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Investigation of the extraction of vanadium and molybdenum from vanadium production waste by alkaline leaching with sodium hypochlorite

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Abstract

Vanadium and molybdenum are critical metals widely used in steel production, alloys, and green energy technologies. With growing global demand, efficient recycling methods for industrial waste are essential. This study investigates the extraction of vanadium (3.44%), molybdenum (0.75%), and nickel (8.82%) from vanadium production waste using hydrometallurgical methods.

Optimal leaching conditions were determined through experiments with sodium carbonate (Na_2CO_3) and sodium hydroxide (NaOH) solutions. The highest vanadium recovery (50.56%) was achieved with 1% Na_2CO_3 at 80°C, while molybdenum extraction reached 56.33% with 3% Na_2CO_3. Alkaline leaching with 0.5% NaOH and sodium hypochlorite (NaClO) at 25°C improved vanadium recovery to 88% and molybdenum to 67.7%. However, increasing the temperature to 85°C led to nickel vanadate ($Ni_3V_2O_8$) formation, reducing vanadium extraction efficiency.

X-ray phase analysis confirmed the presence of aluminium oxides (Al_2O_3), nickel compounds, and vanadium phases in the residues. A proposed processing scheme ensures high metal recovery while minimising environmental impact through closed-cycle operations. This study advances sustainable metal extraction from industrial waste, offering a cost-effective and eco-friendly solution for resource recovery.

Keywords: man-made waste processing, vanadium-containing concentrates, vanadium, molybdenum, hydrometallurgy, alkaline leaching, industrial waste.

1. Introduction

Vanadium and molybdenum are becoming increasingly recognised as important components in various industries such as steel and alloy production, the chemical indus-try, and green energy. These metals play a key role in ensuring the high strength, heat re-sistance, and durability of materials, making them indispensable in fields such as me-chanical engineering, energy, and battery technology. According to forecasts, the global vanadium market is expected to grow from $3.46 billion in 2024 to $4.89 billion by 2032, which corresponds to an average annual growth rate (CAGR) of 4.4% during the forecast period [1]. The molybdenum market, in turn, is estimated at $4.25 billion in 2022 and will grow at an average annual rate of 4.0% until 2032 [2].

The key factors influencing the growth in demand for vanadium and molybdenum are the growing demand in the steel and energy industries, as well as the increasing use of these metals in battery technologies. In particular, molybdenum is actively used as an alloying additive to increase the strength and heat resistance of steel, which makes it im-portant for the production of building and machine structures, as well as for high-temperature processes in the energy industry [3]. Vanadium is in demand in metallurgy, energy, and new technologies like energy storage batteries, which boosts its global economic growth [4]. The main leading regions in the production of these metals remain China, Russia and South Africa, which together account for a significant share of the global supply of vanadium and molybdenum [5], [6]. To meet the growing demand for these materials, efficient and environmentally friendly recycling technologies are needed, including man-made waste containing vanadium, molybdenum and other valuable met-als. Existing waste recycling methods can be divided into pyrometallurgical, hydrometal-lurgical and combined technologies, each of which has its own characteristics, ad-vantages and limitations.

Vanadium was extracted from clinker [7] obtained as a result of calcination firing in three stages of countercurrent acid leaching. At the first stage, leaching was carried out at pH 2.8–3.5, followed by filtration and washing of the solid precipitate with water. In the second stage, the pH was lowered to between 1.8 and 3.5 using a solution of sulphuric acid, which included some of the washing solution from the first stage, and then the solid material was filtered. At the third stage, leaching was carried out at a pH of 0.7–1.5. The pulp was sent for filtration, the solid precipitate was washed, and the filtrate was sent for vanadium extraction. In laboratory conditions, at a temperature of 50-60 °C and a pH of 2.8–3.2, the vanadium dissolution rate reached about 88%, which confirms the effective-ness of the proposed three-stage leaching scheme.

The actual raw materials for the production of vanadium are slags [8] processed using lime-sulphate technology. This method is based on multi-stage weak acid leaching, which ensures efficient extraction of vanadium.

At the first stage of the process, leaching is carried out at a pH of 3.2–4.4, with a pos-sible adjustment of the pH level by adding sulphuric acid. The second stage is carried out at pH 3.0–3.8, providing further dissolution of vanadium compounds. At the third stage, the pH parameters are maintained in the range of 2.5–3.4, contributing to a gradual in-crease in the concentration of dissolved vanadium. The final, fourth–seventh stages are carried out at a pH of 2.1–2.5, which makes it

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possible to achieve maximum extraction of the target element. The temperature regime during leaching is maintained in the range of 22-58°C, ensuring optimal conditions for the process. At the end of the seventh stage, the degree of vanadium extraction reaches 97.5%, which confirms the high efficiency of the proposed technology.

Spent catalysts are an economical secondary source of valuable metals such as mo-lybdenum, vanadium, nickel and cobalt, which make up 10-30%, 1-12%, 0.5-6%, and 1-6%, respectively [9]. This makes the recovery and reuse of these catalysts an important task to reduce their costs and environmental pollution from waste.

There are different ways to recover valuable metals from spent catalysts, such as us-ing chlorination, acid leaching, alkaline leaching, bio-leaching, and firing with alkali metal compounds [10].

The authors of [11] suggested a way to extract vanadium from used RHDP catalysts by combining the Fenton reaction with alkaline leaching. The Fenton reaction, using hydrogen peroxide, was used to decompose oily petroleum substances, which contributed to the hydrophilization of the surface of the raw material and increased the availability of vanadium for subsequent leaching [12].

Next, the treated material was leached with 0.5 M $Na_2S_2O_8$ at a temperature of 70°C for 5 hours, which ensured the oxidation of vanadium sulphide to oxide. The final stage of the process included alkaline leaching at 80 °C for 1 hour. As a result of the studies conducted, it was found that the degree of vanadium extraction reaches 90.92%, while partial dissolution of aluminium and sulphur is observed.

The Balausa Limited Liability Company has carried out the modernisation of the hy-drometallurgical plant, aimed at processing concentrates for the extraction of vanadium and molybdenum [13]. The technology provides for the extraction of vanadium and molybdenum by over 80%, respectively.

The man-made waste generated during the processing of concentrates requires disposal; this waste contains undetected vanadium and molybdenum, as well as nickel and a significant amount of aluminium.

2 Matherials and methods

2.1 Materials

The object of the study was representative samples of man-made waste from the pro-cessing of vanadium-containing concentrates of the Balausa Company hydrometallurgical plant.

