Journal of Sustainable Metallurgy (2023) 9:1744–1758
https://doi.org/10.1007/s40831-023-00762-6

RESEARCH ARTICLE

Recovery of Vanadium and Nickel from a High $CaCO_3$ Containing Petroleum Coke Ash by Roasting and Acidic Leaching

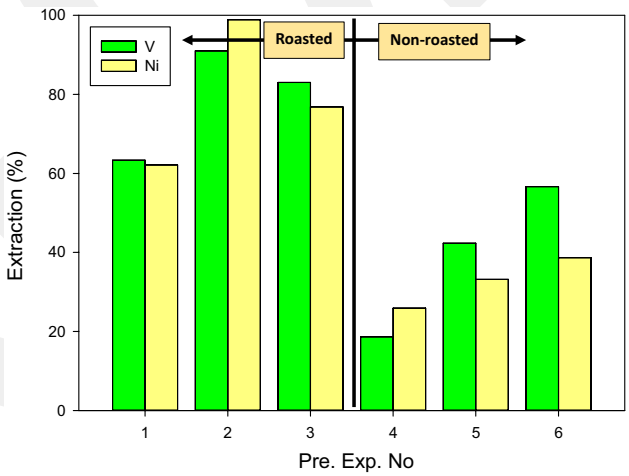
Ozgun Yurtseven[1] · Ahmedaljaali Ibrahim Idrees Ibrahim[1] · Soner Top[2] · Sait Kursunoglu[3] · Mahmut Altiner[1]

Received: 10 May 2023 / Accepted: 18 October 2023 / Published online: 8 November 2023

Abstract

In this study, it was aimed to extract vanadium (V) and nickel (Ni) from a petroleum coke ash (PCA) using a roasting process without additives, followed by leaching with sulfuric acid (H_2SO_4). The experiments were designed based on the Taguchi approach, taking into account the parameters of temperature, acid concentration, time, and solid ratio. Additional leaching tests were conducted on the non-roasted PCA for comparison, to assess the effect of roasting on the extractions of V and Ni. The results showed that no extra reducing agent was needed as the PCA contained high levels of $CaCO_3$, which could be used as a reducing agent during roasting. It was found that roasting was essential for high Ni extractions, but had no strong effects on V extractions. The Ni extraction was found to be between 13.3 and 80.8% for the non-roasted PCA and between 43.6 and 99.3% for the roasted PCA. The V extraction was between 36 and 97.9% for the non-roasted PCA and between 45.4 and 99.9% for the roasted PCA. The optimal leaching conditions were determined to be a sulfuric acid of 4.5 M, a solid ratio of 10%, a temperature of 75 °C, and a time of 75 min. In addition, it was determined that the leaching conditions had a great effect on the oxidation state of vanadium ions, and an increase in the acid concentration led to the formation of V^{3+} ions (green color) instead of VO^{2+} ions (blue color) in the pregnant leach solution. The final pregnant leach solution containing 1056.50 mg/L V, and 251.85 mg/L Ni was achieved with an extraction yields of > 98%. The experimental results were greatly fitted by the shrinking core model and the activation energy (E_a) for V and Ni was calculated as 3.60 and 4.01 kJ/mol, indicating that the leaching mechanism can be explained by the diffusion control model.

Graphical Abstract



Keywords Extraction · Vanadium · Nickel · Petroleum coke · Roasting · Acidic leaching

The contributing editor for this article was Uday Pal.

Extended author information available on the last page of the article

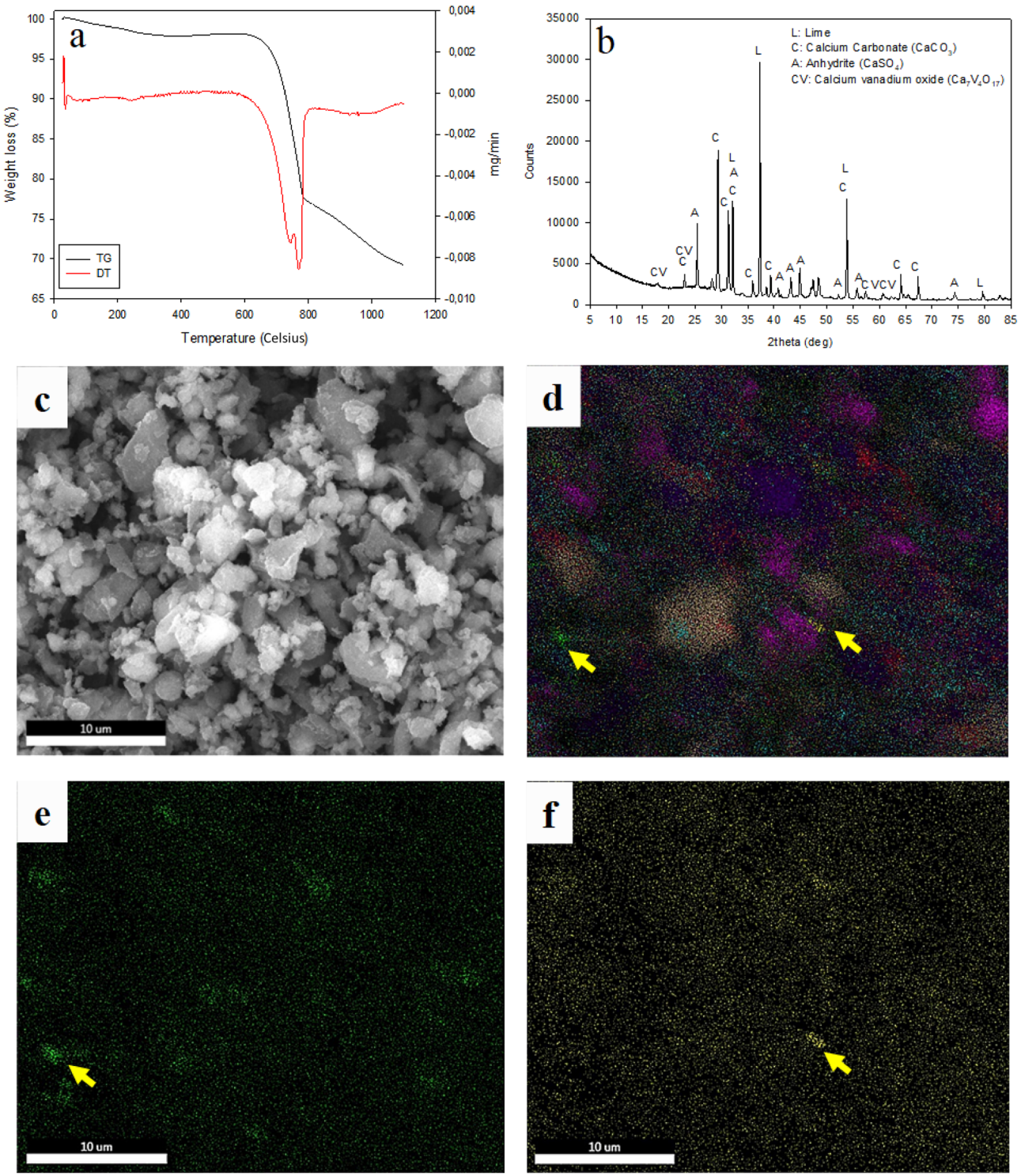


Fig. 1 **a** TG-DT curves of PCA, **b** XRD pattern of PCA (L: lime "96-900-6695", C: Calcium carbonate "01-086-0174", A: Anhydrite "96-500-0041", CV: Calcium vanadium oxide "00-028-0251"), **c** SEM image of PCA, and **d–f** Mapping picture of PCA (green: vanadium, yellow: nickel) (Color figure online)

Table 1 Preliminary leaching test conditions

Exp. no	Roasting	Temperature (°C)	Acid con. (M)	Solid ratio (%)	Time (min)
Pre-1	Yes	60	5	6.67	30
Pre-2	Yes	45	12	5	30
Pre-3	Yes	45	3.76	5	75
Pre-4	No	30	0.9	20	30
Pre-5	No	60	3.76	10	75
Pre-6	No	60	5	6.67	30

Introduction

Vanadium (V) and its compounds have received great attention for many years in various industries, including metallurgy, petrochemicals, electronics, defense, paint, and coatings, due to their exceptional physical and chemical properties, such as mechanics, electrochemistry, and magnetism [1]. Several governments, including Australia, South Korea, Canada, the USA, and Japan, as well as the European community, have declared V as a critical raw material in their reports due to its economic significance and supply risk. Previous researches have shown that nearly 88% of V was extracted globally from a titanomagnetite ore, which was the most significant source of this element [2, 3]. Additionally, V can be produced from various raw materials and secondary residues, such as vanadium slag [4, 5], stone coal [6, 7], petro-coke [8], fly ash [9], black-shale [10] and spent-catalyzer [11, 12].

