



Solvent extraction of vanadium(V) from sulfate solutions using LIX 63 and PC 88A



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ABSTRACT

Extraction of V(V) by LIX 63 and PC 88A was investigated from sulfate solutions under initial pH 1.2–6. Equilibrium pH had a profound effect on the extraction of V(V) by the two extractants. The extraction of V(V) by PC 88A occurred by a cationic exchange, while that by LIX 63 changed as the equilibrium pH increased. The cationic exchange reaction of VO_2^+ by LIX 63 occurred at $\text{pH} < 2.5$, while solvating reaction occurred in the equilibrium pH range of 2.5–5.4. The extraction reactions of V(V) by PC 88A and LIX 63 were identified by applying a slope method.

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Introduction

Vanadium is an important metal which has been widely used in the production of high-quality metal alloys. Due to the wide application of vanadium, processes have been developed to recover vanadium from diverse resources, such as spent catalysts [1–4], stone coal [5], oil flies [6], and sea water and tap water samples [7–9]. Recently, solvent extraction is widely employed to produce vanadium with high purity from the above-mentioned resources [10,11].

Various extractants have been tested for the extraction of vanadium from solutions. Basic extractants, such as $\text{C}_{18}\text{--C}_{24}$ primary amine (Primene JMT) [12], tri-octyl/dodecyl amine (Alamine 336) [1,13], tricaprylmethylammonium chloride (Aliquat 336) [2,14], 1-tridecanol [15], and tri-isooctyl amine (Alamine 308) [16] are used for the extraction of vanadium(IV/V) from acidic and alkaline media. Although high extraction efficiency is obtained, modifiers such as *n*-octanol, iso-decanol or tri-butyl phosphate should be added to organic phase to avoid the formation of emulsion or third phase during solvent extraction process. Compared to basic extractants, acidic organophosphorous (di-(2-ethylhexyl)-phosphoric acid (D2EHPA), 2-ethylhexyl phosphonic

acid mono 2-ethylhexylester (EHEHPA), bis(2,4,4-trimethylpentyl) phosphinic acid (CYANEX 272)) and chelating extractant (5,8-diethyl-7-hydroxydodecane-6-oxime (LIX 63)) offer higher efficiency in the extraction of vanadium(IV/V) via a cationic exchange from weak acidic solutions [3,10]. It has been reported that cationic vanadium species, VO_2^+ at $\text{pH} < 3$ is easily extracted by D2EHPA, EHEHPA and Cyanex 272 and the extraction ability of extractants decreases in the following order: D2EHPA > EHEHPA > Cyanex 272 [10,17]. Zhang et al. [4] and Zeng and Cheng [3] have shown that LIX 63 can extract both Mo(VI) and V(V) from sulfate solution containing Al(III), Co(II), Ni(II), and Fe(III) in the pH range of 1–2.

It has been reported that the extraction of V(V) by LIX 63 from alkaline solution can be represented as solvating reaction [11]. However, the extraction reactions of V(V) by acidic organophosphorous and chelating extractants from acidic sulfate solutions have not been identified. Since stripping is the reverse reaction of a solvent extraction reaction, identification of the solvent extraction is necessary to find an effective stripping reagent. Therefore, in order to identify the extraction reaction of V(V) from acidic sulfate solutions, solvent extraction experiments have been done by using 2-ethylhexyl hydrogen 2-ethylhexyl phosphonate (PC88A) and LIX 63. From initial pH of the sulfate solutions from 1.2 to 6, the extracted species of V(V) were identified by applying a slope analysis method to the extraction data and the extraction reactions by PC88A and LIX 63 were identified.

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Experimental

Commercial extractants, LIX 63 (BASF, USA) and PC 88A (Chemserve Co. Ltd., Japan) were used without further purification. Kerosene (Samchun Pure Chem. Co., Korea) was used as a diluent. The synthetic solution of V(V) was prepared by dissolving NH_4VO_3 (Samchun Pure Chem. Co., Korea) in distilled water. The acidity of the solution was adjusted by using ammonia (Junsei Chem. Co., Japan) or sulfuric acid solution (Duksan Co., Korea).

The extraction experiments were carried out by mixing 10 mL of organic and aqueous phases in a 100 mL screwed cap bottle and the mixtures were shaken for 30 min (extraction equilibrium was reached within 30 min in initial tests) with a wrist action shaker. After shaking, the organic and aqueous phases were separated using separating funnels. The pH of the aqueous phase was determined by a pH meter (Orion Star A211, USA). The concentration of metal ions in the aqueous phase was determined by ultraviolet spectrophotometer (UV 1800, Shimadzu, Japan). The concentration of metal ions in the loaded organic phase was calculated by mass balance. The distribution ratio (D) was defined as the ratio of the concentration of the metal ion present in the organic phase to that present in the aqueous phase at equilibrium.

