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Resource-saving technology for processing of spent vanadium catalysts with vanadium pentoxide production and integrated utilization of secondary products

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ABSTRACT

The objective of this study is to develop a resource-saving technology for processing spent vanadium catalysts (SVCs) aimed at reducing technogenic load and establishing a closed-loop production cycle. The methodology involves acidic leaching of vanadium using sulfuric acid (2–4 M, 80 °C), followed by a two-stage ammonification of the filtrate and thermal decomposition of the resulting precipitate. The results demonstrate high vanadium recovery rates (up to 97–98%) and the production of high-purity vanadium pentoxide (98.2%). By-products include sulfate-ammonium fertilizers (8–10% N, 2–3% P_2O_5, 1–2% K_2O) and structural materials based on oxide impurities (SiO_2, Al_2O_3, Fe_2O_3) used as a support for a new vanadium catalyst. The developed gas purification system ensures 99.5% efficiency in ammonia capture, enabling its return to the technological cycle. The findings confirm the feasibility of industrial implementation of the proposed technology, with potential integration into existing sulfuric acid production facilities, thus enabling environmentally safe disposal of SVCs while simultaneously generating commercially valuable products.

Keywords: spent vanadium catalysts, hydrometallurgy, acid leaching, vanadium oxide, closed-loop cycle.

INTRODUCTION

Vanadium is a relatively abundant element in the Earth's crust and possesses significant technological importance. Although up to 85% of its global production is consumed by the metallurgical industry (for ferrovanadium and steel alloying), vanadium pentoxide (V_2O_5) holds particular industrial value. This compound is an indispensable catalyst in the contact process, which is the primary method for large-scale sulfuric acid production (Imtiaz et al., 2015; Kurniawan et al., 2024; Nasimifar and Mehrabani, 2022).

Metal contamination poses a global environmental challenge, as evidenced by numerous cases of toxic exposure in mining areas, industrial complexes, coal-fired power plants, and agricultural regions (Imtiaz et al., 2015; Petranikova et al., 2020). Soils contaminated with heavy metals adversely affect plant systems, water resources, and ecological balance, creating risks for trophic chains. Among these metal pollutants, vanadium (V) has attracted particular attention from the scientific community due to its high mobility within the soil-plant system and its associated negative effects on human health. The referenced sources summarize current knowledge on vanadium, its environmental role, and recent advances in research on its toxicity, integrating data from previous studies to support an interdisciplinary assessment of the ecological significance of this element.

Spent vanadium catalysts from sulfuric acid production (typical composition: 7.5% V, 9.1% K, 10.2% S, 23.2% Si, 1.4% Fe) represent a secondary source of vanadium, where efficient recovery addresses both environmental concerns and economic viability (Romanovskaia et al., 2021). Two- and three-stage hydrometallurgical methods were applied for their processing, involving acid leaching, reduction, oxidation, and precipitation. Optimization of process parameters (temperature, reagent concentrations – H_2SO_4, Na_2SO_3, $(NH_4)_2S_2O_8$; solid-to-liquid ratio, duration, agitation) yielded a maximum vanadium recovery of 98%. This was achieved through a two-stage process comprising acid leaching (H_2SO_4, pH 1.2–1.3, ultrasound treatment for 5 min), followed by reduction (0.01 mol/L Na_2SO_3, 15 min) at room temperature and oxidation with 30 wt.% $(NH_4)_2S_2O_8$ solution ($n(V_2O_5)/n((NH_4)_2S_2O_8) = 5:1$, 5 min, 80-90 °C), resulting in vanadium pentoxide with a purity of 85–87% and the development of a closed-loop utilization scheme. The three-stage method, tested on an industrial catalyst (FACT Ltd), confirmed the importance of parameter optimization and chemical control of the product (Calderón and Endara, 2016). Fundamental surface studies of $V_2O_5(001)$ using *in situ* bandgap mapping revealed a reversible metal-insulator transition (350–400 K), attributed to anisotropic formation of V=O and oxygen vacancies, which accounts for oxygen loss and phase instability; at elevated temperatures, an irreversible reduction pathway $V_2O_5 \rightarrow V_6O_{13} \rightarrow V_2O_3$ is observed, which is significant for understanding catalyst degradation mechanisms during operation (Blum et al., 2007).