The following reagents were used in leaching experi-ments: Grade B sodium car-bonate (Na_2CO_3 - 99.2%), tech-nical flaked sodium hydroxide (NaOH - at least 98.5%), so-dium hypochlorite (grade A) and distilled water.

2.2. Methods

2.2.1 Analytical methods

A wide range of analytical methods was used for the complex analysis of man-made waste.

Chemical analysis carried out in the factory laboratory allowed us to determine the elemental composition of the initial waste. The titrimetric method was used for the de-ter-mination of vanadium and the photometric method for the determination of molyb-denum [14-16].

X-ray phase analysis was performed on a Bruker D8 ADVANCE apparatus using copper radiation at an acceler-ating voltage of 36 kV and a current of 25 mA and was used to identify the mineralogical composition of both the starting materials and the processed products, as well as to determine the phase composition.

The quantitative content of the elements was determined using X-ray fluorescence analysis on a Venus 200 spectrom-eter from PANalytical B.V. (PANalytical B.V., the Neth-er-lands).

2.2.2 Leaching technique

The process of leaching vanadium and molybdenum from man-made waste was carried out in a thermostatically controlled reactor equipped with an IKA-20 digital me-chanical stirrer, which provides a fixed number of revolutions, under atmospheric condi-tions at different concentrations of carbonate and sodium hydroxide (0.5-3%), the ratios T:W (1:1, 1:2, 1:3, 1:4) and temperature (25-85 °C); the duration of the process is 8 hours to determine the optimal conditions. Leaching with sodium hydroxide was also performed in the presence of sodium hypochlorite (150 g/kg) at 25°C and 85°C.

During the study of the leaching process, samples were taken at regular intervals. Af-ter leaching was completed, the pulp was filtered, the filtrate was analysed for the content of target metals, and the solid residue (cake) was washed ten times with distilled water, after which the precipitate was dried and calcined to obtain a nickel concentrate.

3 Results and discussion

3.1 The results of physico-chemical studies of technogen-ic waste from vanadium production

The objects of the study were representative samples of technological waste from vanadium production, for which chemical, X-ray, spectral, mineralogical, and thermogravimetric methods of analysis were used.

Table 1. The chemical analysis of the feedstock performed in the factory laboratory.

Product Name	**The content of the elements, %**										
	Na	Al	Si	P	S	K	Ca	V	Fe	Ni	Mo
Wastes	3,12	42,05	0,46	0,42	0,46	0,06	0,20	3,44	0.6	8.82	0,75

According to Table 1, the main component of the raw material is aluminium (Al), the content of which is 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. The sample also contains iron (Fe) 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.

In addition, phosphorus (P) was 0.42%, sulphur (S) was 0.46%, silicon (Si) was 0.46%, potassium (K) was 0.06%, and calcium (Ca) was 0.20% in the composition of man-made raw materials. These elements can affect both the processing methods and the purity standards of the final products.

According to X-ray phase analysis studies, the sample includes minerals such as corundum Al_2O_3 – 27.6%; aluminium oxide γ–Al_2O_3 modification – 14.1%; complex magnesium and aluminium oxide $MgAl_2O_4$ – 15.5%; calcium carbonate $CaCO_3$ – 15.1%; nickel silicate Ni_2SiO_4 – 11.6%; iron oxides Fe_2O_3 – 5.9%; binary iron compounds of silicon and vanadium – 5.8% (Fe_2SiV); and nickel Ni – 4.3%. The research results are shown in Figure 1.

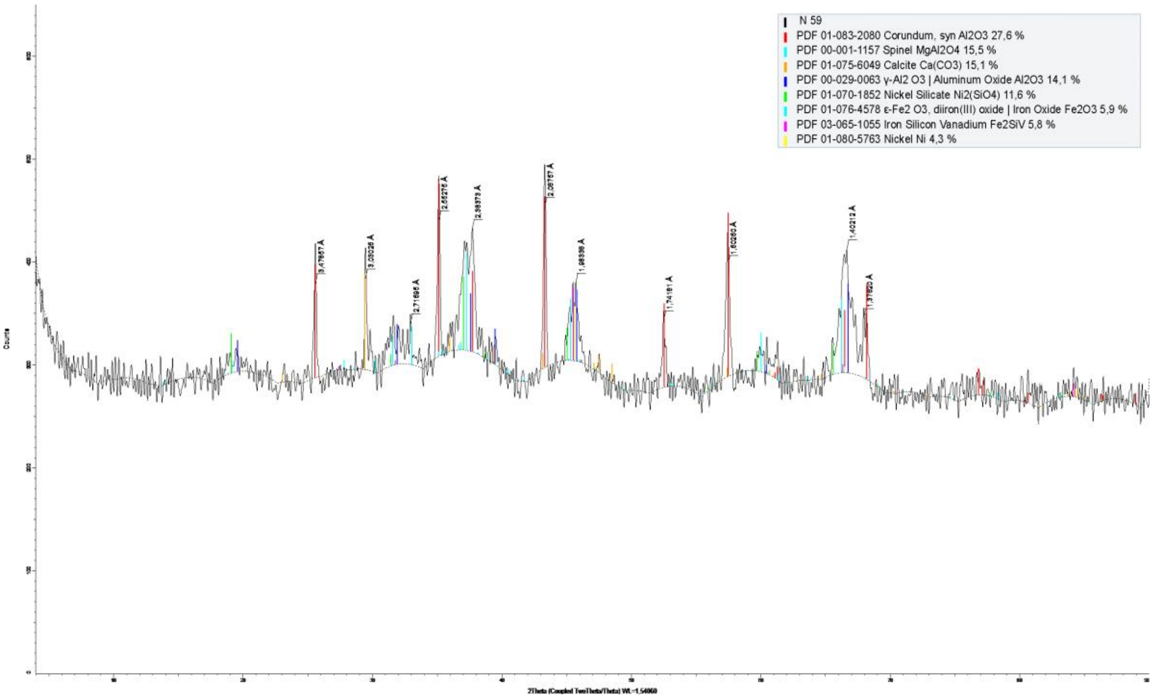


Figure 1. X-rays of the initial industrial product of vanadium production

3.2. The results of physico-chemical studies of technogen-ic waste from vanadium production

Various reagents such as sulphuric acid, soda ash, and sodium hydroxide can be used to leach man-made waste of this composition. However, in the vanadium production of the Balasauskandyk deposit, soda and alkaline leaching technology are used as the best option.

