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Extraction of Molybdenum (VI) and Vanadium (V) from Nitrate Solutions Using Coupling of Acid and Solvating Extractants

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A B S T R A C T

In this study, solvent extraction method has been utilized for separation of molybdenum and vanadium from nitrate solution by utilizing coupling of acid and solvating extractants (D2EHPA and TBP extractants). The outcomes demonstrated that synergistic solvent extraction improves stability of formatted complexes for transfer to the organic phase. The main parameters such as pH value, concentration of extractants, NH_4OH concentration as the stripping agent and contact time were optimized at 0.4, 15% (v/v) D2EHPA, 10% (v/v) TBP, 2 M and 30 min, respectively. The maximum separation factor of 24.84 was obtained with a new synergistic mixture of D2EHPA and TBP diluted with kerosene. According to McCabe-Thiel diagram, more than 99% of the Mo (VI) was obtained in counter current procedure and three stage numbers using 15% (v/v) D2EHPA and its mixture with 10% (v/v) TBP in kerosene, initial aqueous pH value equal to 0.4 and A/O ratio of 4/1 at room temperature for 30 min. In addition, stripping of Mo (VI) reached 98.75% in three counter current stages using 2 M NH_4OH as stripping agent and A/O ratio of 1/4 for 30 min.

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1. INTRODUCTION

Molybdenum is one of the precious and strategic elements in the world that is found with copper in nature [1]. It is one of the most in-demand rare elements [2]. Vanadium has important usages in the steel industry because of the high melting point, abrasion resistance and high tensile strength [3]. Molybdenum and vanadium coexist with other worth materials in various resources [4]. Among the common methods for separation and purification of Mo(VI) and V(V), solvent extraction is viewed as superior to different techniques on account that the initial concentration of metal is higher than 1 g/L [5, 6]. The extraction and separation process for molybdenum and vanadium are difficult because these metals have analogous chemical properties [7]. In recent years, many researchers investigated the process for extraction of molybdenum and vanadium from the leach solution. The process details with consideration of different conditions from various extractants are summarized in Table 1. Natural extractants, including TBP, have been reported in terms of synergist effect, and modifiers for separation of ions such as cobalt [8], nickel [9], zirconium [10], zinc [11] and molybdenum [12, 13].The synergistic solvent extraction could raise the stability of the formatted complexes for transfer to the organic phase, consequently, eliminate emulsification and the extraction reaction is faster. On the other hand, no experimental work for the separation of Mo (VI) from V (V) by a mixture of D2EHPA and TBP has been reported so far. In the present study, for the first time, the impact of some important parameters, such as initial aqueous pH, concentration of extractants, contact time, synergistic enhancement factor and concentration of NH_4OH as the striping agent for the separation of Mo (VI) from V (V) by mixture of D2EHPA and TBP was investigated. In addition, the extraction and stripping stages from equilibrium data were obtained from the McCabe- Thiele diagram.

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TABLE 1. A summary on the solvent extraction of Mo and V by different extractants

Extractant	Aqueous solution	Selectivity	Reference
D2EHPA	H_2SO_4 , pH= 0.8, Mo= 4.45 g/L, V= 0.82 g/L	Mo (VI) over V (V)	[14]
TOA	H_2SO_4 , pH= 1.5, Mo= 4.45 g/L, V= 0.82 g/L, 0.05 M SO_3^{2-}	Mo (VI) over V (IV)	
LIX84I	H_2SO_4, pH= 0.5, Mo= 0.3 g/L, V= 8.5 g/L, Ni= 2 g/L,Al= 2.5 g/L, Fe= 0.28 g/L	Mo over V	[15]
Cyphos IL 101	H_2SO_4, pH= 0.5, Mo= 0.5 g/L, V= 0.5 g/L, Fe=Al=Cu= 1 g/L	Mo over V	[16]
Cyanex272+TBP	H_2SO_4, pH= 0.5,, Mo= 11.62 g/L, V= 0.46 g/L, Fe=31.65 g/L	Mo over V	[17]
PC 88A	H_2SO_4 , pH= 2, Mo= 1.06 g/L, V= 1.5 g/L	Both Mo and V	[18]
Alamine 308 + iso-decanol	H_2SO_4, pH= 1.5, Mo= 18.3 g/L, V= 1.03 g/L Ni= 2.66 g/L, Al=18.6 g/L	Both Mo and V	[19]
Aliquat 336	NaOH , pH= 8.5, Mo= 20 g/L, V= 1 g/L	V over Mo	[20]
D2EHPA+TBP	HNO_3 , pH= 0.4, Mo= 9 g/L, V= 0.1 g/L	Mo over V	This Study