Results and discussion

Distribution of V(V) in aqueous solution

Solvent extraction behavior of V(V) is affected by the nature of predominant species which depend on the solution pH and the concentration of vanadium [15,18–20]. In order to determine the predominant species of V(V) in a sulfate solution containing 200 mg/L V(V), a distribution diagram of V(V) was constructed by considering the complex formation of various complexes. Table 1 lists the values of complex formation constants considered in this study [15,20]. According to Fig. 1, VO_2^+ is predominant in strong acid solution (solution pH < 2.5), while polynuclear species such as $\text{V}_{10}\text{O}_{27}(\text{OH})^{5-}$, $\text{V}_{10}\text{O}_{26}(\text{OH})_2^{4-}$, and $\text{VO}_2(\text{OH})_2^-$ exist in the pH range from 2 to 5. Anionic species, such as $\text{V}_{10}\text{O}_{28}^{6-}$, $\text{V}_3\text{O}_9^{3-}$ and $\text{V}_4\text{O}_{12}^{4-}$ are formed at pH > 5 and a small fraction of VO_2SO_4^- and $\text{VO}(\text{OH})_3$ exist in the pH range of 0–3. On the basis of the distribution diagram of V(V) shown in Fig. 1, V(V) can be extracted by cationic extractants when solution of pH is less than 2.5, while anionic extractants are adequate to extract V(V) from weak acid solution and alkaline solution.

Effect of pH on extraction of V(V) with LIX 63 and PC 88A

In general, the extraction of metals by cationic extractants depends on solution pH. In order to investigate the effect of the solution pH on the extraction of V(V) from acidic sulfate solution, a chelating extractant (LIX 63) and an acidic organophosphorous

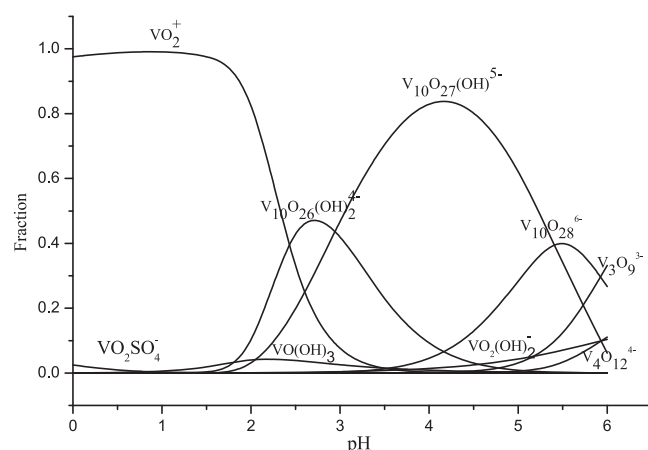


Fig. 1. Distribution diagram of V(V) species in sulfate media as a function of pH with V(V) concentration of 200 mg/L.

extractant (PC 88A) were chosen. The concentration of V(V) in the synthetic solution was 200 mg/L and this solution was contacted with 0.1 M LIX 63/PC 88A. In these experiments, initial pH of the solution was varied from 1.2 to 6. The corresponding equilibrium pH after extraction was from 1.6 to 5.4 for LIX 63 and from 1.4 to 3.5 for PC 88A. Fig. 2 shows the variation in the extraction percentage of V(V) against the equilibrium pH by LIX 63 and PC 88A, respectively. Although both LIX 63 and PC 88A are acidic extractants, the effect of the solution pH on the extraction behavior of V(V) by these two extractants was quite different from each other because of difference in extractant structure.

Fig. 2a clearly shows that the extraction reaction of V(V) by LIX 63 from sulfate solution changed as the equilibrium pH was increased from 1.6 to 5.4. Most V(V) was extracted by LIX 63 in the equilibrium pH range of 1.6–2.5 and then the extraction percentage decreased steadily from 98% to 20% with the increase of the equilibrium pH from 2.5 to 3.7. With the further increase in the equilibrium pH, the extraction of V(V) increased again. The extraction behavior of V(V) with LIX 63 in the equilibrium pH range of 1.6–2.5 agreed with the results reported in the literatures [3,4] which indicated that LIX 63 can extract V(IV/V) from solutions containing other metals in the pH range of 0–2.5. However, the extraction efficiency of LIX 63 for V(IV/V) in the high pH values of the solution was not investigated in these studies. From the obtained results, it is inferred that three reactions are involved in the extraction of V(V) by LIX 63 from the sulfate solution investigated in this study. According to Fig. 1, the mole fraction of VO_2^+ is nearly unity when solution pH is lower than 2.5 where LIX 63 may extract V(V) as a cationic extractant [3,4]. Although anionic species of V(V) are predominant at pH > 2.5, high extraction percentage of V(V) was observed in this pH range. This means that anionic complexes of V(V) were extracted by LIX 63 and the extraction of V(V) by LIX 63 may occur by solvating [21] or chelating formation [22] in the equilibrium pH range of 2.5–5.4. It can be concluded that the extraction reaction of V(V) by LIX 63 from sulfate solution changed as the equilibrium pH was increased from 1.6 to 5.4.

