

SELECTIVE DISSOLUTION OF VANADIUM (V) FROM SPENT PETROLEUM CATALYSTS BY OXALIC ACID SOLUTION

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Abstract

The spent petroleum catalyst contains a significant amount of vanadium and its recovery is essential. Oxalic acid has good metal selectivity due to its complex properties. Leaching experiments with oxalic acid were done to investigate the dissolution behavior of vanadium from spent petroleum catalyst by varying leaching conditions, such as temperature, concentration, pulp density, stirring speed and time. Vanadium and molybdenum were selectively dissolved by oxalic acid from the spent catalysts, while the leaching percentage of other metals was very low. However, the leaching percentage of vanadium by oxalic acid was lower than 45% in most of the leaching conditions. Our results provide some basic information on the dissolution of valuable metals from the spent catalyst by using organic acid.

Keywords: Spent petroleum catalyst; Vanadium; Oxalic acid; Leaching

1. Introduction

Vanadium (V) is a strategic metal which has been widely employed in many applications such as catalysts, steel additive, and manufacturing of alloys [1, 2]. Since the demand for vanadium has increased steadily, its recovery from diverse resources like spent catalysts, oil fly ash, petroleum coke, or black shale is necessary [3–5]. The spent catalysts resulted from the refining of crude petroleum contain a significant amount of vanadium together with other valuable metals [6]. After several cycle processes, the catalyst materials become contaminated and deactivated with time due to the accumulation of some impurities on their surface [7]. They are discarded as solid wastes and have been considered as hazardous waste materials [6]. Considering the environmental burden and recovery of valuable metals, the recovery of valuable metals from the spent catalysts is desirable. Therefore, many processes have been developed to recover valuable metals from the spent catalysts [1, 6, 7].

Both hydro- and pyrometallurgical treatments are generally employed for the extraction of vanadium from the ores or secondary resources. In hydrometallurgy, vanadium is dissolved by leaching with either alkaline (NaOH, KOH, Na₂CO₃, NH₃) or acidic (H₂SO₄, HNO₃, HCl, HF, H₂C₂O₄) solutions, while pyrometallurgy uses the heat treatment at high

temperature like roasting or smelting to extract vanadium [3–5, 8–12]. In hydrometallurgical processes, vanadium is separated from the leaching solutions by several unit operations such as solvent extraction, precipitation, and ion exchange. Many studies have been done for the leaching of vanadium from spent catalyst by employing the above chemical reagents. However, the dissolution behavior of vanadium in each leaching solution is different and strongly depends on the leaching conditions, such as vanadium concentration, the presence of other metallic components, as well as the type of waste materials.

In general, the leaching percentage of vanadium from the spent catalysts by inorganic acids as well as alkalis is high [1, 6, 13]. However, other metal ions can be leached together with vanadium, which makes it difficult to separate vanadium from the leaching solution. In some cases, the spent catalyst contains large amounts of vanadium which results in poor solubility of vanadium by inorganic acid leaching like H₂SO₄ [14]. In order to improve the leaching efficiency of vanadium, the addition of additive agents has been employed. When vanadium exists as a sulfide form, the prior treatment of spent catalysts before leaching would enhance the leaching efficiency of vanadium [9]. Although the acidity of oxalic acid is very weak [15], the oxalate has good chelating properties and thus has a strong tendency to

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form complexes with metals ions [16]. Therefore, oxalic acid can be considered to be a potential reagent for the leaching of vanadium from the spent catalyst. The advantages of oxalic acid are that it can selectively dissolve metal ions and be more environment-friendly than inorganic acids. Oxalic acid can be decomposed at high temperature [17] and thus control of temperature is important in its employment for the treatment of spent catalysts [14, 18].

In the present work, oxalic acid was selected because it can act as a complex reagent to selectively extract vanadium from the spent catalyst [19, 20]. The dissolution behavior of vanadium by oxalic acid from spent petroleum catalyst was investigated. The effect of leaching conditions such as temperature, acid concentration, reaction time, and pulp density on the leaching efficiency of vanadium was investigated. The purpose of this work was to investigate the dissolution behavior of vanadium from the spent petroleum catalysts by oxalic acid solution.