2. Experimental

2. 1. Chemical Agents   The commercial extractants D2EHPA and TBP (99% and 99.5% purity) were purchased from Sigma-Aldrich. Kerosene was provided by Alfa Aesar as the diluent (98% purity). The organic phase was prepared using D2EHPA extractant with concentration between 2.5 to 20% (v/v)) and TBP extractant with concentration of 10% (v/v) diluted in kerosene. The aqueous solutions were prepared by dissolving 9 /L and 0.1 g/L of $Na_2MoO_4.2H_2O$ (Sigma-Aldrich, 99.9% purity) and $NaVO_3$ (Sigma-Aldrich, 99% purity) in distilled water, respectively to imitate leach solution [21]. Sodium hydroxide, and diluted nitric acid were used to adjust the pH of the aqueous solution. Ammonium hydroxide (NH_4OH) was utilized as a stripping solvent, which was supplied by Sigma-Aldrich.

2. 2. Analysis Apparatus   The concentrations of Mo (VI) and V (V) ions in the aqueous phase were measured by ICP instrument (PerkinElmer, Optima 2000 DV). Also, concentrations of ions in the organic phase were obtained with mass balance. Initial aqueous pH was determined with a digital pH meter (Sartorius PB-11). In addition, the mechanical shaker (CH-4103 Bottmingen) was used to contact the two phases at the equilibrium condition.

2. 3. Experimental Procedure   The experiments in two stages (extraction and stripping) were performed by mixing 10 mL of feed solution and organic phase (O/A=1) using a mechanical shaker with 400 rpm shaking rate. After shaking, the combinations of two phases were transferred to a separation funnel at 25 ± 1°C. Then, the metals concentrations in the aqueous phase were determined by ICP-AES analysis. The distribution ratio (D), extraction percentage (E), separation factor (SF), synergistic enhancement factor (SEF) and stripping percentage (S) are defined as follows:

$$D = \frac{[M]_{org}}{[M]_{aq}} \qquad (1)$$

$$E\% = \frac{[M]_{org}V_{org}}{[M]_{org}V_{org}+[M]_{aq}V_{aq}} \times 100 \qquad (2)$$

$$SF = \frac{D_{Mo}}{D_V} \qquad (3)$$

$$SEF = \frac{D_{mix}}{D_{D2EHPA}+D_{TBP}} \qquad (4)$$

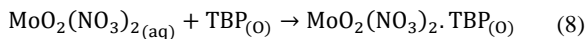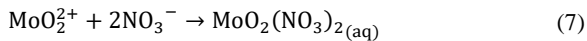$$S\% = \frac{[M]_{aq,s}V_{aq,s}}{[M]_{org}V_{org}+[M]_{aq,s}V_{aq,s}} \times 100 \qquad (5)$$

3. RESULTS AND DISCUSSION

3. 1. Impact of Initial Aqueous pH for Mo (VI) and V (V) Extraction   According to the previous studies, initial aqueous pH and concentration of the metals have a great influence on the type of predominate aqueous species of two ions in the solvent extraction procedure [22]. Molybdenum and vanadium belong to the VIB and VB group of the periodic table, respectively [23]. Molybdenum's overwhelming valencies are +4, +5 and +6 however the stable valence of +6 is considered for the solvent extraction process [24]. On the other hand, vanadium displays +2, +3, +4 and +5 valencies but the stable valencies are +4 and +5 [3]. Tangri et al. [25], investigated the influence of pH on the predominate species of molybdenum and vanadium (Table 2). In addition, the solvent extraction of molybdenum and D2EHPA can be expressed by the following equation [26]:

$$MoO_2^{2+}{}_{(aq)} + 2(HR_2PO_4)_{2(O)} \rightarrow$$
$$MoO_2(R_2PO_4)_2 . 2HR_2PO_{4(O)} + 2H^+{}_{(aq)} \qquad (6)$$
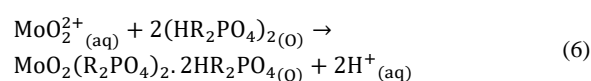
The neutral complex and molybdic acid can be extracted by adding of TBP to the organic phase [27]. According

to Equation (7), the molybdenum cations combine with NO_3^- to form extractable neutral sulfate complex. Therefore, the extraction of Mo (VI) by TBP can be written as Equation (8). The mixture of TBP with D2EHPA for the extraction of V (V) does not have huge synergist impact at lower temperatures [28].

$$MoO_2^{2+} + 2NO_3^- \rightarrow MoO_2(NO_3)_{2_{(aq)}} \qquad (7)$$

$$MoO_2(NO_3)_{2_{(aq)}} + TBP_{(O)} \rightarrow MoO_2(NO_3)_2.TBP_{(O)} \qquad (8)$$