The recovery of V_2O_5 is primarily considered from spent vanadium catalysts. A laboratory approach was developed that combines non-destructive analysis techniques (particle size distribution, XRD, ICP, XRF) to characterize the samples before and after treatment. The focus is placed on environmentally friendly leaching using less hazardous acids and alkalis. A comparative analysis of reagents identified oxalic acid as the most effective for dissolving vanadium ions. Process parameters – including contact time, acid concentration, solid-to-liquid ratio, stirring speed, and temperature – were optimized. The maximum vanadium extraction efficiency in batch mode (67.43%) was achieved using 6 wt.% oxalic acid at a solid-to-liquid ratio of 1:10, stirring speed of 300 rpm, and a temperature of 50 °C. Column leaching (column dimensions: 150 × 5 cm) with the same reagent over a contact period of 144 hours yielded a significantly higher extraction rate of 94.42%. The results confirm the technical feasibility of efficient V_2O_5 recovery from this type of secondary raw material (Al Amayreh et al., 2023).

Numerous studies (Blum et al., 2007; Imtiaz et al., 2015; Mikoda et al., 2020; Nasimifar and Mehrabani, 2022; Romanovskaia et al., 2021; Vinco et al., 2025) focused on the chemical reduction of V_2O_5 from spent catalysts. A critical step in this process is the separation of the oxide from the support material, which is achieved through various leaching schemes. A range of leaching agents was employed under varying conditions, including sulfuric acid (Al Amayreh et al., 2023), oxalic and citric acids (Mikoda et al., 2020), ammonium thiosulfate, and hydrogen peroxide.

In the study by Choi et al. (2018), a method for the direct recovery of vanadium from spent SCR catalysts was proposed, based on alkaline autoclave leaching using NaOH. It was established that optimal process parameters (3 mol/l NaOH, 250 °C, particle size < 150 μm, S/L = 0.4) result in a vanadium extraction efficiency of 91.5%, which is 25–30% higher than that achieved by conventional atmospheric leaching. The key factors contributing to process intensification include temperature-concentration synergy, which accelerates the breakdown of the catalyst matrix, and particle size optimization, which reduces diffusion limitations. Notably, this method eliminates the need for an acid treatment stage.

Spent vanadium catalysts (SVCs) represent a promising secondary source of vanadium, as the V_2O_5 content in these materials can reach 4–8% (Dadakhodzhaev et al., 2025; Tarasov et al., 2022), significantly exceeding the concentrations found in most primary ore sources. After repeated regeneration cycles, during which they lose catalytic activity, selectivity, and conversion efficiency, SVCs are classified as secondary resources requiring processing to recover valuable components. The projected growth in demand for hydrotreating catalysts suggests an increase in the volume of spent materials, thereby highlighting the urgency of developing highly efficient recycling technologies. A review of the scientific literature reveals a wide range of processing methods for SVCs, including acid, alkaline, and soda leaching. However, the effectiveness of existing approaches remains limited, underscoring the need for further research aimed at optimizing vanadium oxide recovery.

The storage of SVCs poses a significant environmental threat due to the potential migration of toxic compounds of vanadium, aluminum, cesium, and silicon into soil and groundwater. This necessitates the mandatory treatment or recycling of such waste. The most rational approach involves the development of integrated technologies aimed not only at the safe extraction of hazardous elements but also at the recovery of marketable products, such as V_2O_5, a highly demanded reagent in metallurgy and the chemical industry. Additionally, processing by-products containing sodium and potassium can be utilized as liquid mineral fertilizers, thereby enhancing both the economic and environmental viability of the process. Accordingly, the objective of this study is to implement resource-saving methods for the processing of SVCs that contribute to reducing technogenic load and establishing a closed-loop system for vanadium-containing raw materials.

MATERIALS AND METHODS

Sample description

The SVC of the SVD (K-D) grade (Technical Specifications TU 48-0323-6-90 with amendments 1.2.3.4) was provided by Kazphosphate LLP (Mineral Fertilizer Plant, Taraz, Republic of Kazakhstan). The material consisted of deactivated cylindrical granules (average diameter: 6 mm; length: 20 mm), collected after discharge from the contact unit of an industrial reactor.

To enhance the efficiency of subsequent leaching, the samples were mechanically ground using a BS BALLMILL-I laboratory ball mill for 15 minutes. The resulting powder was further purified from ferromagnetic impurities (primarily iron) using a magnetic separator. Final sample preparation included drying the material at 100 °C for 1 hour to remove adsorbed moisture. Sulfuric acid conforming to GOST 2184-2013 (Interstate Council for Standardization, Metrology and Certification (ISC), 2014), manufactured by Kazphosphate LLP, was used for vanadium extraction from the SVC and subsequent V_2O_5 production.

Material analysis

A total of fifteen samples were collected from five different layers of the contact apparatus. The morphology, elemental composition, and phase composition of the catalyst surface were examined using a high-performance scanning electron microscope (SEM) JSM-6490LV equipped with an INCA Energy 450 energy-dispersive microanalysis system. The vanadium content ranged from 3.81% to 6.34%, while vanadium oxide content varied between 5.60% and 9.33%, as shown in Table 1. For each condition, at least three samples were analyzed to ensure the reliability and accuracy of the results.