The results of the leaching process are influenced by a number of factors, including the concentration of reagents, the duration of the process, temperature, the size of the solid phase, the ratio of solid to liquid, and other parameters. During the development of a method for extracting valuable components from man-made waste, experimental studies on leaching were carried out under atmospheric conditions to study the effects of the concentration of sodium carbonate and sodium hydroxide, temperature and duration of the process.

Influence of the concentration of soda and alkali and the temperature of the leaching process

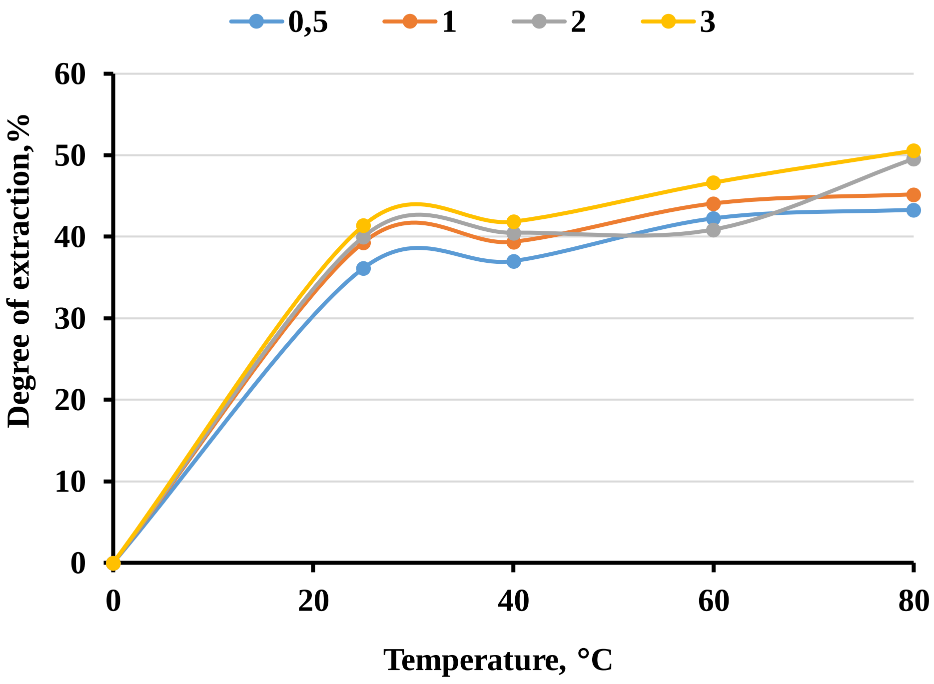
Experiments on the leaching of vanadium and molybdenum using garden and alkaline solutions were carried out at various concentrations of reagents and temperatures.

The concentrations of sodium carbonate and sodium hydroxide used as leaching agents ranged from 0.5 to 3%. This concentration range was chosen taking into account the possibility of working in a closed cycle with the possibility of solvent regeneration, as well as to minimise the volume of solutions formed.

The S:L ratio during the leaching process was 1:2 with a process duration of 8 hours. The leaching of the target metals, vanadium and molybdenum, was carried out according to the previously described method.

The results of these experimental studies are shown in Figures 2-3, which make it possible to analyse the effect of reagent concentration and temperature on the degree of vanadium and molybdenum extraction.

a



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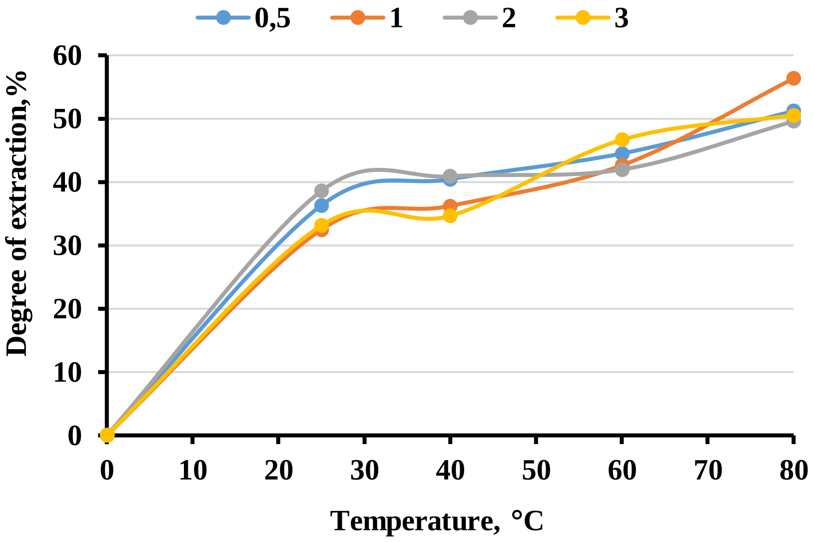


Figure 2. Influence of temperature and concentration of soda on the degree of extraction of molybdenum (a) and vanadium (b)

An increase in the concentration of Na_2CO_3 in the range of 0.5–3% leads to a natural increase in the degree of molybdenum extraction from 32.49% to 56.33% (Figure 2a). At the same time, an increase in the concentration of carbonate ions (CO_3^{2-}) shifts the equilibrium of reactions towards the formation of soluble molybdate complexes. The process intensifies with an increase in temperature to 80 °C, which leads to a maximum degree of molybdenum extraction (56.33%) by accelerating the diffusion of reagents to the particle surface and reducing the viscosity of the solution.

A similar trend is observed in the leaching of vanadium (Figure 2b). With an increase in the concentration of sodium carbonate from 0.5 to 3%, the degree of vanadium recovery is in the range from 36.14 to 50.56%. Increasing the concentration of sodium carbonate to 5% with a S:L ratio of 1:2 did not yield significant results in enhancing the extraction degree of vanadium and molybdenum, as the salt background of the productive solution increased.

Next, Figure 3 shows the dependence of the degree of extraction of molybdenum (Figure 3a) and vanadium (Figure 4b) on the concentration of sodium hydroxide and temperature.