There are several methods including NaCl salt roasting [13], Cl gas [14], Cl_2-N_2 gases [15]), alkaline leaching (NaOH [16]), Na_2CO_3 roasting + water leaching [17], roasting + NH_4HCO_3 leaching [18], ammonium sulfate roasting + diluted H_2SO_4 leaching [4], CaO roasting + H_2SO_4 leaching [19, 20], CaO roasting + NH_4HCO_3 leaching [20, 21], two stages leaching ($NH_4Cl + Na_2CO_3$) [22], a mixture of acid ($H_2SO_4 + HF + NaClO$) [10], ammonium sulfate leaching [23], H_2SO_4 pressure leaching [24]. Furthermore, the use of ultrasonic power [9] or microwave power [25] resulted in an increase in the extraction of V in the presence of organic or inorganic acid. When these powers were used together, V was dissolved in a NaOH solution with an extraction of over 80%, higher than the extraction of V (approximately 60%)

Table 2 Experimental parameters with their levels and the conditions conducted based on the Taguchi approach (L32, 2^1, 4^3)

Parameters			Level 1		Level 2		Level 3		Level 4
Temperature (°C)			< 50 °C		> 75 °C				
Acid concentration (M)			1		2.5		4.5		6
Time (min)			15		45		75		90
Solid ratio (%)			20		10		6.67		5
Exp. no	Temp	Acid con	Time	Solid ratio	Exp. no	Temp	Acid con	Time	Solid ratio
Experimental conditions (L32, 2^1, 4^3)									
1	1	1	15	20	17	2	1	15	5
2	1	1	45	10	18	2	1	45	6.67
3	1	1	75	6.67	19	2	1	75	10
4	1	1	90	5	20	2	1	90	20
5	1	2.5	15	20	21	2	2.5	15	5
6	1	2.5	45	10	22	2	2.5	45	6.67
7	1	2.5	75	6.67	23	2	2.5	75	10
8	1	2.5	90	5	24	2	2.5	90	20
9	1	4.5	15	10	25	2	4.5	15	6.67
10	1	4.5	45	20	26	2	4.5	45	5
11	1	4.5	75	5	27	2	4.5	75	20
12	1	4.5	90	6.67	28	2	4.5	90	10
13	1	6	15	10	29	2	6	15	6.67
14	1	6	45	20	30	2	6	45	5
15	1	6	75	5	31	2	6	75	20
16	1	6	90	6.67	32	2	6	90	10

obtained using only microwave power [26]. In the presence of Na salts (NaCl or Na_2SO_4), roasting may release corrosive gases (Cl_2, SO_2, or HCl) into the atmosphere [27]. However, Na_2CO_3 roasting followed by the water leaching process can be classified as eco-friendlier and more economical compared to other processes [28]. Calcification roasting with CaO or $CaCO_3$ is another option to produce Ca-vanadate, which is insoluble in water and can be dissolved in acid or alkaline methods [29]. V with high recovery from the leachate solution was achieved by precipitation using different chemicals such as urea [30], oxalic acid [31–33], ammonium chloride [34], glutamic acid [35], lysine [36], glycine [37]. These studies have shown that key factors affecting V precipitation include solution pH, temperature, time, and molar ratio of chemical: vanadium content.

In this study, it was aimed to extract V and Ni from a petroleum coke ash (PCA) generated in a lime-processing plant through agent-free roasting and H_2SO_4 leaching processes. The PCA contained high and remarkable concentrations of nickel (NiO: 0.32%) and V (V_2O_5:1.96%), making them economically important [38]. Ni is a strategic metal with increasing usage in batteries, and its price is expected to remain stable according to a previous study that forecasted Ni price using artificial neural network methods [39]. The roasting process in this study was unique as no roasting additive was used, which distinguishes it from previous studies that used Na_2CO_3 roasting followed by water leaching processes [40]. If Na_2CO_3 was used as a roasting agent in this study, the roasted product would contain a mixture of Ca- and Na-vanadates due to the high content of CaO and $CaCO_3$. The leaching behavior of Ca- and Na-vanadates in a H_2SO_4 solution would become complicated, so no roasting agent was added to prevent the formation of Ca- and Na-vanadates in the product after roasting. The extraction of V and Ni from the roasted PCA was investigated based on the Taguchi approach to understand the effect of the following leaching conditions: temperature, acid concentration, time, and solid ratio. For comparison, additional leaching tests were carried out on the non-roasted PCA.

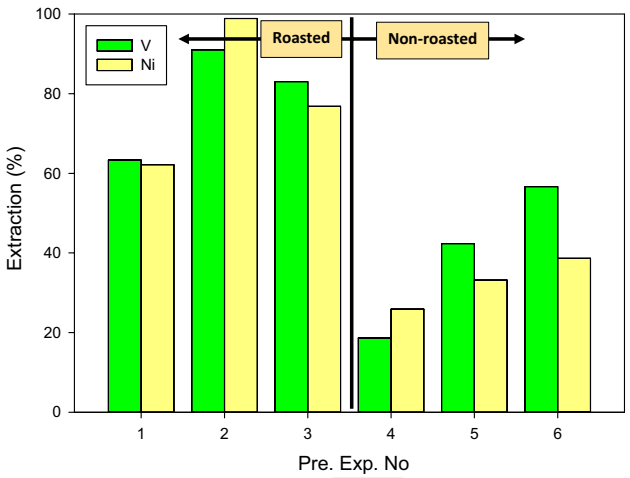


Fig. 2 The extraction of V and Ni from the PCA: preliminary leaching tests results

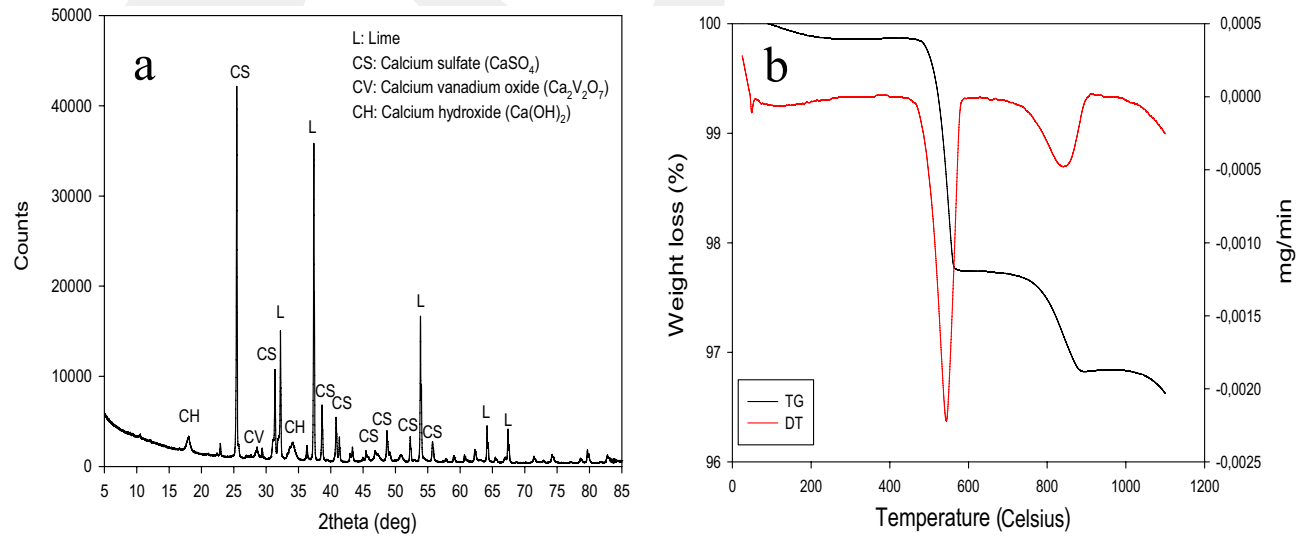


Fig. 3 **a** XRD pattern and **b** TG-DT curves of the roasted PCA [L: lime "01-077-2376", CS: calcium sulfate "01-080-0787", CV: Calcium vanadium oxide "01-072-2312", CH: calcium hydroxide "01-084-1271"]

Material and Methods

Material

The PCA used in this study was obtained from the over-flow of an air-cyclone separator in a lime-processing plant (Kaksan Kireç A.Ş) in Adana/Turkey. Its chemical composition was analyzed using X-ray Fluorescence (XRF, Panalytical MiniPal 4), Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-OES, Perkin Elmer), and Atomic Absorption Spectrometer (AAS, Perkin Elmer 900H). Results showed that the PCA had 15.60% SO_3, 48.35% CaO, 0.55% Fe_2O_3, 0.05% TiO_2, 0.95% K_2O, 0.01% MnO, 0.75% Al_2O_3, 0.18% Na_2O, 1.19% MgO, 1.96% V_2O_5, and 0.32% NiO with a LOI value of 31% (Fig. 1a), which was determined by a Thermogravimetric analyzer (Mettler Toledo, TGA 3 +). The X-ray pattern (XRD, Panalytical Empyrean) given in Fig. 1b showed that PCA was primarily composed of lime (CaO), calcium carbonate ($CaCO_3$), and anhydrite ($CaSO_4$) minerals together with calcium vanadium oxide ($Ca_7V_4O_{17}$) as a minor content. The presence of V and Ni in PCA was further confirmed by a scanning electron microscope (SEM, Fei Quanta 650), as shown in Fig. 1c–f. The chemicals used in this study were of analytical grade (Sigma Aldrich) and used without conducting any purification.