Sample preparation

The preparation of the working solution containing vanadium was carried out in three stages: (1) grinding and leaching; (2) two-step ammonification; (3) decomposition of ammonium metavanadate.

A 100 g batch of spent vanadium-containing catalyst was ground to a particle size of 1–2 mm and placed in a heat-resistant beaker (800 ml). The sample was treated with 200 ml of 2M H_2SO_4 under constant stirring (220 rpm) at a temperature of 70–90 °C for 3 hours.

The resulting suspension was filtered using vacuum filters. The first filtrate (Filtrate I) was transferred to a heat-resistant 800 ml beaker. The precipitate was used to produce a catalyst support as a substitute for natural diatomite. The heated Filtrate I was treated with a 25% ammonia solution at a temperature of 80–90 °C, with continuous stirring, until the pH reached 6.0.

Table 1. Composition of spent vanadium catalyst (SVC)

Catalysts layer No.	C	O	Na	Al	Si	S	K	V	Fe	Cs
1	1.71	44.66	0.90	0.23	25.59	6.21	6.87	6.34	0.64	6.84
2	2.50	49.06	1.98	0.66	19.91	10.77	9.02	4.77	1.32	
3	2.88	47.59	1.63	0.50	24.96	8.70	8.08	5.03	0.64	
4	2.65	43.89	0.94	0.56	25.20	8.39	6.14	5.53	1.64	5.05
5	2.09	49.98	1.25	0.66	25.79	8.40	7.37	3.81	0.64	

The resulting suspension was filtered; the second filtrate (Filtrate II) was used as a liquid fertilizer. The precipitate was washed with hot water and treated with ammonia solution at a concentration of 12–16 g/l (2%).

Ammonium nitrate (NH_4NO_3) was added to the resulting suspension at a concentration of 100 g/l, followed by stirring. The suspension was then filtered. The third filtrate (Filtrate III) was directed for use as a liquid fertilizer, while the resulting ammonium metavanadate (NH_4VO_3) precipitate was dried at 200 °C. After drying, it was calcined in a muffle furnace at 550–600 °C for 3 hours, yielding pure V_2O_5.

Hydrometallurgical method

Hydrometallurgical methods are based on the leaching of vanadium compounds from SVCs into solution, followed by washing, drying, and calcination of the obtained precipitates.

The extraction of vanadium compounds using aqueous hydrochloric acid is limited by the volatility of hydrogen chloride in aqueous media. Previous studies investigated the ionic speciation of vanadium (V) across a wide concentration range in various inorganic acid solutions, including hydrochloric acid (Chen et al., 2025; Singh et al., 2025; Yang et al., 2024). An extraction-based technology was also developed for vanadium recovery from titanium production wastes.

Since the hydrometallurgical method is more intensive compared to solvent extraction, it is carried out at higher temperatures. In practice, leaching of vanadium from SVCs with nitric acid solutions is not applied. The main reason is the decomposition of nitric acid at leaching temperatures, accompanied by the formation of brown gas, as shown in Chemical Reaction 1:

$$HNO_3 \rightarrow NO_2 + H_2O \tag{1}$$

For this reason, diluted sulfuric acid was selected as the leaching agent (Singh et al., 2025). Sulfuric acid exhibits typical acid-metal reactivity and its boiling point depends on concentration – 104.4 °C at 20%, 107.9 °C at 30%, 113.9 °C at 40%, 124.4 °C at 50%, and 141.8 °C at 60%. Diluted sulfuric acid is non-volatile and readily available, as SVCs originate from the sulfuric acid production process.

In the present study, vanadium pentoxide was extracted from SVCs via a hydrometallurgical approach (Dadakhodzhaev et al., 2025; Vinco et al., 2025). The experimental samples were obtained from sulfuric acid workshops at Kazphosphate LLP (Mineral Fertilizer Plant, Taraz, Republic of Kazakhstan). The SVD (K-D) vanadium catalyst (TU 48-0323-6-90 with amendments 1.2.3.4) was provided by the company after its operational cycle and was classified as waste. A total of five samples were collected from five layers of the contact apparatus.

RESULTS

The amounts of vanadium pentoxide, potassium, and silicon oxides are lower compared to those in the active catalyst, which is due to the use of the catalyst during operation. Iron oxide is found in the spent catalyst as iron is present in the alloy used to manufacture the reactor material where the process takes place.

Composition analysis

Elemental analysis was conducted for each layer of the SVC.

Vanadium pentoxide-based catalysts have the following mineralogical composition, as presented in Table 2.