2. Experimental

In this study, the spent petroleum catalysts were taken from a Korean refinery. Firstly, the soluble organic matters which covered the surface of spent catalyst were removed by acetone washing. Then, the obtained materials were dried overnight in an oven at 80°C. Before the leaching study, the material was thoroughly mixed to avoid heterogeneity. The characteristics of spent petroleum catalyst were analyzed by X-ray diffractometer (XRD, X'Pert-PRO, the Netherlands) and X-ray fluorescence spectrometer (XRF, M1MISTRAL, Germany). Muffle furnace (MF-32GH, JeioTech, Korea) was used to pretreat the spent catalyst. Oxalic acid (Duksan Pure Chemical Co., 99%) was employed for the leaching and oxalic acid solutions were prepared by dissolving in distilled water.

Leaching experiments were performed by taking 200 mL of the desired concentration of oxalic acid in a 500 mL three-neck round bottom flask. Leaching parameters were adjusted by using a magnetic stirrer bar in a heating mantle (WiseStir MSH-20D, Daihan Scientific Co., Korea). In all of the leaching tests, the weight ratio of the spent catalyst to leachant (pulp density) was controlled at 20 g/L except the effect of pulp density parameter. Once the reaction temperature was reached, the spent catalysts with the original shape and dimensions were poured into the reaction flask. After the required reaction period, the solution samples were taken and were separated from the residue on the filter paper using vacuum filtration. The concentration of metals in the filtrate was measured by inductively coupled plasma optical emission spectrometers (ICP-OES, Spectro Arcos, Kleve, Germany). The leaching percentage of metals

(component A) was calculated using Eq. (1).

$$A(\%) = \frac{C_A \times V}{W_{A, initial}} \times 100\% \quad (1)$$

where C_A and V are the concentration of component A in the leaching solution and the volume of leaching solution after filtration, respectively. $W_{A, initial}$ refers to the weight of component A in the spent catalyst before the leaching experiments.

3. Results and Discussion

3.1. Characteristics of the material

Fig. 1(a) shows that the shape of the spent catalyst before calcination was black cylindrical with an average particle size of 3 mm × 1 mm. Meanwhile, the spent catalyst after calcination had a dark yellow color as shown in Fig. 1(b). The chemical composition of the spent catalysts before and after heat treatment is presented in Table 1. According to the XRD and XRF data, the spent catalyst before calcination basically contains some metal ions in the form of metal oxides (Al_2O_3 , CoO , Fe_2O_3 , MoO_2 , NiO , SiO_2) and metal sulfides (NiS , MoS_2 , CoS , V_2S_3). The chemical composition of vanadium in the spent catalyst was found to be 36% by weight. The content of vanadium in the current material is much higher than that in the other spent petroleum catalyst [6, 8, 21]. Besides, the presence of sulfur species in the spent petroleum catalyst can slow the dissolution of metals during the leaching process [22]. Therefore, the material was calcined at 800°C in 4 h to oxidize vanadium sulfide before leaching experiments. This step is a fundamental process which has been reported in several publications [9, 23, 24]. As is shown in Table 1, the chemical compositions of metals were changed after heat treatment. The content of vanadium should be decreased because of the loss of sulfur from their sulfide form in the spent catalyst. Chemical composition of some metals such as nickel, molybdenum, and cobalt increased due to the conversion of these metals from sulfide to oxide form. Since the contents of iron and silica were low compared to those of other elements, the effect of calcination on the contents of iron and silica was not manifested in our data. There was an increase in the aluminum content after calcination. This can be ascribed to the hindering of the elemental sulfur on the surface of spent catalyst. After the removal of sulfur, aluminum can be more easily detected by XRF instrument. It has been reported that the presence of sulfur layer on the spent catalyst has a negative effect on the leaching efficiency of the metals from the spent catalyst [6]. It can be concluded that the oxidation of vanadium sulfide has been completed. Once calcination is complete and there is no sulfur left in the spent catalyst, the leaching of metal components from the calcined spent catalysts results



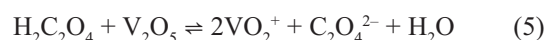
from the leaching of metal oxides. The leaching percentage of metals was calculated from the chemical compositions of the spent catalyst after heat treatment.

