum, sodium hypochlorite was introduced into the system as an oxidiser to increase the process efficiency.

The results shown in **Figs 7-8** demonstrate the positive effect of adding sodium hypochlorite on the leaching of man-made waste.

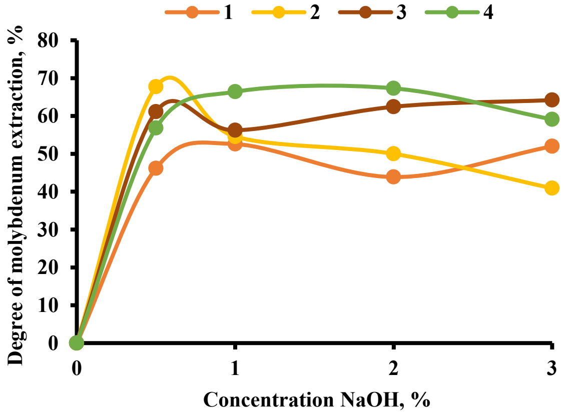
Fig. 7 shows the results of leaching vanadium with alkali and alkali in the presence of sodium hypochlorite.



1- Leaching of V at temperature at a temperature of 25 °C (NaOH-0.5-3%);
2 - Leaching of V at a temperature of 25 °C (NaOH-0.5-3%, (NaOCl);
3- Leaching of V at a temperature of 85 °C (NaOH-0.5%);
4 - Leaching of V at a temperature of 85 °C(NaOH-0.5-3%, (NaOCl)

Fig. 7 - Leaching of vanadium oxides with alkali and alkali in the presence of sodium hypochlorite

Next, **Fig. 8** shows the results of leaching molybdenum oxides with alkali and alkali in the presence of sodium hypochlorite.



1 - Leaching of Mo oxides at a temperature of 85 °C (NaOH-0.5-3%);
2 - Leaching of Mo at a temperature of 25 °C (NaOH-0.5-3%, (NaOCl);
3- Leaching of Mo at a temperature of 85 °C (NaOH-0.5-3%);
4 – Leaching of Mo at a temperature of 85 °C(NaOH-0.5-3%, (NaOCl)

Fig. 8 Leaching of molybdenum oxides with alkali and alkali in the presence of sodium hypochlorite

The optimal leaching conditions for vanadium extraction were achieved using a 0.5 wt.% NaOH solution in the presence of sodium hypochlorite at a temperature of 25 °C, under which the degree of vanadium leaching reached approximately 90–95%, while further increases in alkali concentration or temperature did not result in a significant improvement.

The optimal leaching conditions for molybdenum extraction were achieved using a 0.5-1 wt.% NaOH solution in the presence of sodium hypochlorite at a temperature of 85 °C, under which the degree of molybdenum leaching reached approximately 65–70%, while further increases in alkali concentration did not result in a significant improvement.

Thus, the use of sodium hypochlorite significantly enhances the leaching efficiency of vanadium and molybdenum compared with conventional technology, highlighting its potential as an effective auxiliary reagent.

According to the results of mathematical modelling, the degree of extraction of vanadium is 89-93%, and molybdenum—82-83%. However, the experimentally obtained value was lower than that predicted by the model. To identify the reasons for the reduced extraction efficiency, an X-ray phase analysis of the leached residue was performed.

Figures 9, 10 show X-ray images of precipitation after leaching at 25 °C using NaOH-0.5%, 20 g/l NaOCl and NaOH-0.5%. **Figs 11, 12** show X-ray images of precipitation after leaching at 85°C using NaOH-0.5%, 20 g/l NaOCl and NaOH-0.5%.