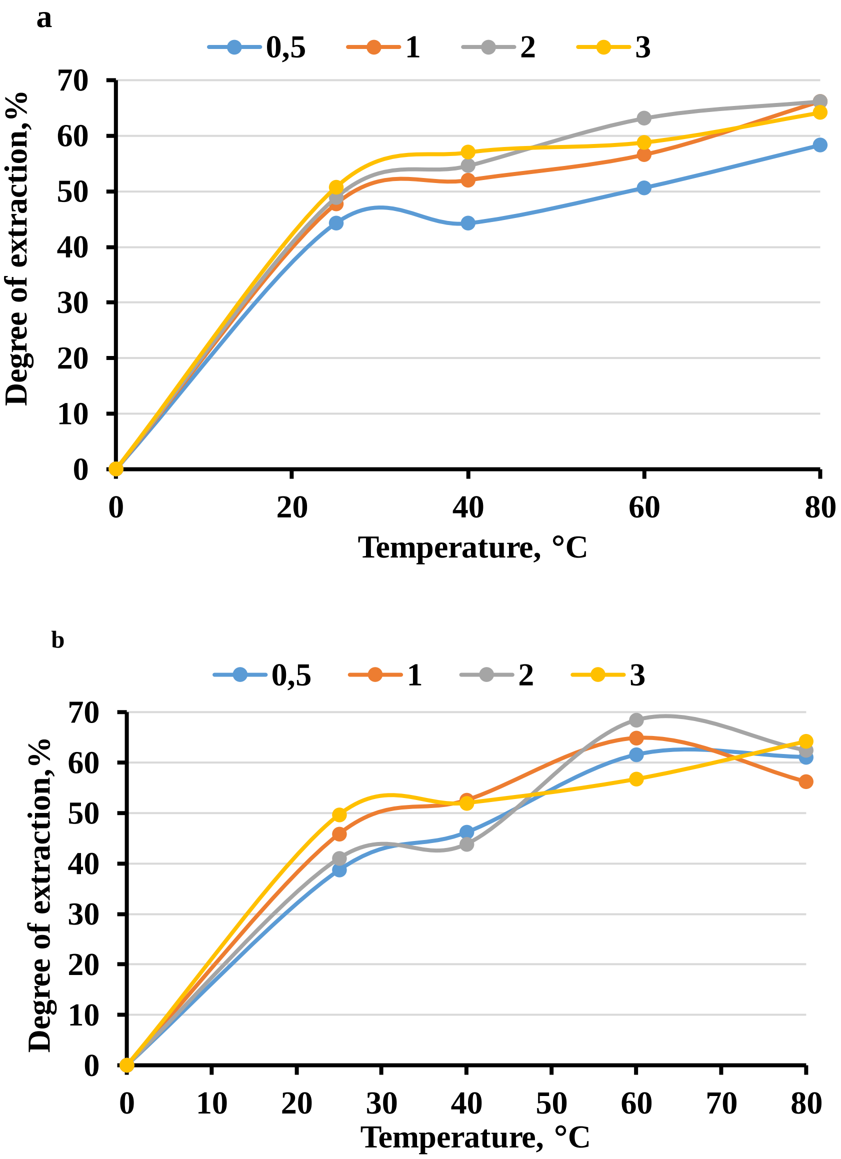
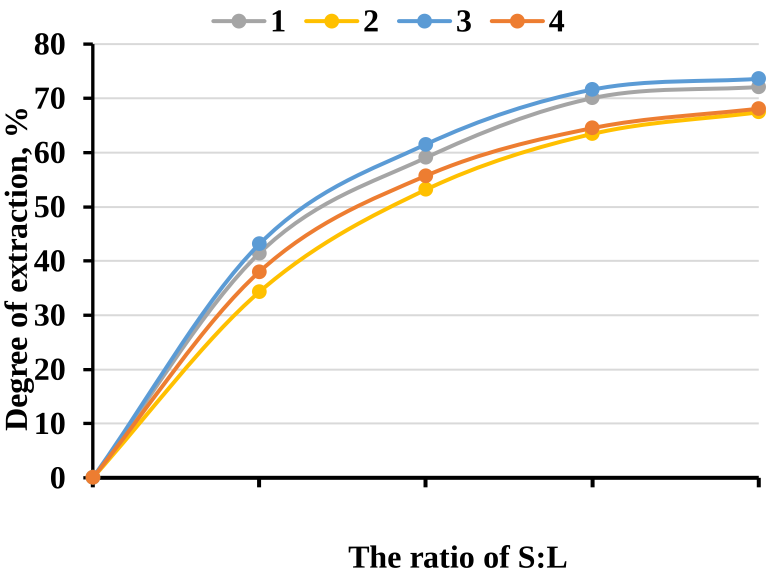


Figure 3. Influence of temperature and concentration of sodium hydroxide on the degree of extraction of molybdenum (a) and vanadium (b)

The study shown in Figures 3 (a) and 3 (b) demonstrates the effect of temperature and NaOH concentration on the degree of molybdenum and vanadium recovery. With increasing temperature, there is a general tendency to increase the degree of extraction of these metals. The maximum recovery rate of molybdenum is 68.44% at a temperature of 60 °C and an alkali concentration of 2%, while for vanadium the maximum recovery rate reaches 66.17% at 80°C and NaOH concentrations in the range of 1-2%.

The results indicate that the optimal alkali concentration is 1-2% at a temperature of 80 °C and a ratio of S:L = 1:2, at which the degree of extraction of vanadium and molybdenum is about 70%.

Next, it was studied how the S:L ratio affected the leaching of vanadium and molybdenum using carbonate and sodium hydroxide (Figure 4). The leaching process was carried out at a ratio of S:L = 1:1, 1:2, 1:3, 1:4 at a temperature of 80 °C. The process duration is 8 hours, and the concentration of Na_2CO_3 is 1% (process 1 and 2) and NaOH is 1% (process 3 and 4).

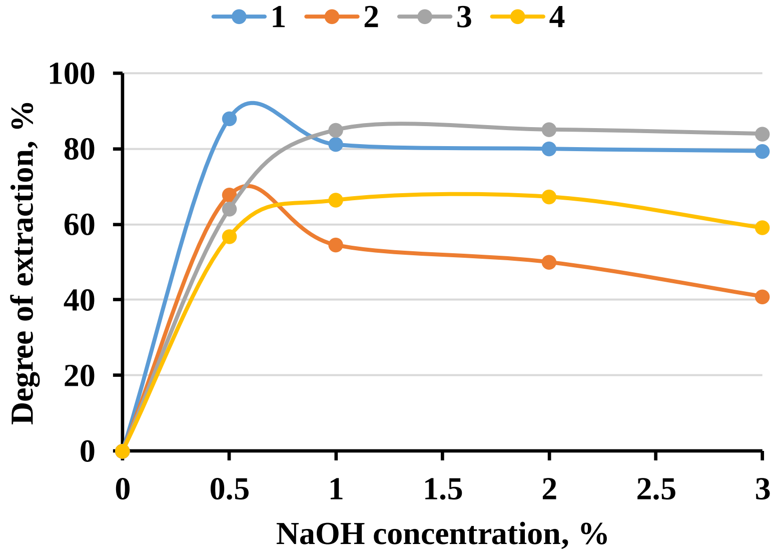


Leaching with Na_2CO_3 solution: 1- V; 2- Mo
Leaching with NaOH solution: 3-V; 4-Mo

Figure 4. Dependence of the degree of extraction of molybdenum and vanadium on the ratio S:L

Figure 4 shows that when using sodium carbonate at a S:L ratio of 1:4, vanadium has a cure rate of 72.1% and molybdenum has 67.43%. When using sodium hydroxide at the same ratio, the cure rates are 73.6% for vanadium and 68.1% for molybdenum. As the S ratio increases, the amount of target metals in the solution decreases, which means more concentration steps are needed. Then, vanadium and molybdenum were extracted using sodium hydroxide with sodium hypochlorite added, at temperatures of 25 and 85 °C. With an increase in the ratio of S, there is a decrease in the concentration of target metals in the productive solution, which requires additional concentration processes.