3.2. Leaching studies

The result of the leaching efficiency of metals from the spent catalyst by oxalic acid is shown in Fig. 2. In the initial test conditions at 60°C, 2 h reaction time, 1 M oxalic acid, 20 g/L pulp density, and 500 rpm stirring speed, the leaching percentage of vanadium was 37%. About 19% of Mo was leached into the oxalic acid solution, while the leaching percentage of other metals such as Al, Co, Ni, Fe, and Si was lower than 11%, indicating that oxalic acid can selectively dissolve vanadium and molybdenum. In these experiments, the composition of the leach liquor was found to be V-2.354 g/L, Al-0.113 g/L, Co-0.0004 g/L, Fe-0.024 g/L, Mo-0.725 g/L, Ni-0.061 g/L, and Si-0 g/L at 1 M oxalic acid. The reported values of stability constants for the complex formation of metals with oxalate are shown in Table 2. It can be said that the complexes of V(V) and Mo(VI) are more thermodynamically stable than that of other metals in oxalic acid solution. Therefore, the difference in the complex formation constant with oxalate ion results in selective dissolution of V(V) and Mo(VI).

Since the leaching efficiency of other metals except vanadium was low (see Fig. 2), the present work focused on the leaching of vanadium from the spent petroleum catalyst. The dissociation reactions of diprotic oxalic acid, the dissolution reaction of vanadium oxide, and subsequent complex formation reactions between

oxalate and vanadate are represented as Eqs. (2-7) [15, 31, 32]. The effect of several leaching conditions such as oxalic acid concentration, temperature, pulp density, and stirring speed on the leaching efficiency of vanadium was investigated as a function of time in the following sections.



3.2.1. Effect of oxalic acid concentration

In order to investigate the effect of acid concentration on the vanadium leaching efficiency from the spent catalyst, the concentration of oxalic acid was varied from 0.1 to 2 M. In these experiments, temperature, pulp density, and stirring speed were fixed at 60°C, 20 g/L, and 500 rpm, respectively. Fig. 3 indicates that the leaching percentage of vanadium increased with an increase in oxalic acid concentration. Since oxalic acid is a weak acid, the concentration of oxalate is proportional to oxalic acid concentration. Therefore, the effect of complex formation became pronounced in high acid concentration. The leaching percentage of vanadium

Table 1. Chemical composition of the spent catalyst by weight (wt.%)

Element	Al	S	Fe	Ni	Mo	Si	Co	V
Before calcination	10.71	19.78	3.53	13.46	14.86	1.6	0.06	36
After calcination	26.03	0	1.12	21.6	19.38	0.12	0.13	31.62

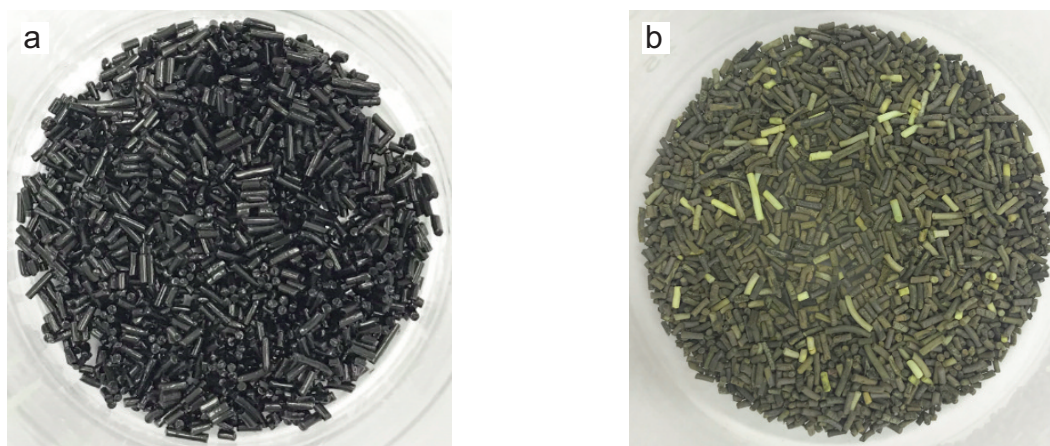


Figure 1. The spent catalyst (a) before and (b) after calcination

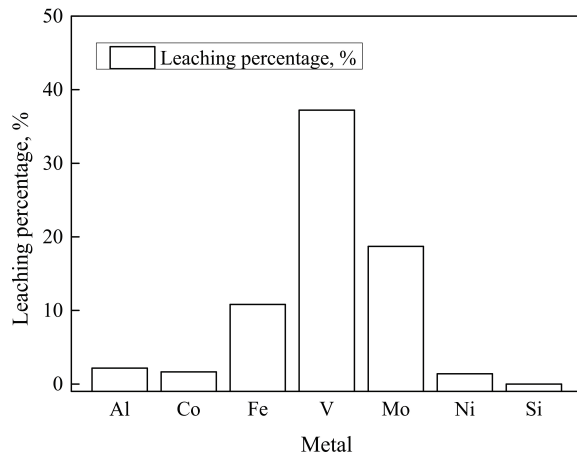


Figure 2. Leaching percentage of metals from the spent catalyst by oxalic acid. Conditions: temperature: 60°C, reaction time: 120 min, oxalic acid concentration: 1 M, pulp density: 20 g/L, stirring speed: 500 rpm