In the case of extraction of V(V) by PC 88A, the extraction percentage of V(V) increased from zero to 87% with the increase of equilibrium pH from 1.4 to 3 and then declined slowly with the further increase of the equilibrium pH from 3 to 3.5 (Fig. 2b). The extraction behavior of V(V) by PC 88A agreed well with the variation of the mole fraction of VO_2^+ in sulfate solution with solution pH from 1.4 to 3.5. Moreover, the obtained results also agreed with the results reported in the literatures [10,17] which indicated that the extraction percentage of V(IV) from the sulfate

Table 1
Stability constants for the formation of complexes of V(V) in aqueous solution at 25 °C.

Equilibria	Thermodynamic constant at 25 °C	Reference
$\text{VO}_2^+ + \text{SO}_4^{2-} = \text{VO}_2\text{SO}_4^-$	$K_1 = 10^{-2}$	[15]
$10\text{VO}_2^+ + 8\text{H}_2\text{O} = 14\text{H}^+ + \text{V}_{10}\text{O}_{26}(\text{OH})_2^{4-}$	$K_2 = 10^{-8.64}$	[20]
$10\text{VO}_2^+ + 8\text{H}_2\text{O} = 15\text{H}^+ + \text{V}_{10}\text{O}_{27}(\text{OH})_2^{5-}$	$K_3 = 10^{-11.61}$	[20]
$\text{VO}_2^+ + 2\text{H}_2\text{O} = \text{H}^+ + \text{VO}(\text{OH})_3$	$K_4 = 10^{-3.3}$	[20]
$\text{VO}_2^+ + 2\text{H}_2\text{O} = 2\text{H}^+ + \text{VO}_2(\text{OH})_2^-$	$K_5 = 10^{-7.09}$	[20]
$\text{VO}_2^+ + 2\text{H}_2\text{O} = 3\text{H}^+ + \text{VO}_3(\text{OH})_2^{2-}$	$K_6 = 10^{-15.19}$	[20]
$10\text{VO}_2^+ + 8\text{H}_2\text{O} = 16\text{H}^+ + \text{V}_{10}\text{O}_{28}^{6-}$	$K_7 = 10^{-16.97}$	[20]
$4\text{VO}_2^+ + 4\text{H}_2\text{O} = 8\text{H}^+ + \text{V}_4\text{O}_{12}^{4-}$	$K_8 = 10^{-18.76}$	[20]
$3\text{VO}_2^+ + 3\text{H}_2\text{O} = 6\text{H}^+ + \text{V}_3\text{O}_9^{3-}$	$K_9 = 10^{-14.46}$	[20]
$\text{HSO}_4^- = \text{H}^+ + \text{SO}_4^{2-}$	$K_{10} = 10^{-2}$	[15]