Further,, vanadium and molybdenum were leached with sodium hydroxide with the addition of an oxidising agent in the form of sodium hypochlorite at temperatures of 25 and 85 °C. The results of the experiment are shown in Figure 5.



Leaching is performed using a NaOH solution at a temperature of 25 °C: 1- V; 2- Mo
Leaching with NaOH solution at T-85 °C: 3-V; 4-Mo

Figure 5. Effect of NaOH concentration on the degree of extraction of vanadium and molybdenum at a temperature of 25°C and 85°C

As shown in Figure 5, the presence of sodium hypochlorite (150 g/kg) has a significant effect on the efficiency of the vanadium and molybdenum leaching process in an alkaline environment. At a temperature of 25 ° C, an increase in the concentration of NaOH from 0 to 0.5% leads to a sharp increase in the degree of extraction of both elements: the yield of vanadium reaches 88%, and molybdenum – 67.7%. However, with a further increase in the alkali concentration (>0.5%), the extraction of Mo decreases, while the degree of extraction of V stabilises at 80%.

When the temperature rises to 85 °C, a similar dependence is observed: in the range of 0-0.5% NaOH, the extraction of vanadium and molybdenum increases to ~85% and ~70%, respectively. In the range of 0.5–1.5%, the yield of both metals remains almost constant, with minor fluctuations. At concentrations of NaOH >1.5%, the recovery rate of V remains at ~80%, while a slight decrease is recorded for Mo.

Table 2 displays the X-ray phase analysis of the materials that formed after being treated with sodium hydroxide and sodium hypochlorite at temperatures of 25°C and 85°C.

Table 2. Results of X-ray phase analysis of sediment after leaching with sodium hydroxide in the presence of sodium hypochlorite at temperatures of 25°C and 85°C

Name	**Formula**	**Alkali concentration %, T-25℃**				**Alkali concentration %, T-85℃**			
		0,5	**1,0**	**2,0**	**3,0**	**0,5**	**1,0**	**2,0**	**3,0**
Aluminum Oxide	$\sigma\text{-}Al_{2.667}O_4$	22,6	26,0	22,1	19,3	18,4	15,7	18,3	20,0
Aluminum Oxide	$\eta\text{-}Al_{21.333}O_{32}$	18,0	24,1	19,6	20,6	18,2	19,1	16,6	19,5
Nickel	Ni	17,3	19,6	17,5	17,0	15,6	22	21,3	13,8
Aluminum Oxide	$\alpha\text{-}Al_2O_3$	15,6	-	15,3	16,6	15,4	17,7	12,5	16,6
Nickel Oxide Hydroxide	$\beta\text{-}NiOOH$	13,3	-	13,4	13,6	13,3	11,0	13,2	13,7
Nickel Aluminum Oxide	$Ni_{0.941}Al_2O_{3.94}$	10,6	15,4	12,0	13,0	10,5	9,2	12,0	10,3
Aluminum Phosphate	$Al(PO_4)$	2,6	14,8	-	-	-	-	-	-
Nickel Aluminum Oxide	$Ni_{0.941}Al_2O_{3.94}$	-	-	-	-	8,7	5,3	6,1	6,2

A study of the phase composition of precipitation presented in Table 2, formed during leaching with sodium hydroxide in the presence of sodium hypochlorite, showed a significant effect of NaOH concentration (0.5–3.0%) and temperature (25 °C and 85 °C) on the distribution of aluminium, nickel and vanadium-containing phases.

The composition of the phases remains, but there is a redistribution of their content. The main change when leaching at 85 °C is that nickel vanadate ($Ni_3V_2O_8$) is formed, and its amount goes down from 8.7% with 0.5% NaOH to 6.2% with 3.0% NaOH. This indicates that sodium hypochlorite promotes the oxidation of vanadium to higher degrees of oxidation, which in combination with nickel leads to the precipitation of $Ni_3V_2O_8$. However, with an increase in the concentration of NaOH, the solubility of vanadium compounds increases, which explains the decrease in the proportion of $Ni_3V_2O_8$ in the sediment.

The formation of the $Ni_3V_2O_8$ compound can lead to undertreatment of vanadium. At the same time, at a temperature of 25 ° C, vanadium completely dissolves, while only aluminium in various modifications and nickel in the form of various compounds remain in the precipitate.

Based on the results obtained (Figure 6), it was found that the best conditions for leaching vanadium and molybdenum are achieved using 0.5% sodium hydroxide in the presence of sodium hypochlorite in an amount of 150 g/kg at a temperature of 25 °C and a ratio of S:L = 1:2.

The chemical composition of the productive solution and sediment after leaching is presented in Tables 2-3.

Table 3. Chemical composition of the productive solution after leaching

Name	**Content, g/l**				
	pH	V_{total}	Mo	P	Al
Productive solution	10,83	17,23	3,33	1,5	4,54

According to the data presented in Table 2, the main target component of the productive solution is vanadium, the total concentration of which (V Total) is 17.23 g/l. In addition, the solution contains molybdenum (3.33 g/l), phosphorus (1.5 g/l) and aluminium (4.54 g/l).

The productive solution was processed according to the method proposed by the authors. [17]

Table 4 below shows the chemical composition of the sediment formed after the alkaline leaching of man-made waste.

Table 4. Chemical composition of sediment after alkaline leaching

Name	**Content, %, g**				
	V	Mo	P	Ni	Al
Sediment after leaching	0,345	0,14	0,15	13,17	36,7

The precipitate after leaching contains a significant amount of aluminium (36.7%), which indicates the incomplete dissolution of the aluminium-containing phases under the selected process conditions. The increased nickel content (13.17%) indicates its concentration in the sediment. Simultaneously, residual amounts of vanadium (0.345%) and molybdenum (0.14%) confirmed their preferential transition to a productive solution.