Methods

Preliminary leaching tests were conducted to determine the effective parameters for extracting V and Ni from non-roasted and roasted PCA. Roasting was performed at 1000 °C for 1 h with no additional agents, as previous studies have shown that the high $CaCO_3$ content in the PCA can serve as a natural reducing agent for the extraction of V [21, 29, 41]. The preliminary leaching tests conditions are summarized in Table 1.

Based on the preliminary results, a series of experiments were designed using the Taguchi approach (L32, 2^1, 4^3). The effects of temperature, acid concentration, time, and solid ratio on V and Ni extraction from both roasted and non-roasted PCA were studied. These extraction values were considered as output parameters, while the experimental conditions were used as input values in the Taguchi approach. The "Larger-to-better" option was selected. The results were calculated into S/N ratios using Eq. 1. Table 2 shows the experimental parameters and their levels.

$$\text{S/N ratio(larger-to-better)} = -10log_{10}\left(\frac{1}{n}\sum\frac{1}{h^2}\right) \quad (1)$$

The leaching tests were carried out in a 250 mL flat-bottomed three-necked jacketed reactor equipped with a glass spiral-type reflux condenser. The slurry was mixed using a magnetic stirrer (MTOPS, MS300HS). The desired temperature was controlled by a circulated water bath (JSR-22 T). At the end of the reaction time, the stirring was stopped and the slurry was centrifuged (ELEKTRO-MAG M415 M). The concentrations of V and Ni in the pregnant leach solution (PLS) were measured using ICP-OES and AAS, and the metal extraction values were calculated using Eq. 2.

$$Extraction(\%) = \frac{C_t \times V_f}{W_o \times H_o} \times 100 \quad (2)$$

where the C_t is the metal concentration in the PLS (mg/L), V_f is the volume of the PLS (L), W_o is the weight of the feeding sample (kg), H_o is the metal concentration in the feeding sample (mg/kg).

Considering all experimental results obtained in this study, the extraction mechanism of V and Ni from the slag at the optimal conditions was evaluated in a range of 40–90 °C for various times by the diffusion control model, which is one of the shrinking core models. The

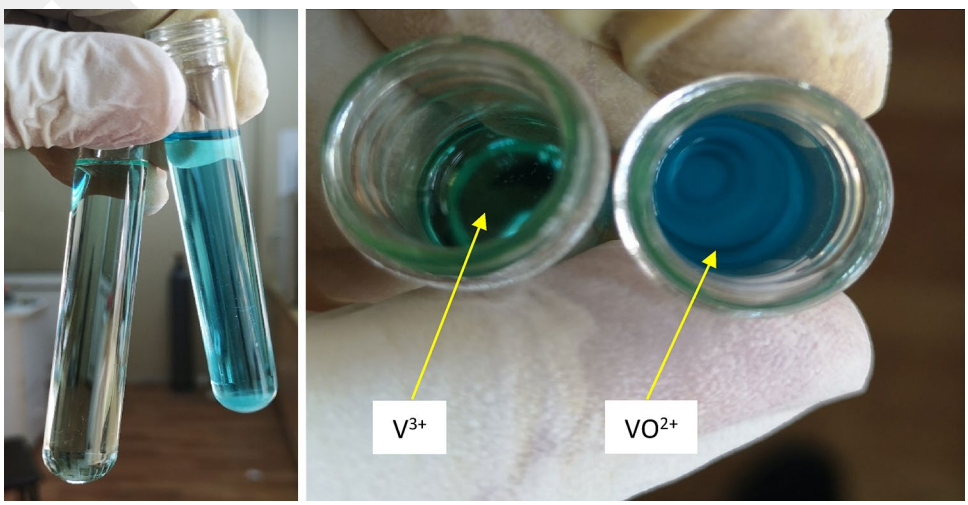


Fig. 4 The PLS with different colors obtained in this study [green solution; Exp. No: 16, blue solution; Exp. No: 27] (Color figure online)

Table 3 Experimental findings obtained in this study based on the Taguchi approach

Exp. no	Temp	Roasted		Non-roasted		Exp. no	Temp	Roasted		Non-roasted	
		V (%)	Ni (%)	V (%)	Ni (%)			V (%)	Ni (%)	V (%)	Ni (%)
1	Temperature: 50 °C	45.65	43.56	36.04	15.72	17	Temperature: 75 °C	78.96	66.30	86.55	44.81
2		67.39	50.13	65.26	23.46	18		96.70	80.75	74.23	53.06
3		69.80	60.46	69.09	22.23	19		95.25	87.12	85.89	51.84
4		70.47	74.78	73.83	20.33	20		95.99	86.75	66.53	32.92
5		52.65	72.56	55.55	16.68	21		90.15	87.96	80.88	46.77
6		69.26	84.66	68.54	22.45	22		99.30	91.81	75.47	61.61
7		75.83	81.01	73.97	22.42	23		98.14	91.85	77.82	57.02
8		79.71	94.93	75.73	21.21	24		97.03	87.71	80.60	56.23
9		78.63	83.16	75.23	17.91	25		95.61	96.98	80.98	57.70
10		85.68	87.20	69.96	13.28	26		99.49	94.91	79.67	64.94
11		90.48	96.34	81.85	19.15	27		97.18	98.37	91.16	68.61
12		98.23	91.85	97.89	19.72	28		98.20	99.25	88.88	65.31
13		90.91	82.13	65.53	19.06	29		90.55	88.32	90.11	61.59
14		85.77	89.28	73.36	23.59	30		99.98	94.72	92.27	80.78
15		97.51	95.40	72.76	21.05	31		97.73	98.89	89.84	62.51
16		98.48	93.81	97.74	26.09	32		94.51	98.88	88.07	67.59

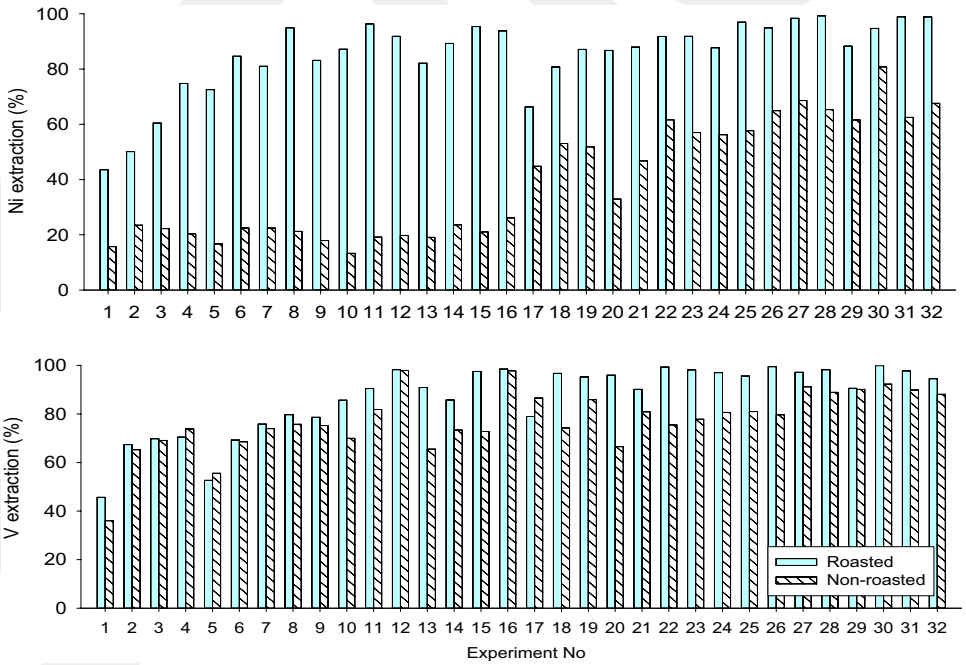


Fig. 5 Extractions of V and Ni from roasted and non-roasted PCA

following equation represents this mechanism. Afterwards, the activation energy (Ea) was calculated for V and Ni, respectively.

$$1-(1-\alpha)^{2/3}=k\times t \tag{3}$$

where $\boldsymbol{\alpha}$ is the fraction reacted (%), t is the reaction time (min), k is the reaction rate constant (min^{-1}).