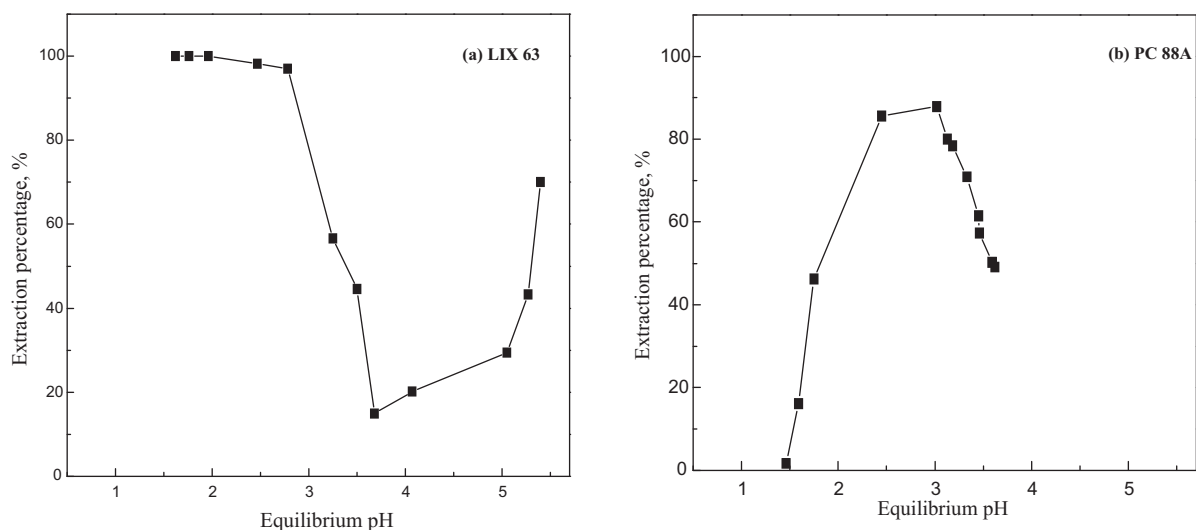


Fig. 2. Effect of equilibrium pH on extraction of V(V) with LIX 63/PC 88A. [V(V)] = 200 mg/L; Initial pH 1.2–6; [LIX 63/PC 88A] = 0.1 M; Diluent: Kerosene; O/A = 1:1.

solutions increases with the increase of equilibrium pH from 0 to 3. It has been reported that the extraction of metals by cationic exchange increases with solution pH until metal hydroxide begins to precipitate. Since the mole fraction of VO_2^+ is negligible at $\text{pH} > 2.5$, the extraction of V(V) by PC 88A decreased in the equilibrium pH range from 3 to 3.5. Hence, the extraction of V(V) by PC 88A in this study may be represented as a cationic exchange reaction.

Extraction reaction of V(V) by PC 88A

It can be inferred from Fig. 2b that the extraction reaction of V(V) by PC 88A from sulfate solution may be represented as a cationic reaction. According to the distribution diagram of vanadium (Fig. 1), cationic species, VO_2^+ are predominant in the pH range from zero to 2.5. Therefore, PC 88A can extract these species by cationic exchange reaction although PC 88A is weak acid. The plot of equilibrium pH vs log D by PC 88A is shown in Fig. 3. It is seen that the logarithm of the distribution ratio of V(V) increased with the increase of equilibrium pH value from 1.6 to 3 with a slope of unity, indicating that 1 mol of H^+ ion is involved in the extraction of 1 mol of V(V). The influence of PC 88A concentration on the extraction of V(V) was studied by varying

PC 88A concentration from 0.05 to 1 M, keeping the initial pH at 4. The extraction of V(V) increased from 83 to 97% with the increase of extractant concentration from 0.05 to 1 M, while the equilibrium pH of the solution decreased from 3.5 to 2.9. Fig. 4 shows that the plot of log [extractant] vs log D was linear with a slope value of 0.5, indicating that 1 mol of PC 88A was associated in extraction of 1 mol of V(V). The effect of metal concentration on the extraction behavior of V(V) by 0.1 M PC 88A was investigated by varying the metal concentration between 50 and 400 mg/L. The plot of log $[\text{V(V)}]_{\text{aq}}$ vs log $[\text{V(V)}]_{\text{org}}$ gave a straight line with a slope of 0.6, indicating that a monomeric metal species was extracted into the organic phase (Fig. 5). Based on the obtained results, the extraction reaction of VO_2^+ by PC 88A in the equilibrium pH range of 1.4–3.5 can be represented as the following equation:



where HA denotes PC 88A.

Extraction reaction of V(V) by LIX 63

Extraction reaction in the equilibrium pH range of 0–2.5

Zhang et al. [4] reported that the extraction of V(IV) by LIX 63 increases steadily with the increase of equilibrium pH from zero

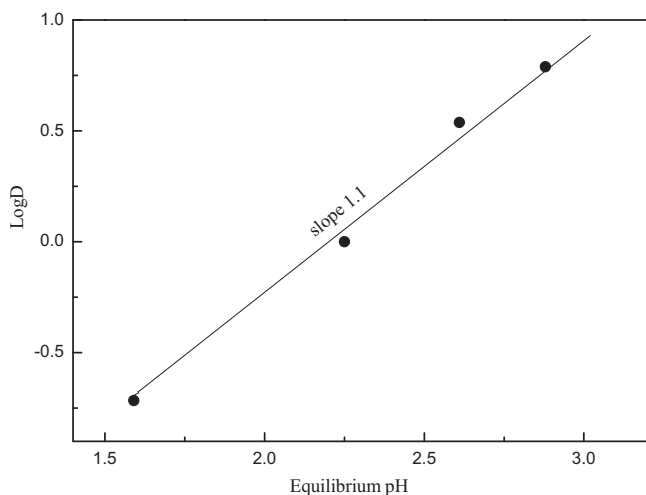


Fig. 3. Plot of log equilibrium pH vs log D . [V(V)] = 200 mg/L; [PC 88A] = 0.1 M; Diluent = Kerosene; O/A = 1:1; Initial pH 1.2–3.