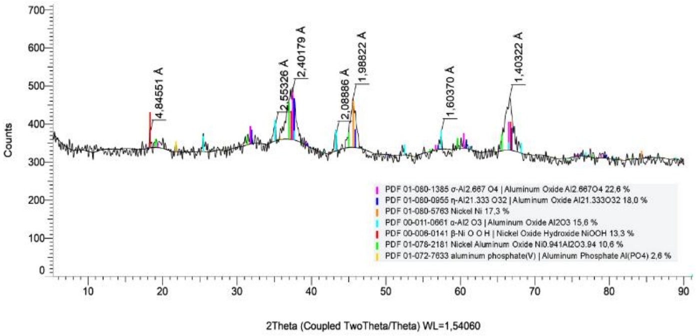


Fig. 9 Sediment after leaching 25°C (NaOH-0.5%, 20 g/l NaOCl

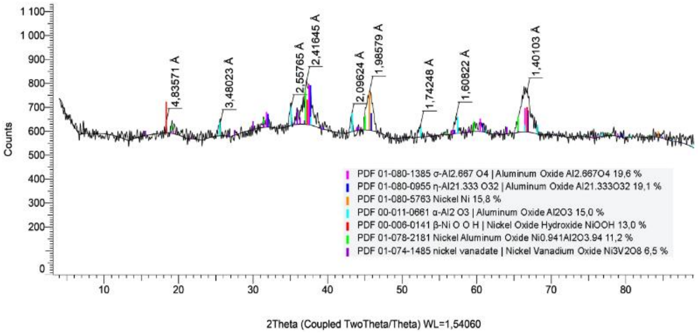


Fig. 10 Sediment after leaching 25°C (NaOH-0.5%)

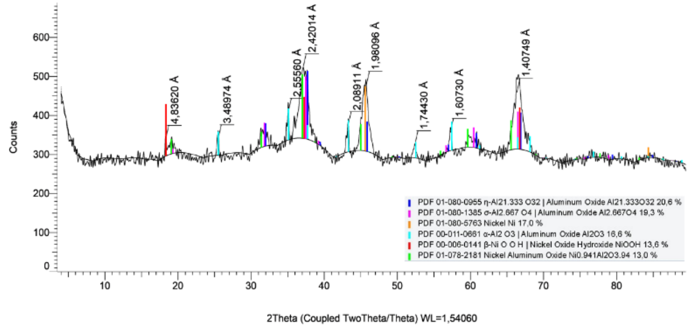


Fig. 11 Sediment after leaching 85°C (NaOH-0.5%, 20 g/l NaOCl)

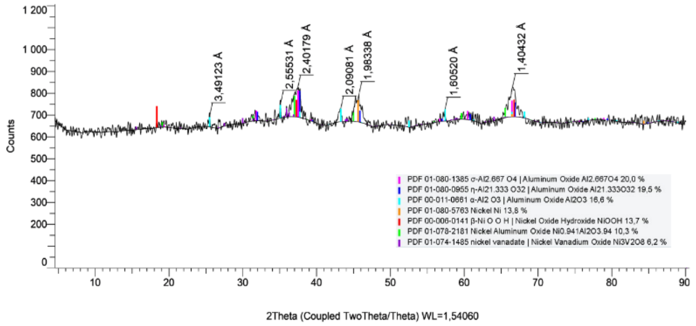


Fig. 12 Figure caption Sediment after leaching 85°C (NaOH-0.5%)

The results obtained after analysing the radiographs in **Figs 9-12** are shown in **Table 6**.

Table 6 X-ray phase analysis of precipitation after leaching

Compound Name	Formula	%
Sediment after leaching 25°C (NaOH-0.5%, 20g/l NaOCl)		
σ-$Al_{2.667}$ O_4 | Aluminum Oxide	$Al_{2.667}O_4$	22.6
η-$Al_{21.333}$ O_{32} | Aluminum Oxide	$Al_{21.333}O_{32}$	18.0
Nickel	Ni	17.3
α-Al_2O_3 | Aluminum Oxide	Al_2O_3	15.6
β-NiOOH | Nickel Oxide Hydroxide	NiOOH	13.3
Nickel Aluminum Oxide	$Ni_{0.941}Al_2O_{3.94}$	10.6
Aluminum Phosphate	$Al(PO_4)$	2.6
Sediment after leaching 25°C (NaOH-0.5%)		
σ-Al2$_{.667}$ O_4 | Aluminum Oxide	$Al_{2.667}O_4$	19.6
η-$Al_{21.333}$ O_{32} | Aluminum Oxide	$Al_{21.333}O_{32}$	19.1
Nickel	Ni	15.8
α-Al_2O_3 | Aluminum Oxide	Al_2O_3	15.0
β-NiOOH | Nickel Oxide Hydroxide	NiOOH	13.0
Nickel Aluminum Oxide	$Ni_{0.941}Al_2O_{3.94}$	11.2
Nickel Vanadium Oxide	$Ni_3V_2O_8$	6.5
Sediment after leaching 85°C (NaOH-0.5%, 20 g/l NaOCl)		
η-$Al_{21.333}O_{32}$ | Aluminum Oxide	$Al_{21.333}O_{32}$	20.6
σ-$Al_{2.667}O_4$ | Aluminum Oxide	$Al_{2.667}O_4$	19.3
Nickel	Ni	17.0
α-Al_2O_3 | Aluminum Oxide	Al_2O_3	16.6
β-NiOOH | Nickel Oxide Hydroxide	NiOOH	13.6
Nickel Aluminum Oxide	$Ni_{0.941}Al_2O_{3.94}$	13.0
Sediment after leaching 85°C (NaOH-0.5%)		
σ-$Al_{2.667}O_4$ | Aluminum Oxide	$Al_{2.667}O_4$	20.0
η-$Al_{21.333}O_{32}$ | Aluminum Oxide	$Al_{21.333}O_{32}$	19.5
α-Al_2 O_3 | Aluminum Oxide	Al_2O3	16.6
Nickel	Ni	13.8
β-NiO H | Nickel Oxide Hydroxide	NiOOH	13.7
Nickel Aluminum Oxide	$Ni_{0.941}Al_2O_{3.94}$	10.3
Nickel Vanadium Oxide	$Ni_3V_2O_8$	6.2