Table 2. The stability constants for the formation of metal complexes in oxalic acid solution

Metal	Complex	Log K	Ref.
V	$[\text{VO}_2(\text{C}_2\text{O}_4)_2]^{3-}$	10.2	[25]
Mo	$[\text{Mo}_2\text{O}_5(\text{OH})(\text{C}_2\text{O}_4)_2]^{3-}$	14.6	[26]
Ni	NiC_2O_4 , $[\text{Ni}(\text{C}_2\text{O}_4)_2]^{2-}$	3.7, 6.6	[27]
Co	CoC_2O_4 , $[\text{Co}(\text{C}_2\text{O}_4)_2]^{2-}$	3.25, 5.6	[25]
Fe	$[\text{Fe}(\text{C}_2\text{O}_4)_3]^{4-}$, $[\text{Fe}(\text{C}_2\text{O}_4)_3]^{3-}$	-2.48, 4.32	[28]
Al	$\text{Al}_2(\text{OH})_3(\text{C}_2\text{O}_4)_3$, $[\text{Al}_2(\text{OH})_2(\text{C}_2\text{O}_4)_4]^{4-}$	-4.28, -4.62	[29]
Si	$[\text{Si}(\text{OH})_3(\text{C}_2\text{O}_4)]^{-}$, $[\text{Si}(\text{OH})_4(\text{C}_2\text{O}_4)]^{2-}$	-8.5, 0.04	[30]

after 180 min increased from 33 to 44% in oxalic acid concentration range of 0.1-2 M. At low acid concentration (0.1 M), the dissolution rate of vanadium slightly increased in the first 60 min and remained a constant with increasing time. Meanwhile, the dissolution of vanadium gradually increased with time at higher oxalic acid concentrations. It is interesting that the formation of crystalline oxalic acid was observed after leaching experiments by employing high acid concentrations (1.5-2 M). This requires an extra step to separate the solids from the leaching solution. Therefore, 0.5 M oxalic acid was considered to be an optimum condition. The selection of oxalic concentration is in good agreement with previous studies [14, 18].

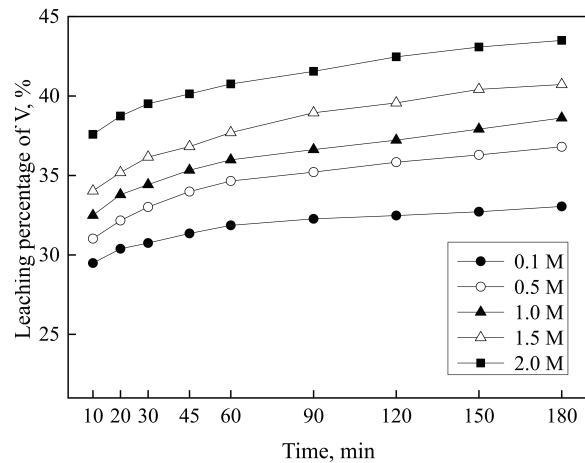


Figure 3. Effect of oxalic acid concentration on vanadium leaching. Conditions: temperature: 60°C, pulp density: 20 g/L, stirring speed: 500 rpm

3.2.2. Effect of pulp density

Fig. 4 displays the obtained results on the effect of pulp density on vanadium leaching efficiency as a function of time. The other parameters were fixed as follows: 0.5 M oxalic acid solution, 20 g/L pulp density, 60°C temperature, and 500 rpm stirring speed. The results show that the pulp density has a noticeable effect on the leaching of vanadium. As is shown in Fig. 3, the higher leaching percentage of vanadium was obtained at lower pulp density. When the pulp density was high, the amount of oxalic acid was not enough for the leaching. The leaching percentage of vanadium increased within 60 min but then became constant with the rise of time at a pulp density of 50 g/L (see Fig. 4). The increase in pulp density can cause rapid consumption of the reactant and hence further experiments were done at a pulp density of 20 g/L.