Results and Discussion

Figure 2 shows the preliminary experimental findings obtained under different conditions to identify the range of parameters that could have a significant impact on the extraction of V and Ni from both roasted and non-roasted PCA. The V and Ni were dissolved with extractions ranging

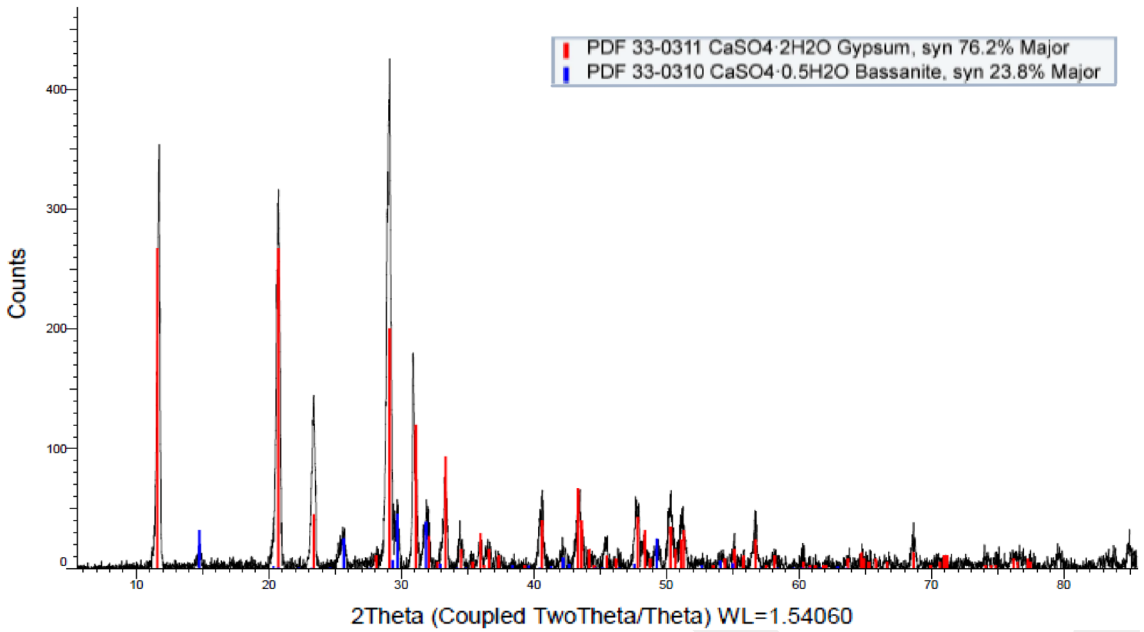


Fig. 6 XRD pattern of the leach residue

from 18.65 to 91.02% and 25.90 to 98.88%, respectively, based on the experimental conditions.

The XRD pattern after roasting the PCA at 1000 °C for 1 h, shown in Fig. 3a, revealed the disappearance of peaks representing the $CaCO_3$ mineral, while new peaks of CaO were observed. The roasted PCA was composed of calcium sulfate ($CaSO_4$, 74%), calcium hydroxide ($Ca(OH)_2$, 2%), lime (CaO, 17%), and calcium vanadium oxide ($Ca_2V_2O_7$, 7%) minerals. No Ni-bearing mineral was detected in the pattern, likely due to the detection limit of XRD. The loss on ignition (LOI) value was found to be around 3%, confirming the presence of $Ca(OH)_2$ in the roasted PCA (Fig. 3b). The possible reaction for V extraction using H_2SO_4 is shown in Eq. 4.

$$2Ca_2V_2O_7 + 8H_2SO_4 + O_2 \rightarrow 4(VO_2)SO_4 + 4CaSO_4 \downarrow + 8H_2O \quad (4)$$

The roasting of the PCA prior to leaching had a significant impact on the extraction of metals, especially Ni. For example, Ni was dissolved with an extraction of 62.16% from the roasted PCA with 5 M H_2SO_4 solution (Experiment No. Pre-1), which was almost double of the extraction from non-roasted PCA (Experiment No. Pre-6, ~ 38%). The extractions of V from the roasted and non-roasted PCA were 63.34% (Experiment No. Pre-1) and 56.65%, (Experiment No. Pre-6), respectively. It was obvious that the effect of roasting on the extraction of V was lower compared to Ni. In each leaching test, the solution temperature increased to a value higher than the desired temperature (approximately + 10 °C). This can be explained by the exothermic reaction of CaO in the PCA with acidic water that resulted in not only the formation of $Ca(OH)_2$ but also the release of heat energy, as described in Eq. 5.

$$CaO + H_2O \rightarrow Ca(OH)_2 + \text{heat}(\Delta H^o = -65.15kj) \quad (5)$$

It is recognized that there are four different species of V in solution: (1) purple "V^{2+}", (2) green "V^{3+}", (3) blue "VO^{2+}", and (4) yellow "$VO_2{}^{+}$". Previous research has emphasized the influence of pH on the oxidation state of V in solution [20, 42]. Herein, a decrease in solution pH due to the addition of H_2SO_4 resulted in obtaining PLS with different colors (shown in Fig. 4). For example, increasing the acid concentration from 4.5 M (Exp. No. 27) to 6 M (Exp. No. 16) turned the PLS color to green from blue. These reactions can be represented by Eqs. 6 and 7.

$$VO_2^+(aq) + 2H^+(aq) + e^- \rightarrow VO^{2+}(aq) + H_2O(aq) \quad \Delta G^\circ_{298} = -96.709kj(\textit{blue color}) \quad (6)$$

$$VO^{2+}(aq) + 2H^+(aq) + e^- \rightarrow V^{3+}(aq) + H_2O(aq)\Delta G^\circ_{298} = -91.621kj(\textit{green color}) \quad (7)$$

The experimental findings are listed in Table 3. Results indicated that the extraction of Ni was strongly influenced by the roasting process, as demonstrated in Fig. 5. Ni had a lower extraction (13.28–80.78%) when the PCA was not roasted, while the extraction of V ranged from 36.04 to 97.89% depending on the leaching conditions. Conducting

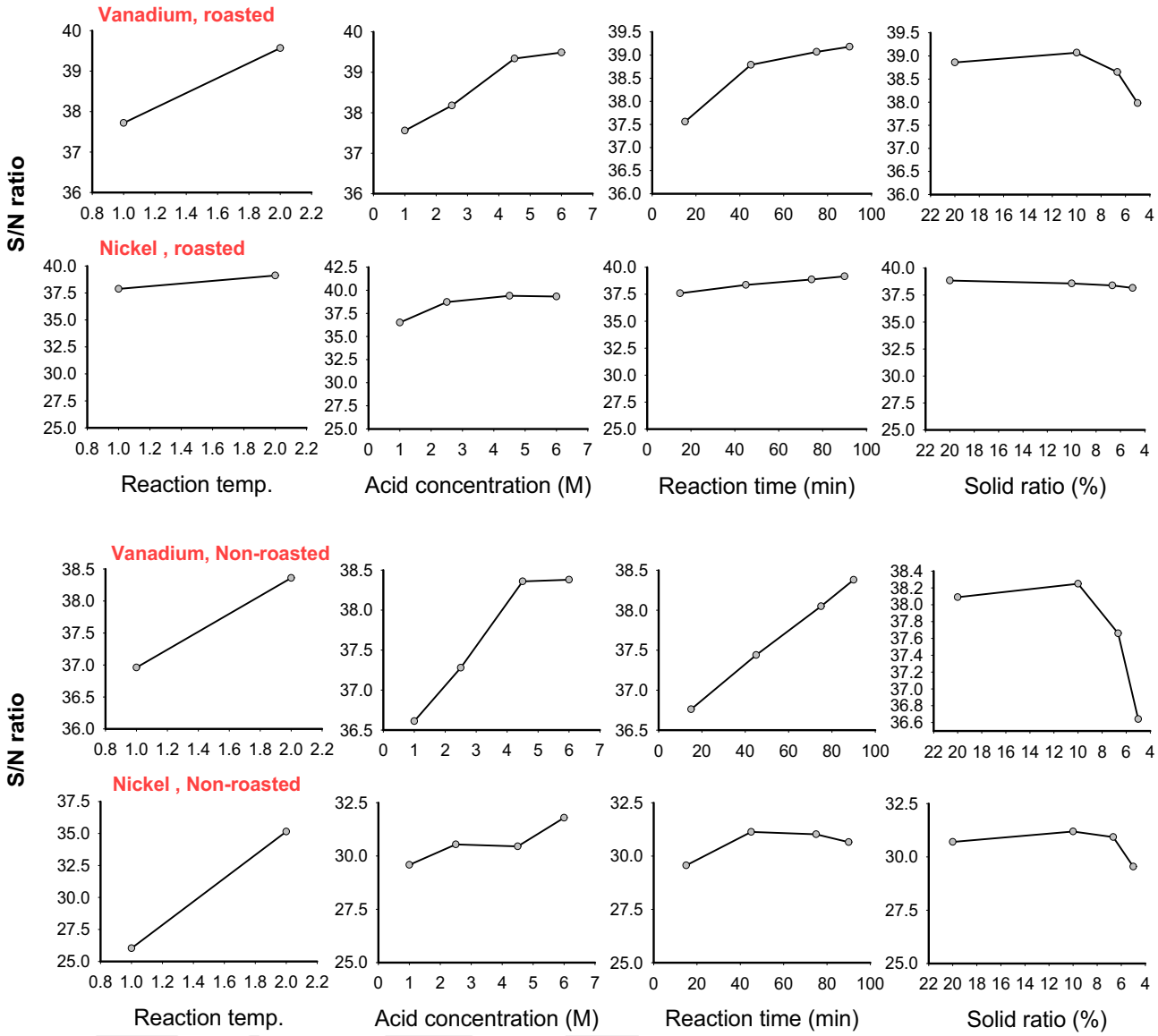


Fig. 7 Effects of leaching parameters on S/N ratios

the roasting process before the leaching increased the extractions of V and Ni. It was determined that the extractions of V and Ni from the roasted PCA were in a range of 45.43–99.98% and 43.56–99.25%, respectively.