X-ray phase analysis of precipitation after leaching showed that the main phases under all studied conditions (25 and 85°C, with and without the addition of NaOCl) are aluminium oxides (σ-Al_2O_3, nn-$Al_{1.333}O_3$) and nickel-containing compounds (NiO, β-NiOOH, Ni-Al-oxides), the total content of which exceeds 60%.

During leaching without an oxidiser (NaOH 0.5%), the formation of nickel-vanadium oxide $Ni_3V_2O_8$ (6-8%) was recorded, which indicates the fixation of part of the vanadium in a difficult-to-dissolve form. This phase was not detected under conditions of NaOCl application; however, a high proportion of aluminium and nickel oxides remains, limiting the extraction of vanadium and molybdenum.

An increase in temperature to 85°C is accompanied by a higher content of aluminium and nickel oxides, which further reduces the process efficiency.

Thus, the decrease in the degree of extraction of V and Mo is explained by their fixation in stable oxide and mixed phases, as well as the formation of insoluble compounds under alkaline leaching conditions.

CONCLUSION

This study demonstrated the feasibility of recovering vanadium and molybdenum from technogenic vanadium-production waste (filter cake) by alkaline leaching and optimizing the process parameters using response surface methodology (RSM). Chemical and X-ray diffraction analyses confirmed that the waste contains appreciable amounts of V (3.44%) and Mo (0.75%), with vanadium predominantly occurring in sodium–vanadium bronze phases (β-$Na_{0.33}V_2O_5$ and α-NaV_2O_5), alongside stable Al- and Ni-bearing phases that influence leaching behavior.

A central composite design enabled the evaluation of the effects of key operating variables and their interactions on metal extraction. The developed regression models were statistically significant and suitable for describing the process response within the investigated factor ranges, as confirmed by ANOVA and diagnostic residual analyses.

The optimisation results indicated that maximal vanadium extraction is achieved at relatively low temperatures in alkaline media, whereas molybdenum recovery benefits from elevated temperatures. Under the selected conditions, vanadium extraction reached ~88–89% and was further enhanced upon addition of sodium hypochlorite, which promotes oxidation of V^{4+} to V^{5+} and increases the fraction of soluble vanadate species. For molybdenum, the best results were obtained at higher temperatures, with extraction typically in the range of 80–83% under optimised conditions and further improvement in the presence of NaOCl.

X-ray phase analysis of leach residues revealed that the persistence of thermodynamically stable aluminum oxides and nickel-containing phases, and the possible formation of poorly soluble mixed compounds (e.g., Ni–V oxides in the absence of oxidant), can limit the completeness of metal recovery and explain deviations between predicted and experimental values.

Overall, the combined application of alkaline leaching with an oxidizing additive and statistical modeling provides an effective route for process optimization and enhanced recovery of valuable metals from multicomponent industrial wastes. The obtained results form a basis for developing resource-efficient, environmentally oriented technologies for processing technogenic raw materials in Kazakhstan.

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