3.2.3. Effect of temperature

The effect of temperature on the leaching efficiency of vanadium by oxalic acid is shown in Fig. 5. The leaching experiment was carried out in the range of temperature from 25 to 80°C in 1 M oxalic acid solution at a pulp density of 20 g/L within 180 min leaching time and a stirring speed of 500 rpm. The temperature affected the leaching of vanadium in oxalic acid solution. It is seen in Fig. 5 that the leaching percentage of vanadium increased with the increase in temperature. Moreover, the leaching efficiency of vanadium gradually increased as a function of time in 120 min and slowly increased with the increase of leaching time. The fact that the dissolution rate of metal is faster at initial stage and becomes slow indicates that mass transfer through product layer is important in the leaching kinetics.

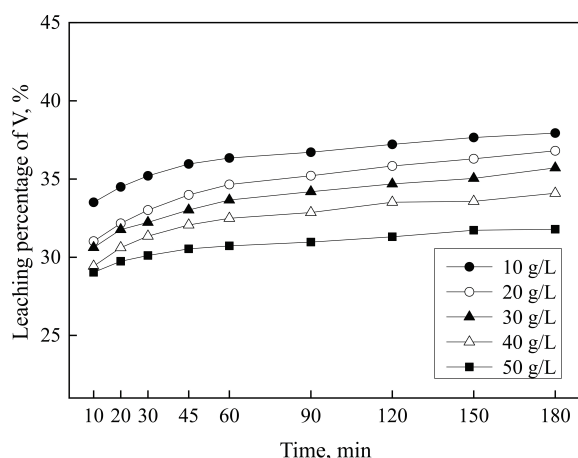


Figure 4. Effect of pulp density on vanadium leaching. Conditions: temperature: 60°C, oxalic acid concentration: 0.5 M, stirring speed: 500 rpm

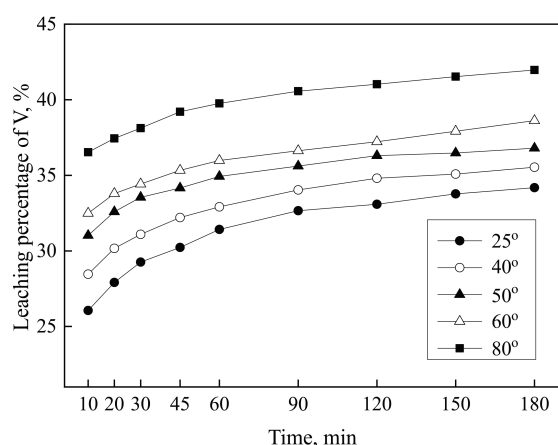


Figure 5. Effect of temperature on vanadium leaching. Conditions: oxalic acid concentration: 1 M, pulp density: 20 g/L, stirring speed: 500 rpm

The leaching time of 120 min can be considered enough to reach equilibrium. The effect of reaction temperature on the leaching percentage of vanadium was not significant. For instance, the leaching percentage of vanadium at 60 and 80°C after 120 min leaching time was 37% and 41%, respectively. Therefore, reaction temperature of 60°C was chosen in further leaching experiments. Some works show that reaction temperature of 50-60°C is optimum to dissolve vanadium by oxalic acid [14, 19]. Since temperature mildly affects the leaching efficiency of vanadium, the dissolution process of vanadium does not seem to be controlled by chemical reaction.

3.2.4. Effect of stirring speed

For the reaction to be effective in the acid leaching process, the stirring speed is necessary to provide efficient mixing as well as suspend the particles for

the dissolution of metal. The effect of stirring speed over time was varied in the range from 300 to 700 rpm at 60°C with a pulp density of 20 g/L and oxalic acid concentration of 0.5 M. As shown in Fig. 6, the stirring speed has a small effect on the dissolution of vanadium in the range of 300-500 rpm at the initial stage. After 30 min of leaching time, the stirring speed showed a negligible effect on vanadium leaching when increasing the stirring speed up to 700 rpm. This data indicates that the mass transfer of the reactants from the solution to the surface of the spent catalyst was fast. Mass transfer depends on several factors, such as stirring speed, temperature, and viscosity of the leaching solution. In some leaching systems, stirring speed should be high enough to maintain the dispersion of solids in the leaching solution [33]. Therefore, the experiments were conducted at a stirring speed of 500 rpm.