During the leaching, the stirring of the solution was difficult at higher solid ratios (> 10%) and lower acid concentrations (≤ 2.5 M) due to the formation of new phases in the leach residue, which was composed of anhydrite ($CaSO_4$) and basanite ($CaSO_4 \cdot 0.5H_2O$) minerals (Fig. 6).

However, it was observed that an increase in the acid concentration and temperature made the stirring of the solution better even though $CaSO_4$ was formed under each experimental condition. The values of S/N ratio calculated from the experimental findings are given in Fig. 7. The effects of leaching conditions can be clearly evaluated from these graphs based on the highest S/N ratio. As such, the order of the effect of leaching conditions (from the highest to the lowest) on the extraction of Ni from the roasted PCA was: (1) acid concentration, (2) reaction time, (3) temperature, and (4) solid ratio. The effect of leaching conditions on the extraction of V from the roasted PCA was in the following order (from the highest to the lowest): (1) temperature, (2) acid concentration, (3) reaction time, and (4) solid ratio. Although the solid ratio had no effect on the leaching of V and Ni from the roasted PCA, the optimal solid ratio was selected as 10% to avoid stirring difficulties during the leaching caused by the formation of $CaSO_4$.

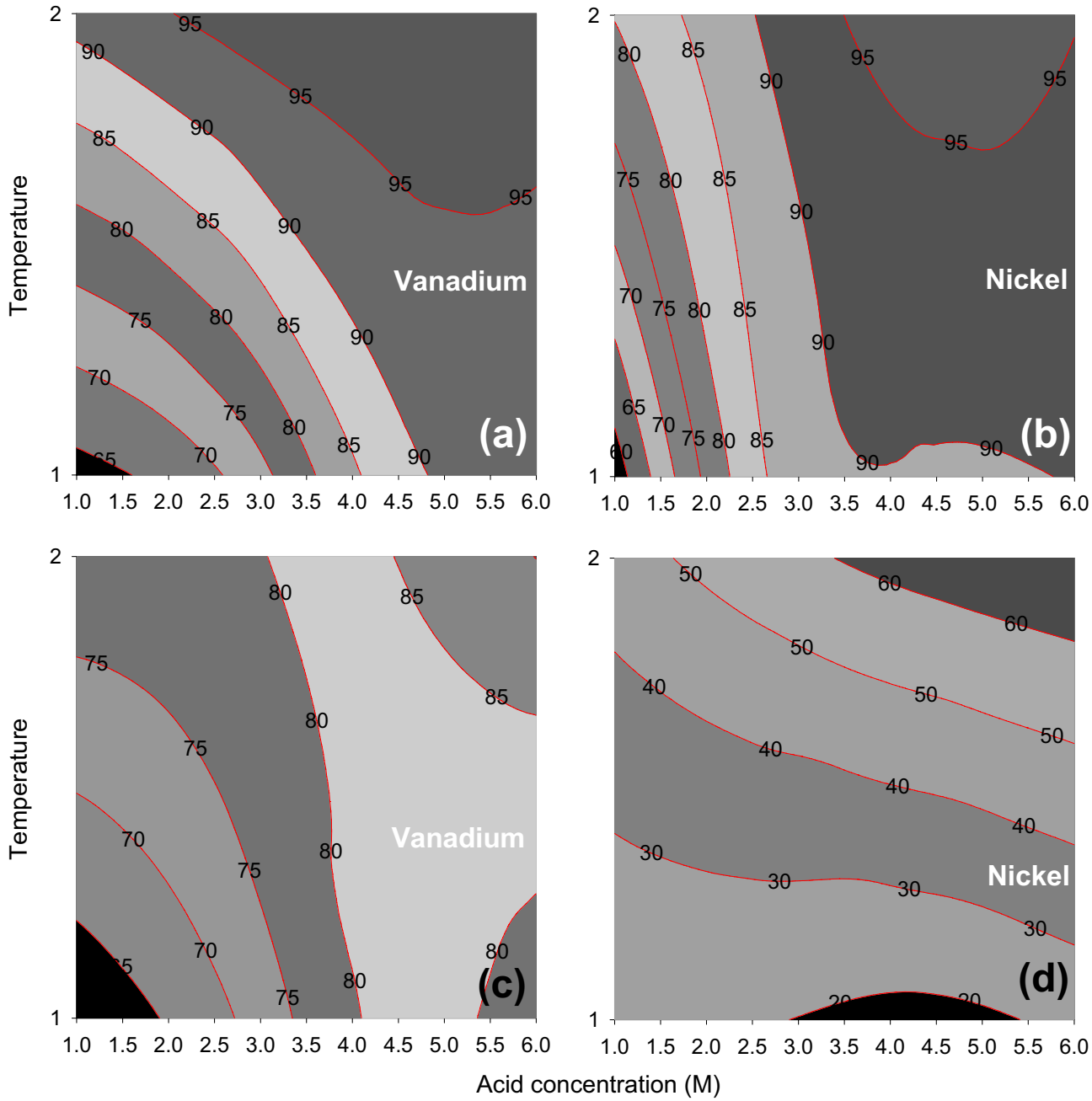


Fig. 8 Extractions of **a** V and **b** Ni from the roasted PCA, **c** V, and **d** Ni from the non-roasted PCA

Contour plots shown in Fig. 8 indicate that the roasting process had little effect on the extraction of V from the PCA. There was a negligible difference in V extraction before and after roasting. The extractions of V were high both in the non-roasted PCA (Fig. 8a) and roasted PCA (Fig. 8c). Figure 8b and d shows that the leaching behavior of Ni was strongly influenced by the roasting process. The use of the roasting process makes Ni dissolution with high extractions. For example, Ni with an extraction over 90% was dissolved from the roasted PCA in 3 M H_2SO_4 solution under all reaction temperatures, whereas the extraction of Ni from the non-roasted PCA was below 70% under all conditions.

The interaction plot in Fig. 9 reveals the relationship between parameters and the extraction recoveries of V and Ni. According to previous research [43], parallel plots show a weaker interaction between parameters, while intersecting plots suggest that interactions exist. It is clearly seen in Fig. 9 that the interaction plot clearly demonstrates positive interactions between temperature and the other parameters