Vanadium leaching from SVCs, as a technological process, depends on several factors, including temperature, sulfuric acid concentration, leaching duration, and the L/S ratio.

Vanadium extraction from SVCs using sulfuric acid and ammonia solutions was carried out on a laboratory-scale setup consisting of a glass reactor equipped with a paddle stirrer and placed in a thermostatically controlled vessel.

IR spectra of the samples were recorded using a Shimadzu IRPrestige-21 Fourier-transform infrared (FTIR) spectrometer equipped with a Miracle attenuated total reflectance (ATR) accessory from Pike Technologies.

Samples for analysis were prepared by mixing the material with KBr followed by pellet pressing. IR spectra were recorded in the range of 400–1300 cm^{-1}, with a spectral slit width of 4 cm^{-1} at 1000 cm^{-1}. Spectral analysis was performed using a Shimadzu IRPrestige-21 FTIR spectrometer equipped with a Pike Technologies Miracle ATR accessory. The results are presented in Table 3.

Table 2. Mineralogical composition of spent vanadium catalysts from Kazphosphate LLP, wt.%

Catalysts layer No.	K_2O	Na_2O	V_2O_5	FeO	Al_2O_3	SiO_2	Cs_2O
1	8.28	1.21	9.33	0.82	0.43	54.74	7.25
2	10.87	2.67	7.02	1.70	1.25	42.59	
3	9.74	2.20	7.40	0.82	0.95	53.39	
4	7.40	1.27	8.13	2.11	1.06	53.90	5.35
5	8.88	1.69	5.60	0.82	1.25	55.16	

Note:

🟨	Compounds included in the composition of the liquid fertilizer
🟩	Target product
🟧	Precipitate after the first filtration

Table 3. Comparative data on vanadium stretching vibrations

Compounds	Initial SVC	Solid residue
V=O	1098	1099.78
V-O-C	981.81	-
V-O-V	792.7	793.94
V-O	619.52	621085
$O\text{-}(V)_3$	654.49	-
-OH	481.62	483.65
	3384.97	
$-SO_4$	1642.60	-

DISCUSSION

Investigation of the effect of sulfuric acid concentration on the vanadium leaching efficiency

The concentration of sulfuric acid has a significant impact on the efficiency of vanadium leaching from spent catalysts. Higher acid concentrations generally enhance the extraction rate, and the leaching kinetics can be described by different models depending on the specific process conditions and catalyst composition (EL Hasbaoui et al., 2025; Guardiani et al., 2007).

The effect of sulfuric acid concentration on the degree of vanadium leaching from SVCs was studied under the following conditions: room temperature, liquid-to-solid ratio of 3:1, leaching duration of 2 hours, and sulfuric acid concentrations ranging from 0.5 to 4.0 M. The catalyst material was ground to a particle size of 1–2 mm. After each experiment, the mixture was filtered after 2 hours, and the residue on the filter was rinsed with hot water and analyzed for V_2O_5 content. The extent of vanadium extraction into the solution was then calculated. The initial V_2O_5 concentration in the SVC was 4.54%. The solid residue was washed and dried at 200 °C to constant weight. The results are presented in Table 4 and Figure 1.

The data in Table 4 show that during aqueous leaching, an acidic environment is formed due to SO_3 ions adsorbed in the pores of the SVC, which facilitates the dissolution of V_2O_5 into the solution, reaching approximately 19%.

As the concentration of sulfuric acid increases, the vanadium leaching efficiency improves, reaching up to 94.0% (Ju et al., 2015). This enhancement is attributed to the increased concentration of hydrogen ions in the solution. For example, at a sulfuric acid concentration of 0.5 M, the vanadium extraction rate is 71%, whereas increasing the acid concentration to 1.5 M, 2 M, 2.5 M, 3 M, and 4.0 M results in efficiencies of 90%, 94%, 94%, 94%, and 94%, respectively. It can be expected that at 2 M sulfuric acid and an elevated temperature of 70–90 °C, the leaching efficiency may further increase.

As shown in Table 5 and Figure 2, extending the leaching duration to 3 hours increases the vanadium extraction efficiency to 94.10%. Further increasing the leaching time to 4 and 6 hours results in marginal improvements, with extraction efficiencies of 94.50% and 94.55%, respectively.

Thus, the study of the effect of leaching time on vanadium extraction from SVCs demonstrated that the influence of process duration becomes negligible after 2 hours of interaction. Increasing the leaching time threefold – from 1 hour to 6 hours – results in only a 19.3% improvement in extraction efficiency. This finding supports the conclusion that, for pilot-scale implementation of the sulfuric acid leaching technology at temperatures of 70–90 °C, a leaching duration of 3 hours is sufficient.