The best condition for the leaching of vanadium from the spent catalyst in oxalic acid solution was obtained as follows: temperature, 60°C; oxalic acid concentration, 0.5 M; pulp density, 20 g/L; leaching time, 120 min; and stirring speed, 500 rpm. At these conditions, the leaching percentage of vanadium was found to be 36%. The final chemical composition of leach liquor obtained under the optimum leaching conditions is as follows: V-2.266 g/L, Al-0.103 g/L, Co-0.0002 g/L, Fe-0.017 g/L, Mo-0.701 g/L, Ni-0.057 g/L, and Si-0 g/L at 0.5 M oxalic acid solution. The purity of vanadium in the leaching solution was about 72%. After the leaching process, it is important to find an effective separation method to recover vanadium with high purity. Our result is similar to the leaching data of vanadium by oxalic acid from spent hydroprocessing catalyst (34%) [34]. Compared to inorganic acids, the leaching percentage of vanadium by oxalic acid is low [13, 35, 36]. Some works

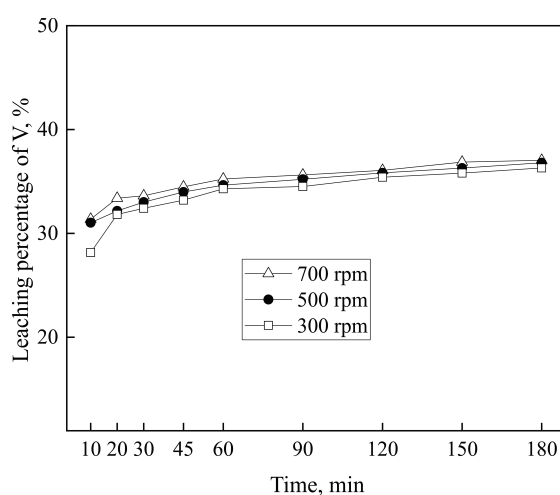


Figure 6. Effect of stirring speed on vanadium leaching. Conditions: temperature: 60°C, oxalic acid concentration: 0.5 M, pulp density: 20 g/L



indicated that the leaching efficiency of vanadium from spent catalysts by oxalic acid is higher than that by sulfuric acid solution [14, 19]. The change in the leaching percentage of vanadium can be ascribed to the leaching conditions and the difference in vanadium composition of the spent catalysts.

4. Conclusions

The leaching of vanadium from the spent petroleum catalyst by oxalic acid was studied. Characteristics of the material showed that the spent catalyst used in this work contains a significant amount of vanadium. In order to investigate the dissolution of vanadium from the spent catalyst by oxalic acid, leaching experiments were done by varying some leaching conditions. The best condition for vanadium leaching was obtained as follows: temperature of 60°C, pulp density of 20 g/L, oxalic acid concentration of 0.5 M, and stirring speed of 500 rpm. Oxalic acid showed selectivity for the dissolution of vanadium and molybdenum, while the leaching percentage of the other metals was very low. Further works need to be done to improve the leaching efficiency of vanadium from the spent catalysts by oxalic acid.

Acknowledgments

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SELEKTIVNO RASTVARANJE VANADIJUMA (V) IZ ISTROŠENIH NAFTNIH KATALIZATORA U PRISUSTVU OKSALNE KISELINE

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Apstrakt

Istrošeni naftni katalizator sadrži značajnu količinu vanadijuma i njegovo izdvajanje je od velikog značaja. Oksalna kiselina ima dobru metalnu selektivnost zbog svojih složenih osobina. Eksperimenti luženja u prisustvu oksalne kiseline su urađeni da bi se ispitalo ponašanje vanadijuma iz istrošenih naftnih katalizatora prilikom rastvaranja tako što su menjani uslovi tokom postupka luženja, kao što su temperatura, koncentracija, gustina pulpe, brzina mešanja i vreme. Vanadijum i molbiden su se selektivno rastvorili u prisustvu oksalne kiseline, dok je procenat ostalih metala dobijenih postupkom luženja bio nizak. Međutim, procenat vanadijuma dobijenog luženjem u prisustvu oksalne kiseline je bio niži od 45% pod većinom uslova prilikom luženja. Ovi rezultati pružaju osnovni uvid u rastvaranje dragocenih metala iz istrošenih katalizatora u prisustvu organske kiseline.

Ključne reči: *Istrošeni naftni katalizator; Vanadijum; Oksalna kiselina; luženje*