At the leaching stage, the recovery rate of vanadium was 88.0% and of molybdenum 67.7%. The precipitate after leaching contains a large amount of nickel and aluminium. Figure 6 shows an X-ray of the resulting sediment.

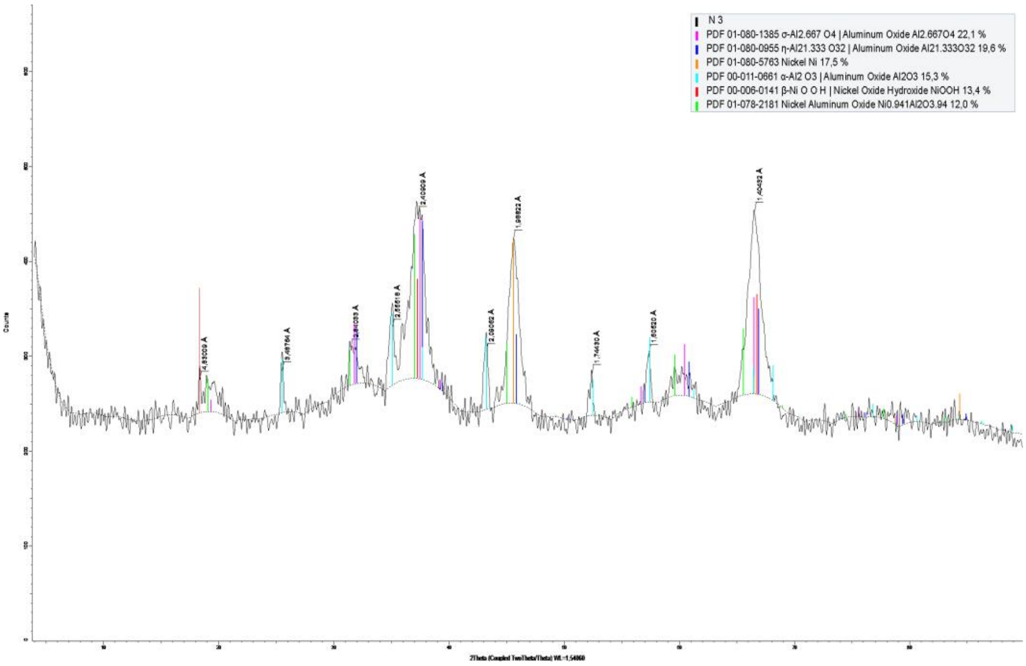
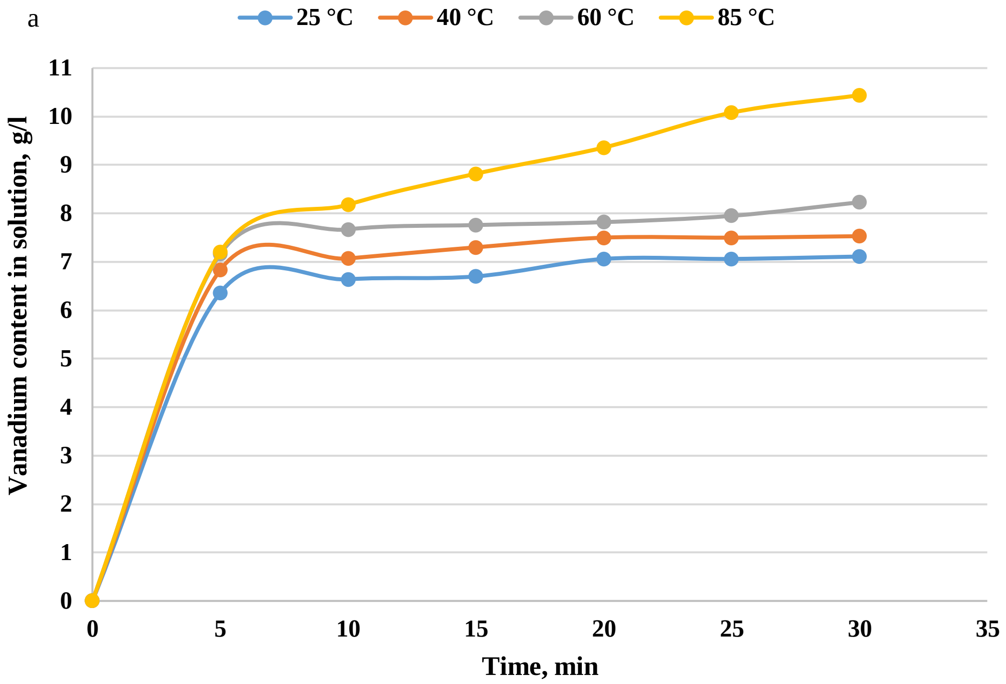


Figure 6. X-rays of sediment after leaching

The X-ray image of the sediment shows a complex multicomponent composition, including several phases. The main phase is σ-$Al_{2.667}O_4$ (aluminium oxide) with a content of 22.1%, followed by n-$Al_{21.333}O_{32}$ (aluminium oxide) with a content of 19.6%. Metallic nickel (Ni) is present in an amount of 17.5%, which indicates the presence of an elementary form of nickel in the sample. α-Al_2O_3 (corundum) – 15.3%, β-NiOOH (nickel oxide-hydroxide) – 13.4%, and mixed nickel and aluminium oxide $Ni_{0.941}Al_2O_{3.94}$ – 12.0% were also detected.

The series of studies has demonstrated that it's possible to extract vanadium and molybdenum using low-concentration solutions of alkali and soda from processed concentrates, achieving a high level of extraction.

The following graphs show the dependence of vanadium (Figure 7a) and molybdenum (Figure 7b) concentrations in the presence of sodium hypochlorite during leaching at various temperatures: 25°C, 40°C, 65°C, and 85°C.