Table 4. Effect of sulfuric acid concentration on V_2O_5 extraction efficiency

H_2SO_4 concentration, M	Filtrate volume, ml	Solid residue weight, g	V_2O_5 concentration in filtrate, g/l	Percentage of V_2O_5 transferred to solution, %
0	380	71.5	2.39	19
0.5	322	67.8	7.49	71
1.5	270	63.4	15.9	90
2.0	267	59.8	16.92	94
2.5	260	58.3	16.8	94
3.0	258	59.0	16.71	94
4.0	258	59.0	16.71	94

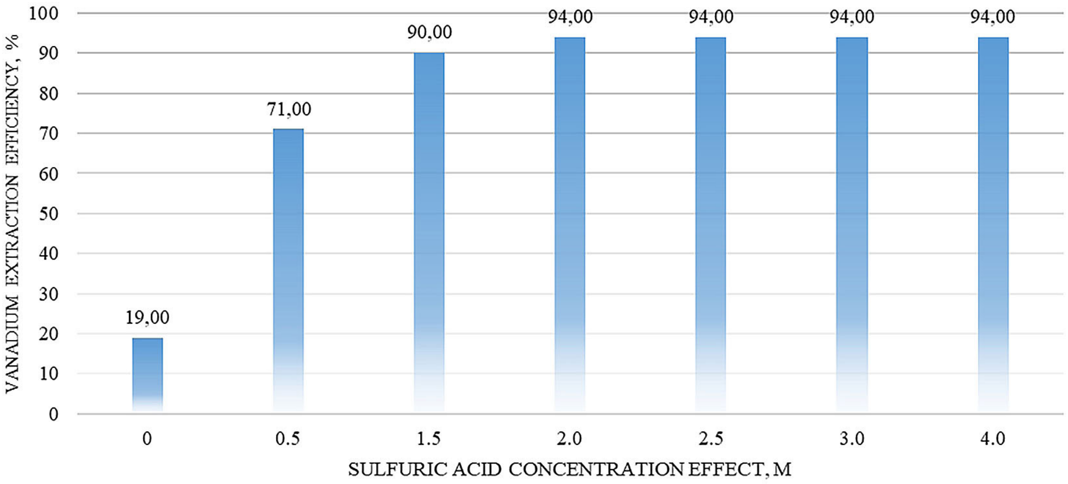

Figure 1. Effect of sulfuric acid concentration on V_2O_5 extraction efficiency

Table 5. Effect of leaching duration on V_2O_5 extraction efficiency

Time, h	Filtrate volume, ml	Solid residue weight, g	Vanadium content in solid residue, %	Vanadium extraction efficiency, %
1	225	55.6	0.788	74.70
2	212	52.3	0.429	89.50
3	233	68.5	0.314	94.10
4	240	67.8	0.298	94.50
6	242	66.4	0.282	94.55

Investigation of the effect of temperature on vanadium leaching efficiency

It should be noted that temperature plays a critical role in the efficiency of vanadium leaching from spent catalysts. Moderate temperatures around 50 °C are generally effective for various leaching agents, while higher temperatures can further enhance the leaching rate (Mazurek, 2013, 2014; Mazurek et al., 2010; Wu et al., 2021).

The vanadium leaching efficiency was studied at a fixed initial sulfuric acid concentration of 2 M over a duration of 2 hours and a liquid-to-solid ratio of 1:1. The SVC samples were ground, and particles with a size less than 150 µm were used.

The leaching efficiency was calculated based on the difference in vanadium content in the initial SVC and the solid residue after leaching, according to Equation 2:

$$\left[\frac{(Cv)\text{initial};-(Cv)\text{concentr}}{(Cv)\text{initial}}\right]\cdot 100 \qquad (2)$$

where: $(C_v)_{initial}$ – the vanadium concentration in the initial SVC, and $(C_v)_{concentr}$ – the vanadium concentration in the solid residue. The experimental results are presented in Table 6 and Figure 3.

An increase in temperature within the range of 30–40 °C has little effect on vanadium extraction from spent vanadium catalysts (SVCs). However, elevating the temperature to 80–90 °C significantly enhances the leaching efficiency of vanadium in the form of V_2O_5, reaching up to 94.6%. This indicates the potential to improve vanadium recovery in 2 M sulfuric acid solutions at higher temperatures.

The variation in vanadium concentration in the solid residue depending on leaching temperature shows that at 70–80 °C, the leaching efficiency reaches 94.6%, while at 30–40 °C, 50–60 °C, 60–70 °C, and 80–90 °C, the efficiency is 83.5%, 86.2%, 89.3%, and 94.4%, respectively. These results suggest that vanadium recovery above 94.6% is achievable at leaching temperatures of 70–80 °C and higher.