To evaluate the effect of initial aqueous pH on the extraction of Mo (VI) and W (VI), tests were performed with various values of initial pH of the solution. It can be seen from Figure1 that the initial aqueous pH is one of the most important factors because the metals separation factor is largely dependent on the pH value. The concentration of D2EHPA in the kerosene, ratio of aqueous phase to organic phase (A/O) and contact time were kept constant at 15% (v/v) , 1/1, and 30 min, respectively. The percentage of Mo (VI) extraction decreased with increasing pH value whereas the percentage of V (V) extraction increased with increase in initial aqueous pH and reached a maximum of 57.7%, but after reaching to its maximum, it fell with further increment in pH value. Therefore, the pH value of 0.4 was chosen for subsequent experiments.

3. 2. Impact of Extractants Concentration for Mo (VI) and V (V) Extraction

To improve the separation factor of the extraction stage between both ions, a mixture of [D2EHPA] and [TBP] was applied in the experimental data. To comprehend the significance of the impact of extractants concentration on the separation factor, the concentration of D2EHPA and TBP were various in the range of 2.5 to 20% (v/v) and 5to 20% (v/v), respectively. Changing the separation factor as a function of D2EHPA concentration at various TBP concentrations is shown in Figure 2. The maximum value for separation factor is reached when the D2EHPA and TBP concentrations are equal to 15% (v/v) and 10% (v/v), respectively and a further increment in D2EHPA or TBP concentrations results in a decrease in separation factor of extraction stage. The reduction in separation factor was probably a result of a viscosity increase in the organic phase. These phenomena cause undesirable disturbances and consequently separation factor decreases. According to Equation (4), the synergistic enhancement factor results were obtained for Mo (VI) and V (V) to investigate synergism in the mixture of extractants. The results of solvent extraction, when these metals were extracted using a mixture of D2EHPA and TBP in the organic phase are given in Table 3.

3. 3. Impact of Contact Time for Mo (VI) and V (V) Extraction

Figure 3 shows the influence of phase contact time for Mo (VI) and V (V) extraction from the aqueous phase. It is clearly shown that the equilibrium condition was obtained after 20 min. Be that as it may, the contact time of 30 min was selected to guarantee complete equilibration. Therefore, the mixing time of 30 min was chosen as an optimum equilibrium time in the experimental conditions.

3. 4. Isotherm of Mo (VI) Extraction
Basically, in the solvent extraction process, which to formation of

TABLE 2. Effect of various aqueous phase pH on the formation of predominate species of Mo and V [25]

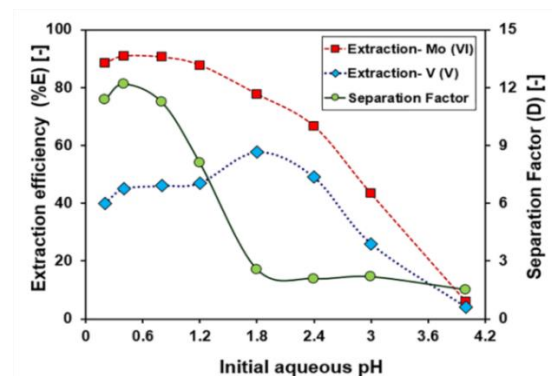| Predominate Species			
pH	Molybdenum	pH	Vanadium
> 6.5	MoO_4^{2-}	> 13	VO_4^{3-}
4	$Mo_7O_{24}^{6-}$	9	$V_2O_7^{4-}$
2	$Mo_8O_{26}^{4-}$	6	$V_3O_9^{3-}$
≤ 1	MoO_2^{2+}	2	$V_{10}O_{28}^{6-}$
		< 2	VO_2^{+}

Figure 1. Effect of initial aqueous pH on the extraction percentage of Mo (VI) and V (V) and separation factor (15% (v/v) D₂EHPA, [Mo(VI)]=9 g/L, [V(V)]=0.1 g/L, contact time=30 min, A/O=1 and T= 298.15K)
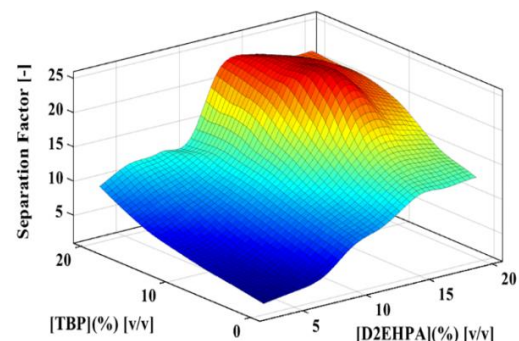