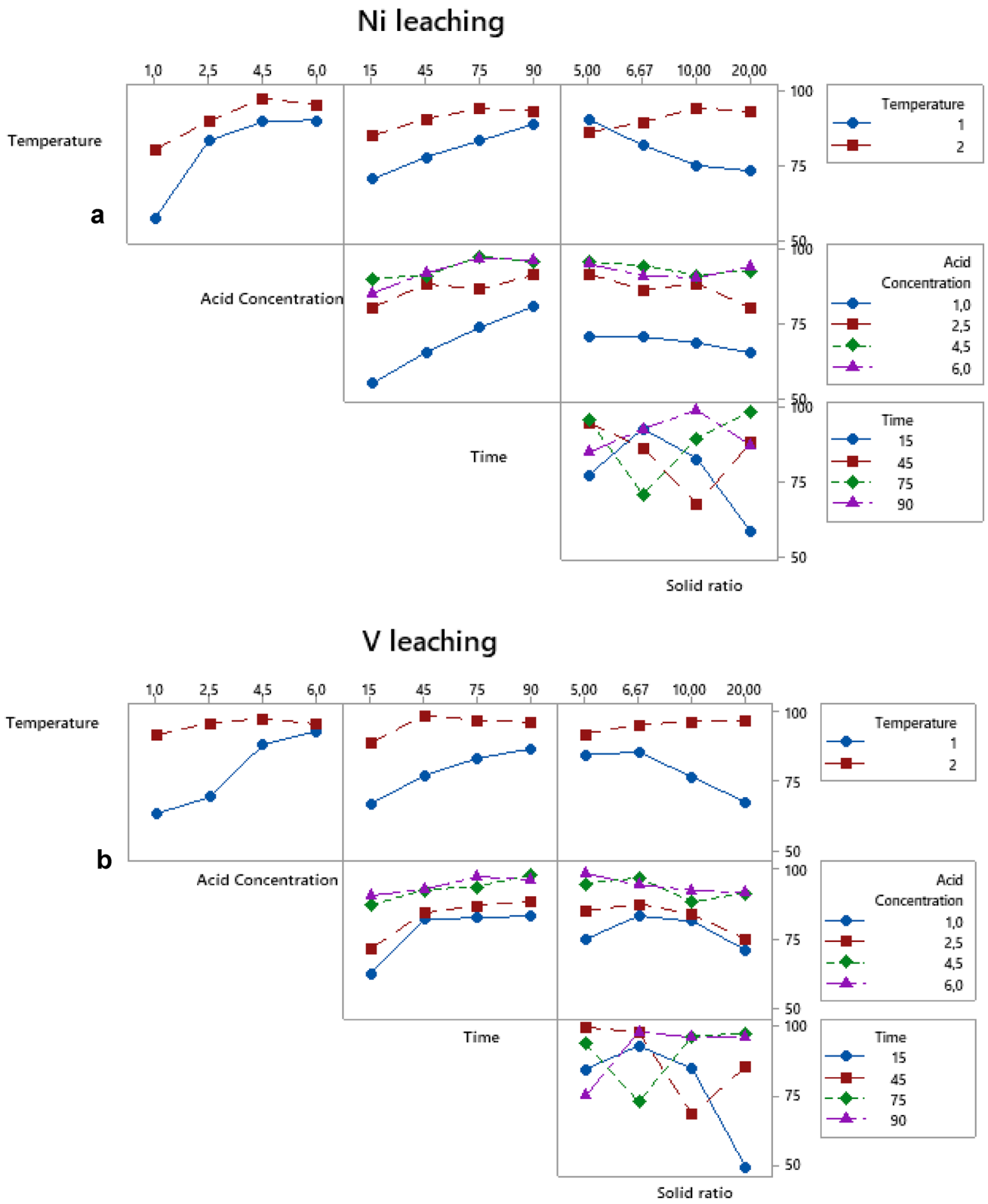


Fig. 9 Interaction plots showing the extraction behaviors of **a** Ni and **b** V from the roasted PCA

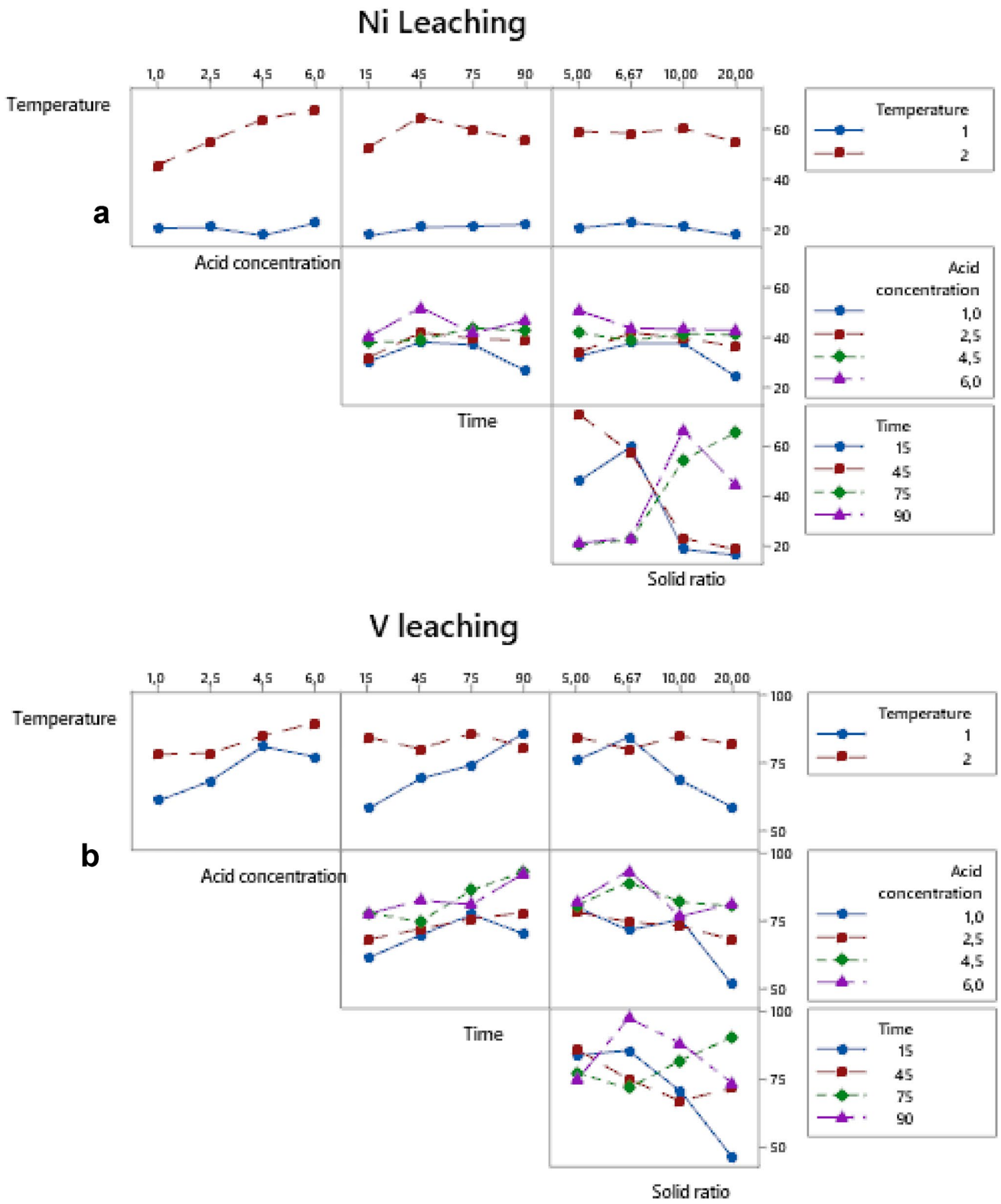


Fig. 10 Interaction plots showing the extraction behaviors of **a** Ni and **b** V from the non-roasted PCA

Table 4 The elemental concentration of the final PLS

Element	Concentration (mg/L)	Extraction (%)
V	1056.50	> 98
Ni	251.85	> 99
Al	354.34	89.18
Fe	366.16	95.06
K	739.50	93.72
Mg	706.52	98.39
Ca	433.51	1.25

(acid concentration, reaction time, and solid ratio). However, there was no notable interaction between reaction time and solid ratio as the line is parallel to the x-axis. Similar trends in lines were observed when the leaching behaviors of V and Ni of the roasted PCA were evaluated as shown in Fig. 10.

The interaction plots for the non-roasted PCA are presented in Fig. 10.

Finally, V and Ni ions were recovered from the roasted PCA with extractions of > 99% under the optimal leaching conditions, which are as follows: a sulfuric acid of 4.5 M, a solid ratio of 10%, a temperature of 75 °C, and a time of 75 min. The concentrations of elements in the final PLS are summarized in Table 4. The PLS contained high undesirable elements (Fe, Al, Mg, and K) but their precipitation behaviors at different pH are different and it is possible to precipitate desired elements in hydroxide form with the addition of different bases. Furthermore, solvent extraction can be a good option for the separation process.

The extraction behavior of V and Ni from the slag was further investigated in a range of 40–0 °C and the obtained results were examined by the diffusion control model, as shown in Fig. 11a and b. It is clear that the diffusion control model was greatly fitted to the obtained experimental results.