Multifactorial nature of the acid leaching process

The authors' investigations confirm the complex nature of the dissolution of vanadium-bearing phases, necessitating a revision of traditional views on the mechanism of this process. As demonstrated in the works of Nannan et al. (2016) and Romanovskaia et al. (2021), in addition to the primary reactions, several key factors influence the system: the catalytic role of iron-containing impurities in the oxidation of V^{4+}, the formation of intermediate vanadate-sulfate complexes, and the effect of local pH gradients at the particle surfaces.

Of particular note is the authors' identification of a previously undescribed phenomenon – the "chemical shielding" effect observed for larger particles (> 150 µm), as illustrated in Figure 4. The simplified reaction: $V_2O_4 + 2H_2SO_4 \rightarrow 2VOSO_4 + 2H_2O$ does not fully represent the process. In practice, the following concurrent reactions are observed:

Oxidation of V^{4+} to V^{5+} by residual oxygen: $4VOSO_4 + O_2 + 2H_2SO_4 \rightarrow 4VO_2SO_4 + 2H_2O$.

Vanadium extraction into the filtrate: 94–95% of the initial vanadium content.

In addition to the primary reaction, sulfuric acid interacts with oxide impurities as follows:

1. $FeO + H_2SO_4 \rightarrow FeSO_4 + H_2O$
2. $4FeSO_4 + O_2 + 2H_2SO_4 \rightarrow 2Fe_2(SO_4)_3 + 2H_2O$

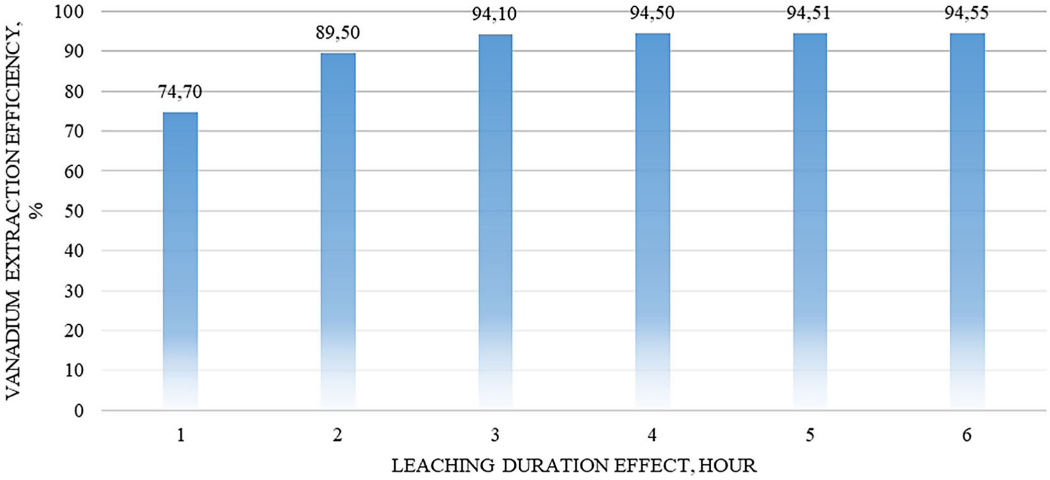

Figure 2. Effect of leaching duration on V_2O_5 extraction efficiency

Table 6. Effect of leaching temperature on V_2O_5 extraction efficiency

Temperature, °C	Filtrate volume, ml	Solid residue weight, g	Vanadium content in solid residue, %	Vanadium leaching efficiency, %
30–40 °C	252	83.0	0.710	83.5
50–60 °C	237	77.2	0.680	86.2
60–70 °C	265	72.5	0.676	89.3
70–80 °C	286	69.7	0.497	94.6
80–90 °C	280	71.2	0.481	94.4