Figure 2. Effect of D₂EHPA and TBP concentrations on the metals separation factor in the extraction stage (pH=0.4, [Mo(VI)]=9 g/L, [V(V)]=0.1 g/L, contact time=30 min and T=298.15K)

the third phase is possible, increasing the volume of the aqueous phase leads to undesirable disturbances, which will result in phase separation longer than usual [29]. On the other hand, the high cost of the organic phase also limits its application in the industry. The tendency is to increase the ratio of the aqueous phase to organic phase and increase in the number of extraction stages. In the experiments, a mixture of 15% D2EHPA and 10% TBP were contacted with an initial aqueous pH 0.8, in the various O/A phase ratios from 0.125:1 to 2:1.

The McCabe-Thiele diagram for the extraction of molybdenum is shown in Figure 4. As can be seen, three theoretical extraction stages are required for the complete extraction of Mo (VI) at an A/O ratio equivalent at 4:1 value.

3. 5. Impact of Stripping Reagent Concentration on the Stripping of Mo (VI) and V (V)   Molybdenum

TABLE 3. Extraction percentage values, synergistic enhancement factor and metals separation factor in the extraction stage using mixture extractants (pH=0.4, [Mo(VI)]=9 g/L, [V(V)]=0.1 g/L, 10% (v/v) TBP, contact time=30 min and T=298.15K)

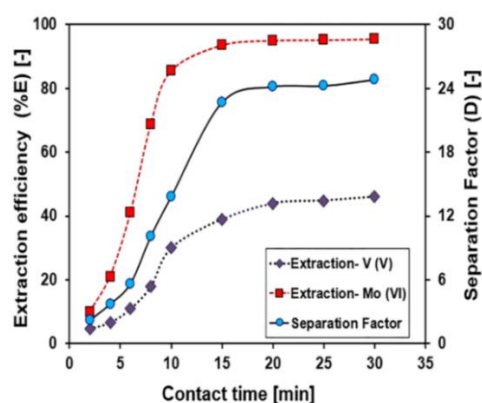| D$_2$EHPA % (v/v)	Extraction percentage (%)		SEF		SF
	Mo (VI)	V (V)	Mo (VI)	V (V)	
2.5	56.78	23.1	2.19	0.95	4.39
5	65.67	28.1	2.01	1.02	4.91
7.5	79.95	33.8	2.34	1.03	7.81
10	87.23	37.6	1.83	1.08	11.32
12.5	91.56	40	1.97	1.02	16.26
15	95.49	46.2	2.11	0.96	24.84
17.5	95.18	47.9	1.78	0.97	21.43
20	95.45	52.1	1.62	1.01	19.38

Figure 3. Effect of contact time on the extraction percentage of Mo (VI) and V (V) and metals separation factor (pH=0.4, [Mo(VI)]=9 g/L, [V(V)]=0.1 g/L, 15% (v/v) D2EHPA, 10% (v/v) TBP and T=298.15K)

and vanadium concentrations in the loaded organic phase were 8.594 and 0.056 g/L, respectively. When ammonium hydroxide solution was used for stripping of molybdenum, the emulsification and the development of a third phase happened but adding TBP as a function of phase modifier could raise the stability of the formatted complexes in the organic phase, eliminate emulsification and the stripping of molybdenum reaction was faster [30]. It can be seen from Table 4 that the maximum stripping separation factor was observed when the concentration of ammonium hydroxide was 2M.

3. 6. Isotherm of Mo (VI) Stripping   In this work, the stripping of molybdenum (VI) was investigated in diverse directions from the loaded organic phase. Various volume ratios of aqueous to organic phases from 1:8 to 2:1 of NH$_4$OH were used to study for the required number of theoretical stages in the stripping of MO (VI). The calculation of stages number is basic in the plan of mixer–pioneer units to achieve the noteworthy extraction efficiency and separation factor. As can be seen in Figure5, three equilibrium stages are required to strip 98.75% of molybdenum (VI) from the loaded organic phase.
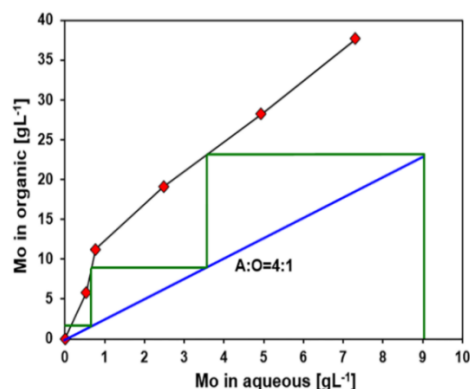