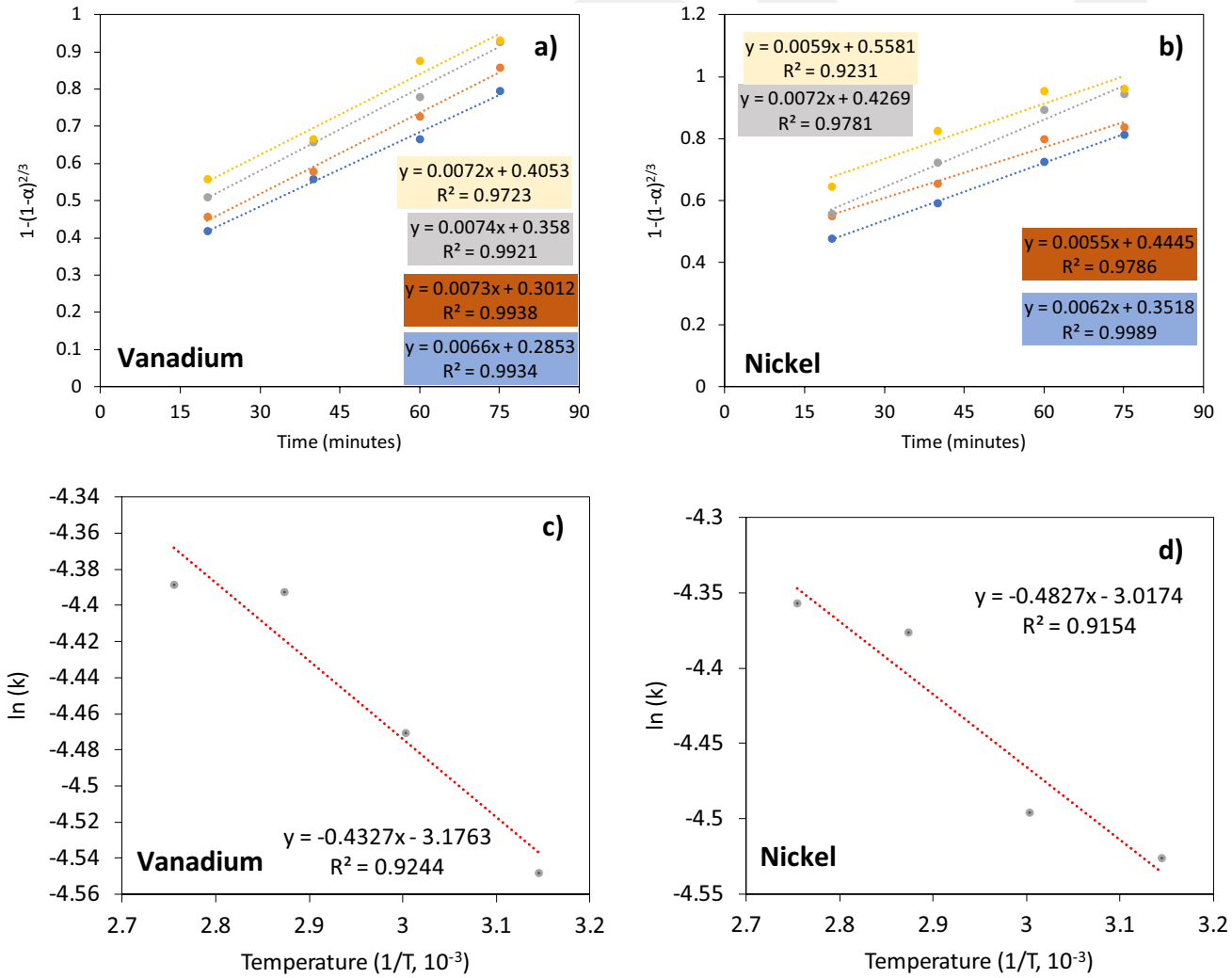


Fig. 11 The fitting results of leaching by the diffusion control. **a** vanadium leaching, **b** nickel leaching, **c** and **d** lnk 1000/T

The classification of the reaction mechanism was strongly dependent on the temperature and activation (E_a) value [44]. The mechanism of the leaching process was identified as follows: (i) diffusion control, If E_a lower than 20 kJ/mol, (ii) mixed control, If E_a in a range of 20–42 kJ/mol, and (iii) chemical control, if E_a higher than 42 kJ/mol. The Ea value for V and Ni leaching was found to be 3.60 and 4.01 kJ/mol, respectively. These values are much lower compared to those of previous studies in which spent catalysts, sintered nickel alloy, nickel matte, and laterite ores were leached with different reagents [45–50]. In a different study, microwave-assisted leaching was applied to spent catalysts to recover V and Ni, simultaneously. The activation energy of V and Ni was calculated to be 3.28 and 34.41 kJ/mol, respectively [51]. It was understood from these studies that the chemical content and mineralogical composition of the material used as Ni or V source played a key role in the leaching mechanism. Therefore, Ea values may be calculated in a range of 3–50 kJ/mol depending on the properties of the raw material.

Finally, it was aimed herein to show such tailings having good potential as a raw material for Ni and V, which are highly demanded in many industries. Ni content in the tailings may be too low to apply the beneficiation process based on the Ni current price, but we believe that the demand for Ni will continue to rise as it is used in the production of Li-based batteries. Also, previous studies indicated that Ni consumption in battery production increase to 37% at the end of 2030 and its uses will replace stainless steel in the battery production on 2040 [52, 53]. Moreover, the presence of V in the tailings makes this study valuable. Also, the residue after leaching was mainly composed of gypsum, anhydrite or $CaSO_4 0.5H_2O$ (as shown in Fig. 6) that can be used as an additive in the production of concrete [54] and therefore zero waste was obtained after the process conducted in this study. Moreover, CO_2 gas released during the roasting can be captured and used in the production of precipitated calcium carbonate (PCC) particles from gypsum wastes in the presence of various alkali sources [55].

Conclusions

The extraction of V and Ni from a petroleum coke ash (PCA) by pyro- and hydro-metallurgical methods, including roasting in the absence of additive and H_2SO_4 leaching, was investigated. Furthermore, the obtained results were fitted by the shrinking core model. Results showed that $CaCO_3$ in the PCA served as the roasting agent, negating the need for additional agents. Moreover, additional leaching tests were carried out on the non-roasted PCA for comparison. Results indicated that V extractions were similar between the roasted and non-roasted PCA. However, it was only possible to dissolve Ni with a high extraction (> 99%) in a H_2SO_4 solution from the PCA after the roasting process. The leaching process was designed based on the Taguchi approach, which showed that the parameters could be ordered as follows: acid concentration > reaction time > temperature > solid ratio and temperature > acid concentration > reaction time > solid ratio for the Ni and V extractions, respectively. It was observed that increases in acid concentration changed the color of PLS due to the formation of V ions with different oxidation states. The optimum leaching conditions were found to be a sulfuric acid of 4.5 M, a solid ratio of 10%, a temperature of 75 °C, and a time of 60 min for the leaching process resulting in > 98% V and Ni extraction from the roasted PCA. The final PLS contained 1056.56 mg/L V and 251.85 mg/L Ni. Moreover, the Ea value found to be 3.60 kJ/mol for V and 4.01 kJ/mol for Ni, respectively. These value showed that the leaching mechanism can be explained by the diffusion control model. In a follow up study, the separation and purification of V and Ni from the PLS will be investigated by solvent extraction and precipitation methods.

Acknowledgements The study was the part of the first author's master thesis and financially supported by Cukurova University [Project ID: FYL-2022-14905].

Funding This study is supported by Cukurova University [Project ID: FYL-2022-14905].

Conflict of interest The authors declare that they have no conflict of interest.

References

1. Wang Z-H, Zheng S-L, Wang S-N, Biao L, Wang D-W, Hao D et al (2014) Research and prospect on extraction of vanadium from vanadium slag by liquid oxidation technologies. Trans Nonferrous Metals Soc China 24(5):1273–1288
2. Chen D, Zhao H, Hu G, Qi T, Yu H, Zhang G et al (2015) An extraction process to recover vanadium from low-grade vanadium-bearing titanomagnetite. J Hazard Mater 294:35–40
3. Li W, Guo R, Li Y, Dong Z (2022) Recovery of vanadium from direct acid leaching solutions of weathered crust vanadium-titanium magnetite via solvent extraction with N235. Hydrometallurgy 213:105913
4. Zhang G, Luo D, Deng C, Lv L, Liang B, Li C (2018) Simultaneous extraction of vanadium and titanium from vanadium slag using ammonium sulfate roasting-leaching process. J Alloy Compd 742:504–511
5. Mirazimi SMJ, Rashchi F, Saba M (2015) A new approach for direct leaching of vanadium from LD converter slag. Chem Eng Res Des 94:131–140
6. Yuan S, He M, Wang R, Jin Y, Li Y (2022) Multistage suspension roasting of refractory stone coal: enhanced extraction based on decarburization and vanadium oxidation. Powder Technol 405.
7. Tang Z, Zhou Z, Jin J, Sun Y, Han Y, Zhang Y (2022) Vanadium extraction from stone coal using a novel two-stage roasting technology. Fuel 321:124031
8. Litvinenko VS, Kudinova AA, Kondrasheva NK, Rudko VA (2021) Influence of leaching parameters on the vanadium extraction from petroleum coke. E3S Web Conf 266:08002