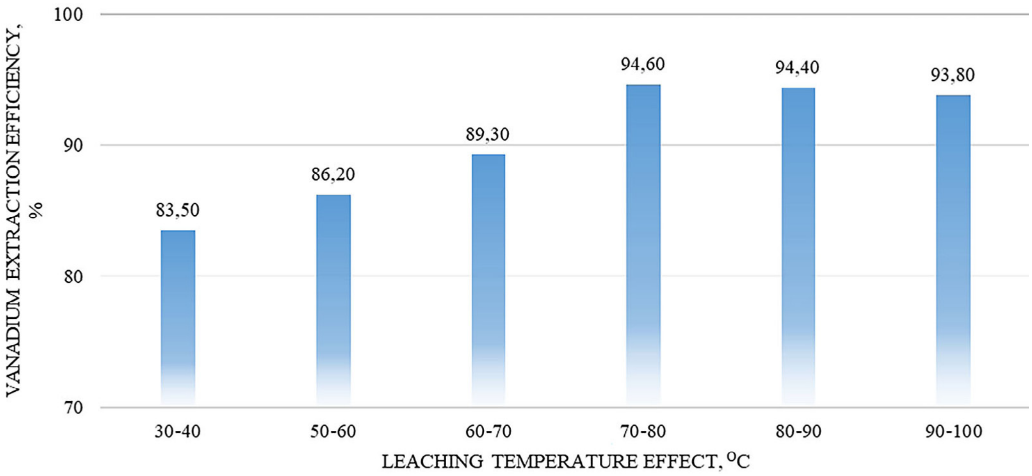

Figure 3. Effect of leaching temperature on V_2O_5 extraction efficiency

3. $Fe^{2+} + 2H_2O \rightarrow Fe(OH)_2 + 2H^+$
4. $Al_2O_3 + 3H_2SO_4 \rightarrow Al_2(SO_4)_3 + 3H_2O$
5. $2Al_2O_3 + 3H_3SO_4 \rightarrow Al_2(SO_4)_3 \cdot Al_2O_3 + 3\ H_2O$
6. $SiO + H_2SO_4 \rightarrow Si(OH)_4 + SO_2\uparrow$ (upon heating)
7. $2SiO + O_2 \rightarrow 2SiO_2$ (insoluble)
8. $SiO_2 + H_2O \rightarrow H_2SiO_3$ (colloidal)
9. $Cs_2O + H_2SO_4 \rightarrow 2Cs^+ + SO_4^{2-} + H_2O$
10. $Cs_2O + 2\ H_2SO_4 \rightarrow 2CsHSO_4 + H_2O$

Following leaching, the solid residue retains SiO_2 (100%), Al_2O_3 (98–99%), Fe^{+2} (90–92%), and Cs^+ (100%), whereas vanadium (as the target component) and alkali metals K^+/Na^+ (100%) are transferred into the filtrate.

Optimization of double ammonification – new approaches

The proposed precipitation scheme, despite achieving high vanadium recovery (94–95%), raises key concerns:

- Selectivity of precipitation: The results indicate that at pH 5.8–6.2, substantial co-precipitation of Fe^{3+} occurs – up to 8% of the total iron content – contradicting theoretical predictions. This is exemplified by the following reaction: $Fe^{3+}+3NH_4OH \rightarrow Fe(OH)_3\downarrow+3NH_4^+$
- Thermodynamic considerations: The residual concentration of V^{4+} in solution (0.8–1.2 g/l) suggests a need to re-evaluate the generally accepted stability constants of vanadium complexes.

These findings are consistent with recent studies (Elhoucine et al., 2024; Fu et al., 2025; Singh et al., 2025; Wang and Yang, 2022).

In this part of the experiment, the precipitate was washed with water at a liquid-to-solid (L:S) ratio of 8:1 and re-treated with 2% NH_4OH at 60–70 °C for 2 hours. Afterward, NH_4NO_3 was added at a concentration of 100 g/l. Within 5–7 minutes, the salt fully dissolved in the solution, which was subsequently used as a liquid fertilizer. The final precipitate was subjected to thermal treatment.

Hidden challenges of thermal decomposition

Phase analysis of the thermolysis products revealed several unexpected effects. The formation of a metastable V_6O_{13} phase was observed in the temperature range of 350–450 °C. Partial reduction to V_2O_4 occurred due to insufficient aeration. At temperatures above 550–600 °C, an "autocatalytic" particle sintering effect was triggered. These observations call for a reassessment of conventional thermal behavior models of NH_4VO_3 described in classical studies.

- Identified phase transitions:
  - 200–350 °C: $NH_4VO_3 \rightarrow (NH_4)_2V_6O_{16} + NH_3 + H_2O$
  - 350–450 °C: $(NH_4)_2V_6O_{16} \rightarrow V_6O_{13} + 2NH_3 + H_2O$
  - 450 °C: $V_6O_{13} + O_2 \rightarrow 3V_2O_5$

CONCLUSIONS

The authors developed a resource-efficient technology for processing spent vanadium catalysts, enabling comprehensive recovery of valuable components with minimal environmental impact. A key achievement is the reduction of