Figure 4. McCabe-Thiele diagram for the extraction of molybdenum (15% (v/v) D2EHPA and 10% (v/v) TBP in kerosene, pH 0.4, phase contact time of 30 min, O/A ratio of 1:1 and Mo(VI) concentration in aqueous phase= 9 g/L)

TABLE 4. Effect of ammonium hydroxide on the extraction percentage of Mo (VI) and V (V) and metals separation factor in the stripping stage (8.594 g/L Mo (VI), 0.056 g/L V (V), A/O=1, t=30 min and T=298.15K)

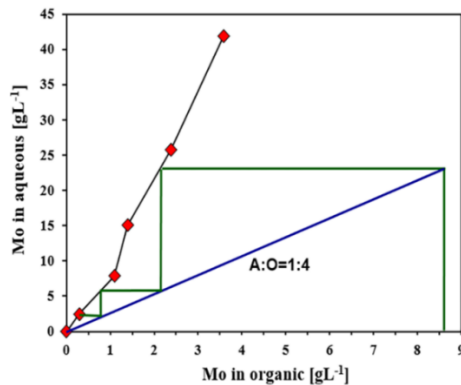| Stripping agent	Stripping percentage (%)		Striping separation factor (SF$_s$)
NH$_4$OH [M]	Mo (VI)	V (V)	
0.5	23.27	12.5	2.12
1	65.51	32.14	4.01
1.5	93.08	56.13	10.11
2	93.24	55.14	11.06

Figure 5. Stripping isotherm for loaded organic of molybdenum (Mo(VI) concentration in loaded organic phase=8.594 g/L, [NH$_4$OH]=2 M, t=30 min and T=298.15K)

4. CONCLUSION

In this study, solvent extraction using synergistic extractants of D2EHPA and its mixture with TBP in kerosene was done to accomplish the particular separation of molybdenum from vanadium. The separation factor of extraction stage reached 24.84 using a mixture of 15% (v/v) D2EHPA and 10% (v/v) TBP in the kerosene with initial pH value equal to 0.4, A/O ratio of 1/1 at room temperature for 30 min. Besides, stripping stage performances of Mo (VI) and V (V) from the loaded organic phase were investigated by ammonium hydroxide. Based on the results obtained, the maximum stripping separation factor of 11.06 was determined using 2 M NH$_4$OH in the aqueous solution with A/O ratio of 1/1 and at room temperature for 30 min. The McCabe-Thiele diagrams for both the extraction and stripping stages of Mo (VI) were constructed. The diagrams indicated that after three counter current stages, almost all of the Mo (VI) was extracted in the extraction stage and 98.75% of Mo(VI) was recoverd in the stripping stage.

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RESEARCH NOTE

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چکیده

در این مطالعه، روش استخراج حلالی در جداسازی مولیبدن و وانادیم از محلول نیتراتی با استفاده از ترکیب استخراج‌کننده‌های اسیدی و حل‌شونده به کار گرفته شد. فاز آلی حاوی D2EHPA و مخلوط آن با استخراج‌کننده تری‌بوتیل فسفات و کروزن به عنوان رقیق‌کننده تهیه شد. اثر مقدار pH، غلظت استخراج‌کننده‌ها، غلظت NH₄OH به عنوان فاز بازیابی و زمان تماس برای استخراج Mo(VI) و V(V) مورد مطالعه قرار گرفت. نتایج نشان داد که هم‌افزایی استخراج حلالی موجب پایداری کمپلکس‌های تشکیل شده در انتقال به فاز آلی می‌شود. ماکزیمم فاکتور جداسازی برابر با ۲۴/۸۴ از مخلوط هم‌افزایی جدید شامل (v/v%) ۱۵ از D2EHPA و (v/v%) ۱۰ از TBP رقیق شده در کروزن بدست آمد. براساس دیاگرام مک کیب تیل، بیش از ۹۹٪ از Mo(VI) در فرآیند جریان ناهمسو با سه مرحله از مخلوط (v/v٪) ۱۵ از D2EHPA و (v/v٪) ۱۰ از TBP رقیق شده در کروزن با مقدار pH اولیه فاز آبی برابر با ۰/۴ و نسبت فاز آبی به آلی برابر ۴ به ۱ در دمای اتاق و در مدت زمان ۳۰ دقیقه، بدست آمد. به علاوه، بازیابی Mo(VI) با ۹۸/۷۵٪ در سه مرحله ناهمسو با NH₄OH برابر با ۲ M و نسبت فاز آبی به آلی برابر با یک به ۴ به مدت ۳۰ دقیقه بدست آمد.

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