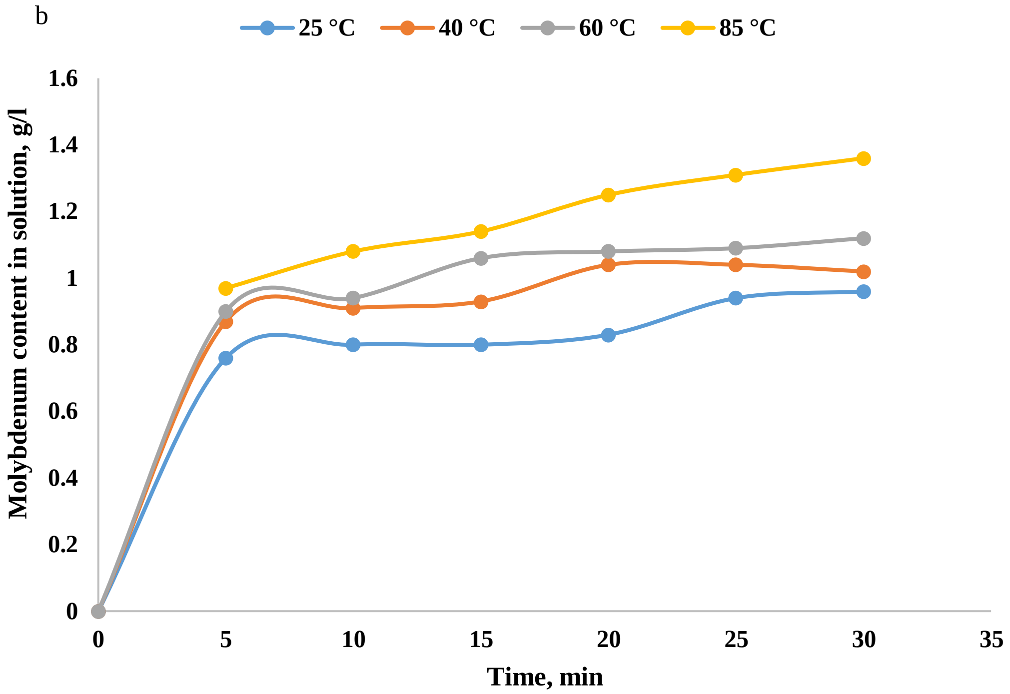


Figure 7. Dependence of vanadium (Figure 7a) and molybdenum (Figure 7b) concentrations in the presence of sodium hypochlorite during leaching at various temperatures: 25°C, 40°C, 65°C, and 85°C.

Figure 7a shows the variation of vanadium concentration in solution over time at different temperatures. As the temperature increases, the vanadium content in the solution also increases significantly. The leaching process is most effective at 85°C, where the vanadium concentration continuously grows, reaching about 10.6 g/l after 30 minutes.

Figure 7b presents the molybdenum concentration in solution over time at the same temperatures. Similar to vanadium, the molybdenum concentration increases with temperature. The highest molybdenum content is achieved at 85°C, reaching 1.36 g/l after 30 minutes.

Table 5 below shows the results of the X-ray phase analysis of the cake after leaching of man-made raw materials with an alkaline solution of sodium hypochlorite.

Table 5. The results of the X-ray phase analysis of the cake after leaching of man-made raw materials with an alkaline solution of sodium hypochlorite

Name	**Formula**	**Alkali concentration %, T-25°C**				**Alkali concentration %, T-40°C**				**Alkali concentration %, T-60°C**				**Alkali concentration %, T-85°C**			
		0,5	1,0	2,0	3,0	0,5	1,0	2,0	3,0	0,5	1,0	2,0	3,0	0,5	1,0	2,0	3,0
Aluminum Oxide	σ-$Al_{2.667}O_4$	22,6	26,0	22,1	19,3	20,0	20,6	19,6	20,6	19,6	19,0	19,9	20,0	18,4	15,7	18,3	20,0
Aluminum Oxide	η-$Al_{21.333}O_{32}$	18,0	24,1	19,6	20,6	17,5	18,7	18,2	19,1	19,1	18,6	18,8	19,5	18,2	19,1	16,6	19,5
Nickel	Ni	17,3	19,6	17,5	17,0	13,6	15,3	16,1	16,9	15,8	18,2	16,8	16,6	15,6	22	21,3	13,8
Aluminum Oxide	α-Al_2O_3	15,6	-	15,3	16,6	14,4	14,6	15,1	13,4	15,0	14,7	14,3	13,8	15,4	17,7	12,5	16,6
Nickel Oxide Hydroxide	β-NiOOH	13,3	-	13,4	13,6	13,8	13,2	12,8	13,3	13,0	11,8	13,3	13,7	13,3	11,0	13,2	13,7
Nickel Aluminum Oxide	$Ni_{0.941}Al_2O_{3.94}$	10,6	15,4	12,0	13,0	10,9	11,9	11,9	12,7	11,2	11,6	12,3	10,3	10,5	9,2	12,0	10,3
Aluminum Phosphate	$Al(PO_4)$	2,6	14,8	-	-	-	-	-	-					-	-	-	-
Nickel Vanadium Oxide	$Ni_3V_2O_8$	-	-	-	-	6,1	5,7	6,4	4,1	6,5	6,1	4,6	6,2	8,7	5,3	6,1	6,2
Vanadium Oxide	V_2O_3					3,7%	-	-	-								

Based on the results of X-ray phase analysis (Table 5), the mechanism of formation of nickel vanadate ($Ni_3V_2O_8$) during leaching of technogenic raw materials with an alkaline solution of sodium hypochlorite was suggested.

The process of formation of nickel vanadate ($Ni_3V_2O_8$) during the leaching of man-made raw materials using an alkaline solution of sodium hypochlorite (NaOCl) includes several key stages due to redox reactions and features of complexation in the system.

At the first stage, sodium hypochlorite, being a strong oxidising agent in an alkaline environment, oxidises vanadium contained in the feedstock (for example, in the Fe_2SiV compound) from low oxidation states (+3 or +4) to the highest degree of oxidation, +5. This leads to the formation of soluble vanadate ions by the following reactions:

For V^{3+}: $2V^{3+} + 3ClO^- + 6OH^- \rightarrow 2VO_4^{3-} + 3Cl^- + 3H_2O$

For V^{4+}: $V^{4+} + ClO^- + 2OH^- \rightarrow VO_4^{3-} + Cl^- + H_2O$

The resulting vanadate ions VO_4^{3-} pass into solution, providing effective leaching of vanadium.

Nickel in the starting material is present in the form of metallic nickel (Ni_0), Ni^{2+} compounds (for example, in nickel silicates such as Ni_2SiO_4), and also, possibly, in nickel-aluminate phases ($NiAl_2O_4$-like compounds). In an alkaline environment, nickel is characterised by extremely low solubility. Even with partial oxidation of nickel with hypochlorite to form β-NiOOH, a significant part of it remains in insoluble form – in the form of metal, oxides, hydroxides, silicates and aluminates.