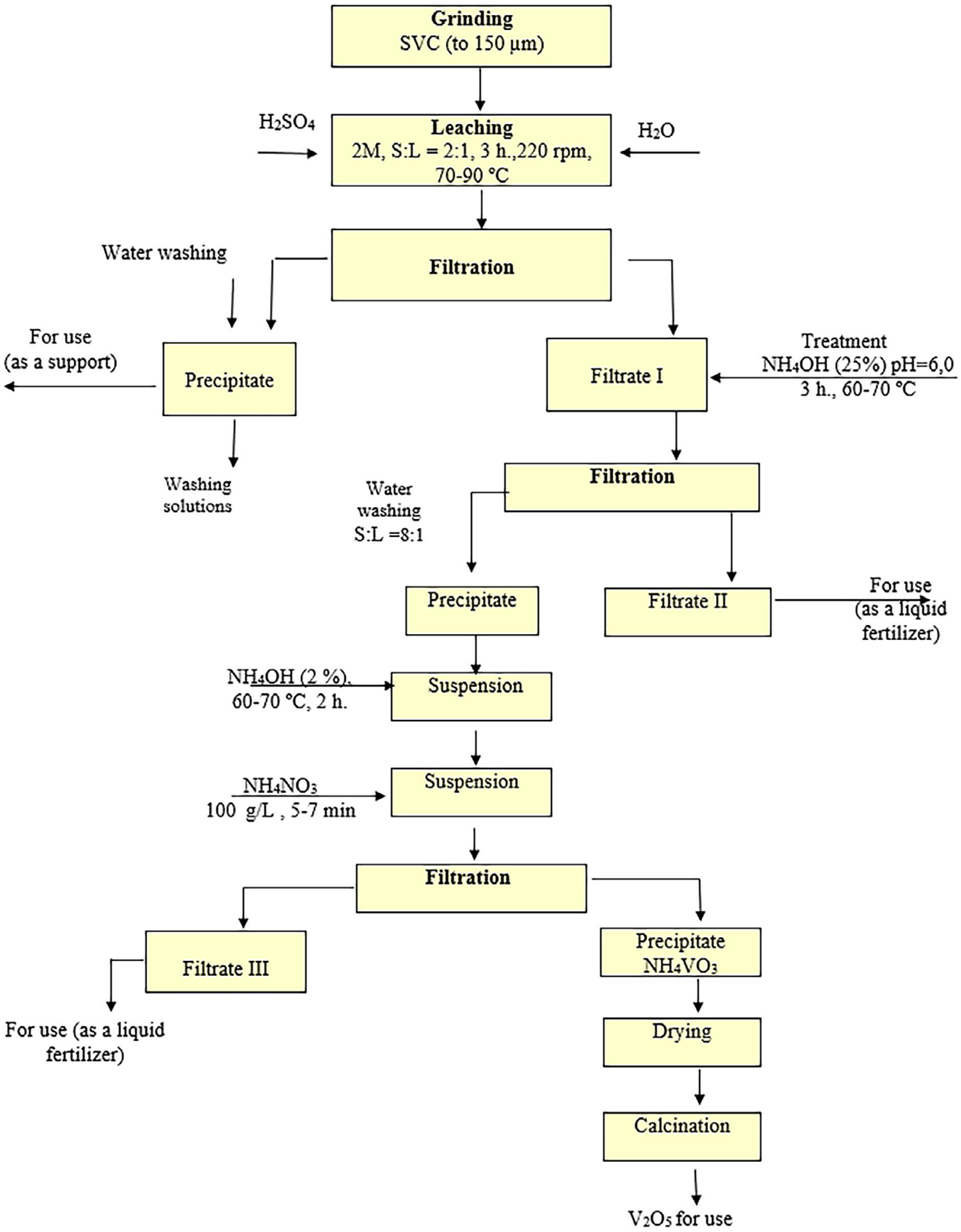

Figure 4. Schematic diagram of V_2O_5 production by salting-out method

vanadium content in solid waste from 5–8% to 0.5–2% through optimized acid leaching, while the resulting oxide impurities (SiO_2, Al_2O_3, Fe^{3+}) and cesium compounds are converted into catalytic supports. The two-stage ammonification of vanadium-containing solutions allows for the recovery of 97–98% of the target material in the form of high-purity V_2O_5 (98.2%), while simultaneously producing balanced N-P-K fertilizers (8–10% N, 2–3% P_2O_5, 1–2% K_2O). The implementation of a closed-loop reagent regeneration system, including ammonia capture with 99.5% efficiency, ensures compliance with stringent environmental regulations. Prospects for industrial application are linked to integration into existing sulfuric acid production facilities, the development of zero-waste water-recirculation systems, and further investigation of selective precipitation mechanisms. Future research should focus on the development of energy-saving thermolysis protocols and the optimization of process equipment for scaling up. The proposed approach offers a unique opportunity to transform hazardous waste into economically valuable products, setting a new benchmark in sustainable catalyst recycling.

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