9. Rahimi G, Rastegar SO, Rahmani Chianeh F, Gu T (2020) Ultrasound-assisted leaching of vanadium from fly ash using lemon juice organic acids. RSC Adv 10(3):1685–1696
10. Li M, Wei C, Fan G, Wu H, Li C, Li X (2010) Acid leaching of black shale for the extraction of vanadium. Int J Miner Process 95(1–4):62–67
11. Erust C, Akcil A, Bedelova Z, Anarbekov K, Baikonurova A, Tuncuk A (2016) Recovery of vanadium from spent catalysts of sulfuric acid plant by using inorganic and organic acids: laboratory and semi-pilot tests. Waste Manag 49:455–461
12. Liu Q, Quan S, Liu Z, Liu Q (2022) Leaching of vanadium and tungsten from spent V2O5-WO3/TiO2 catalyst by ionic liquids. Hydrometallurgy 213:105938
13. Shlewit H, Alibrahim M (2006) Extraction of sulfur and vanadium from petroleum coke by means of salt-roasting treatment. Fuel 85(5–6):878–880
14. Murase K, Nishikawa K-I, Ozaki T, Machida K-I, Adachi G-Y, Suda T (1998) Recovery of vanadium, nickel and magnesium from a fly ash of bitumen-in-water emulsion by chlorination and chemical transport. J Alloys Compd 264(1–2):151–156
15. Du G, Fan C, Yang H, Zhu Q (2019) Selective extraction of vanadium from pre-oxidized vanadium slag by carbochlorination in fluidized bed reactor. J Clean Prod 237:117765
16. Zhang Y, Yang L (2015) Alkali leaching of vanadium from petroleum coke and kinetics analysis. Int J Environ Eng 7(1):90–100
17. Jung M, Mishra B (2017) Vanadium recovery from oil fly ash by carbon removal and roast-leach process. JOM 70(2):168–172
18. Li M, Liu B, Zheng S, Wang S, Du H, Dreisinger DB et al (2017) A cleaner vanadium extraction method featuring non-salt roasting and ammonium bicarbonate leaching. J Clean Prod 149:206–217
19. Yang Z, Li H-Y, Yin X-C, Yan Z-M, Yan X-M, Xie B (2014) Leaching kinetics of calcification roasted vanadium slag with high CaO content by sulfuric acid. Int J Miner Process 133:105–111
20. Hui X, Zhang J, Liang Y, Chang Y, Zhang W, Zhang G (2022) Comparison and evaluation of vanadium extraction from the calcification roasted vanadium slag with carbonation leaching and sulfuric acid leaching. Separation Purif Technol 297:121466
21. Li H-Y, Wang K, Hua W-H, Yang Z, Zhou W, Xie B (2016) Selective leaching of vanadium in calcification-roasted vanadium slag by ammonium carbonate. Hydrometallurgy 160:18–25
22. Akita S, Maeda T, Takeuchi H (1995) Recovery of vanadium and nickel in fly ash from heavy oil. J Chem Tech Biotechnol 62:345–350
23. Muthukumar K, Patel KM, Mohapatra D, Padh B, Reddy BR (2020) Selective recovery of vanadium as AMV from calcium vanadate sludge by direct AS leaching process: an industrial approach. Waste Manag 102:815–822
24. Li M, Wei C, Qiu S, Zhou X, Li C, Deng Z (2010) Kinetics of vanadium dissolution from black shale in pressure acid leaching. Hydrometallurgy 104(2):193–200
25. Zhang YL, Tian XG, Huang WQ (2015) Physical chemical properties of leachate in the process of improving vanadium leaching from petroleum coke: part I. Pet Sci Technol 33(20):1728–1734
26. Wang Z, Chen X, Chu W, Zhang YL (2019) Microwave-ultrasonication method for leaching vanadium from petroleum coke. Pet Sci Technol 37(14):1634–1639
27. Zhang Y-M, Bao S-X, Liu T, Chen T-J, Huang J (2011) The technology of extracting vanadium from stone coal in China: history, current status and future prospects. Hydrometallurgy 109(1–2):116–124
28. Chen D, Zhao L, Liu Y, Qi T, Wang J, Wang L (2013) A novel process for recovery of iron, titanium, and vanadium from titanomagnetite concentrates: NaOH molten salt roasting and water leaching processes. J Hazard Mater 244:588–595
29. Li X-S, Xie B (2012) Extraction of vanadium from high calcium vanadium slag using direct roasting and soda leaching. Int J Miner Metall Mater 19(7):595–601
30. Peng H, Yang L, Wang L, Guo J, Li B (2019) Recovery of vanadium with urea in acidic medium. Environ Chem Lett 17(4):1867–1871
31. Kang Q, Zhang Y, Bao S (2019) An environmentally friendly hydrothermal method of vanadium precipitation with the application of oxalic acid. Hydrometallurgy 185:125–132
32. Chen X, Zhang J, Yan B (2021) Hydrothermal precipitation of V3+ ions in V-bearing oxalic acid leachate to prepare V2O3. Hydrometallurgy 205:105734
33. Chen X, Zhang J, Yan B (2021) A clean method of precipitation vanadium from the vanadium bearing oxalic acid leaching solution. Miner Eng 165:106864
34. Akbari N, Pourmatin A, Niknafs D, Almasian MR, Mosavi SE, Ghorbanian SA et al (2021) Recovery of vanadium by ammonium chloride precipitation method using response surface methodology. Analy Methods Environ Chem J 4(04):64–77
35. Peng H, Qiu H, Wang C, Yuan B, Huang H, Li B (2021) Thermodynamic and kinetic studies on adsorption of vanadium with glutamic acid. ACS Omega 6(33):21563–21570
36. Peng H, Tang D, Liao M, Wu B, Wu Y, Fan X et al (2022) Efficient recovery of vanadium using lysine. J Water Process Eng 49:103030
37. Peng H, Zhang C, Hao Z, Jiang S, Guo J, Huang H et al (2022) Vanadium recovery by glycine precipitation. Environ Chem Lett 20(3):1569–1575
38. Armas CED (1980) The extraction of vanadium and nickel from petroleum cokes. Doctor of Philosophy of the University of London, PhD:258.
39. Ozdemir AC, Buluş K, Zor K (2022) Medium-to long-term nickel price forecasting using LSTM and GRU networks. Resour Policy 78:102906
40. Goncharov KV, Kashekov DY, Sadykhov GB, Olyunina TV (2020) Processing of fuel oil ash from thermal power plant with extraction of vanadium and nickel. Non-ferrous Metals. https://doi.org/10.17580/nfm.2020.01.01
41. Peng H, Li B, Shi W, Liu Z (2022) Efficient recovery of vanadium from high-chromium vanadium slag with calcium-roasting acidic leaching. Minerals 12(2):160
42. Xiang J, Huang Q, Lv X, Bai C (2017) Kinetic study on the leaching of vanadium-bearing converter slag with dilute sulfuric acid. Proceedings of the Pan American Materials Congress. Springer, pp 547–556
43. Kurnia JC, Haryoko LAF, Taufiqurrahman I, Chen L, Jiang L, Sasmito AP (2022) Optimization of an innovative hybrid thermal energy storage with phase change material (PCM) wall insulator utilizing Taguchi method. J Energy Storage 49:104067
44. Brahim JA, Hak SA, Achiou B, Boulif R, Beniazza R, Benhida R (2022) Kinetics and mechanisms of leaching of rare earth elements from secondary resources. Miner Eng 177:107351
45. Nikiforova A, Kozhura O, Pasenko O (2016) Leaching of vanadium by sulfur dioxide from spent catalysts for sulfuric acid production. Hydrometallurgy 164:31–37
46. Feng QM, Shao YH, Le-min O, Zhang GF, Lu YP (2009) Kinetics of nickel leaching from roasting-dissolving residue of spent catalyst with sulfuric acid. J Cent South Univ Technol 16:410–415
47. Wang Y, Chang X, Chen M, Qin W, Han J (2023) Effective extraction of nickel and cobalt from sintered nickel alloy via reduction roasting and leaching. Miner Eng 203:108336
48. Liu X, Huang J, Zhao Z, Chen X, Li J, He L, Sun F (2023) Nickel leaching kinetics of high-grade nickel matte with sulfuric acid under atmospheric pressure. Hydrometallurgy 215:105987

49. Borda J, Torres R (2023) Effect of pretreatments to improve nickel leaching from laterites in carboxylic media: mechanism and kinetic model. S Afr J Chem Eng 46:12–21
50. Yunita FE Mubarok MZ (2021) Nickel leaching from laterite ores by combination of organic and sulfuric acid. In AIP Conference Proceedings, Vol. 2382, No. 1. AIP Publishing.
51. Zheng D, Zhang Y, Liu T, Huang J, Cai Z, Zhang R (2022) Research on leaching of V and Ni in spent FCC catalyst using oxalic acid/H_2O_2 under microwave-assisted conditions. Minerals 12:834
52. Bai Y, Zhang T, Zhai Y, Jia Y, Ren K, Hong J (2022) Strategies for improving the environmental performance of nickel production in China: insight into a life cycle assessment. J Environ Manage 312:114949
53. Xiao T, Mu W, Shi S, Xin H, Xu X, Cheng H, Luo S, Zhai Y (2021) Simultaneous extraction of nickel, copper, and cobalt from low–grade nickel matte by oxidative sulfation roasting-water leaching process. Miner Eng 174:107254
54. Xue W, Twenda C, Alam MS, Xu L, Wang Z (2023) Experimental study on seepage characteristics and stress sensitivity of desulfurization gypsum based concrete under triaxial stress. J Market Res 24:6425–6437
55. Altiner M, Top S, Kaymakoğlu B (2021) Ultrasonic-assisted production of precipitated calcium carbonate particles from desulfurization gypsum. Ultrason Sonochem 72:105421

Authors and Affiliations

Ozgun Yurtseven[1] · Ahmedaljaali Ibrahim Idrees Ibrahim[1] · Soner Top[2] · Sait Kursunoglu[3] · Mahmut Altiner[1]

✉ Mahmut Altiner
maltiner@cu.edu.tr

[1] Division of Mineral Processing, Department of Mining Engineering, Cukurova University, 01330 Adana, Turkey

[2] Department of Engineering Science, Abdullah Gul University, 38100 Kayseri, Turkey

[3] Department of Petroleum and Natural Gas Engineering, Batman University, 72100 Batman, Turkey