The key stage of the mechanism is the interaction of soluble vanadate ions with nickel ions, which are locally released from the surface of solid nickel-containing particles or from poorly soluble complexes. At a temperature of at least 40°C and under conditions of sufficient alkali concentration (0.5–3.0%), a precipitation reaction occurs between VO_3^- and Ni^{2+}, resulting in the formation of insoluble nickel vanadate:

$3Ni^{2+} + 2VO_4^{3-} \rightarrow Ni_3V_2O_8\downarrow$

The leaching temperature plays an important role in this process. An increase in temperature increases the ion diffusion rate, accelerates reactions at the phase boundary, and probably promotes partial dissolution of the surface layers of nickel-containing compounds, facilitating access of vanadate ions to nickel ions. On the other hand, a sufficient concentration of alkali is necessary to stabilise vanadium in a soluble form (VO_3^-) until it reacts with nickel. If the alkalinity is insufficient, other, less soluble forms of vanadium may be formed, which reduces the leaching efficiency.

The formation of $Ni_3V_2O_8$ is a side reaction that reduces the yield of vanadium into solution, since part of the vanadium precipitates again in Thus, with an increase in temperature and alkali concentration, favourable conditions are created both for the effective leaching of vanadium and for its partial binding to the insoluble phase of nickel vanadate.

Experimental data confirm the proposed mechanism: the formation of $Ni_3v_2o_8$ is observed at leaching temperatures of 40°C and above, even at a minimum alkali concentration (0.5%). At a lower temperature (25°C), nickel vanadate is not detected in the precipitate. At the same time, nickel in various phases (Ni, β-NiOOH, nickel-aluminates) is constantly present in the residue, providing a source of Ni^{2+} for the precipitation reaction. It was also found that at a temperature of 40°C and an alkali concentration of 0.5% in the sediment, partial preservation of vanadium in the form of V_2O_3 is possible, which indicates an incomplete degree of oxidation of vanadium under these conditions, but the main contribution to the retention of vanadium in the sediment is the formation of $Ni_3V_2O_8$.

Further, Figure 8 shows the basic technological scheme for processing man-made waste.

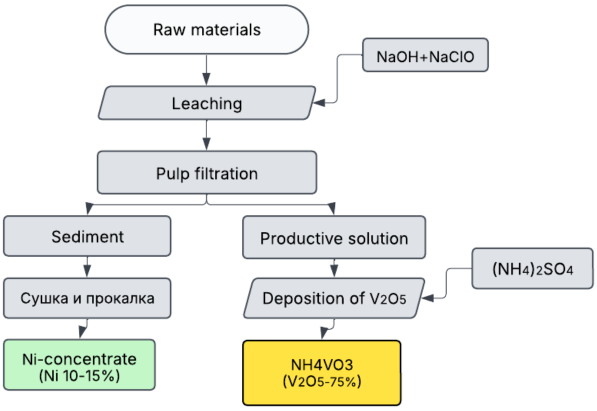


Figure 8. Basic technological scheme of processing of technogenic waste from vanadium production

The basic technological scheme is aimed at the complex processing of man-made waste, including such valuable components as vanadium (V), molybdenum (Mo), nickel (Ni) and aluminium (Al).

At the first stage, alkaline leaching is carried out using aqueous solutions of sodium hydroxide (NaOH) and sodium hypochlorite (NaClO) at a temperature of 25 °C. Optimal process conditions include a NaOH concentration within 0.5% and a solid-liquid phase ratio (S:L) of 1:2. As a result of leaching, vanadium and molybdenum pass into solution, while nickel and aluminium are concentrated in the solid residue.

The resulting pulp is filtered, during which it is separated into a productive solution and a solid residue. Vanadium (up to 17.23 g/l) and molybdenum (up to 3.33 g/l) are concentrated in the productive solution. The precipitate contains up to 13.17% nickel and 36.7% aluminium.

The solid residue is sent to drying and calcination, resulting in a precipitate with a nickel content of 10-15%. Further processing can be carried out by pyrometallurgical or hydrometallurgical methods to extract nickel and aluminium.

After filtering, the useful solution is used to get vanadium as ammonium metavanadate (NH_4VO_3), which can be turned into V_2O_5 with up to 75% content after heating. For precipitation, $(NH_4)_2SO_4$ and NaOH are used, maintaining the required pH level and ensuring the selectivity of the process.

Molybdenum extraction can be organised in the following stages using selective precipitation or sorption methods. The developed technological scheme is focused on environmental safety: it provides for a closed cycle with waste minimisation and reagent regeneration. This approach makes it possible to achieve high metal recovery rates (up to 88% for vanadium).

4. Conclusions

This research presents a comprehensive investigation into the extraction of vanadium and molybdenum from man-made waste generated during vanadium production, using alkaline leaching in the presence of sodium hypochlorite. The experimental results confirmed that under optimal conditions—namely 0.5% NaOH, 150 g/kg NaClO, at 25 °C and a solid-to-liquid (S:L) ratio of 1:2—it is possible to achieve high recovery rates of vanadium (88.0%) and molybdenum (67.7%). These results demonstrate the high efficiency of mild alkaline oxidative leaching as an alternative to more aggressive chemical processes, supporting its use in industrial-scale recycling of vanadium-containing waste.

A key finding of the study was the role of sodium hypochlorite in enhancing vanadium oxidation and solubility. However, the formation of nickel vanadate ($Ni_3V_2O_8$) at higher temperatures ($\geq$40 °C) was identified as a limiting factor, potentially reducing the vanadium yield due to secondary precipitation. This insight provides valuable guidance for controlling process parameters to avoid losses of the target element. X-ray phase and chemical analyses of the residue confirmed the phase composition of the solid waste and helped clarify the mechanisms of vanadium and molybdenum behavior during leaching.

Furthermore, a technological scheme was proposed for the integrated processing of the productive solution and solid residue. It enables the recovery of vanadium as ammonium metavanadate, molybdenum through selective precipitation, and nickel and aluminium from the solid cake via subsequent treatment. This holistic approach not only improves resource efficiency but also reduces environmental impact by minimizing waste generation and enabling potential regeneration of leaching agents.

The developed methodology could be adapted for processing similar types of waste from other metallurgical operations, thereby supporting the circular economy and reducing dependency on primary resources.

Author Contributions

Conceptualization,.O.B.; methodology, S.K.; validation, O.B.; formal analysis, M.A.; investigation, M.S., M.A.; data curation, Ye.M.; writing—original draft preparation, S.Yu.; writing—review and editing, A.K.; visualization, Ye.M.; supervision, S.K., A.K.; project administration, S.Yu., A.K.; funding acquisition, S.Yu. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement

The original contributions presented in the study are included in the article, further inquiries can be directed to the corresponding author.

Conflicts of Interest

The authors declare no conflicts of interest.

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