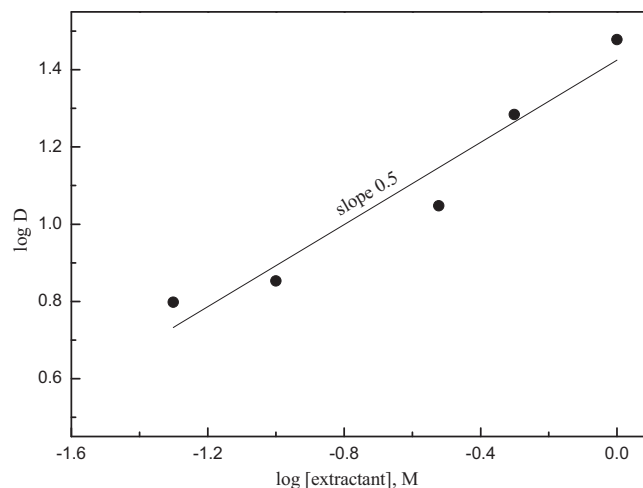


Fig. 4. Plot of log [extractant], M vs log D . [V(V)] = 200 mg/L; Initial pH 4; [PC 88A] = 0.05–1 M; Diluent = Kerosene; O/A = 1:1.

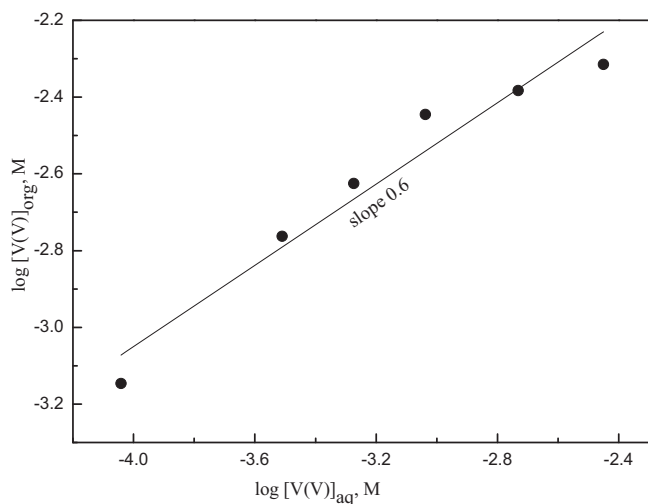


Fig. 5. Plot of $\log [V(V)]_{aq}$, M vs $\log [V(V)]_{org}$, M. $[V(V)] = 50\text{--}400$ mg/L; Initial pH 4; $[PC\ 88A] = 0.1$ M; Diluent = Kerosene; O/A = 1:1.

to 3, which was similar to that by PC 88A. In our experiments, most of V(V) was extracted by LIX 63 in the equilibrium pH range of 1–2.5 (see Fig. 2a) because V(V) has much higher affinity to LIX 63 than V(IV) [3]. It means that like PC 88A, LIX 63 can extract V(V) by cationic extraction reaction in the equilibrium pH range of 0–2.5. Some works [3,4] have reported that the extraction of V(V) with LIX 63 in the equilibrium pH range of 0–2.5 occurs by cationic exchange reaction and this agreed well with our data. Hence, the extraction reaction of V(V) by LIX 63 in the equilibrium pH from zero to 2.5 may be represented as Eq. (1).

Extraction reaction in the equilibrium pH range of 2.5–3.7

The plot of the equilibrium pH vs $\log D$ gave a straight line with a negative slope of unity, indicating that 1 mol of H^+ is extracted into LIX 63 (Fig. 6). The influence of LIX 63 concentration shows that the extraction of V(V) increased with the increase of extractant concentration. The solution pH decreased from 3.9 to 3.2 as LIX 63 concentration increased from 0.05 to 1 M. Fig. 7 shows that the plot of $\log [\text{extractant}]$ vs $\log D$ is linear with a slope value of 0.3, indicating that 1 mol of LIX 63 was associated in extraction of 1 mol of V(V). In order to investigate the existence of V(V) in the loaded organic, the concentration of V(V) was varied from 50 to

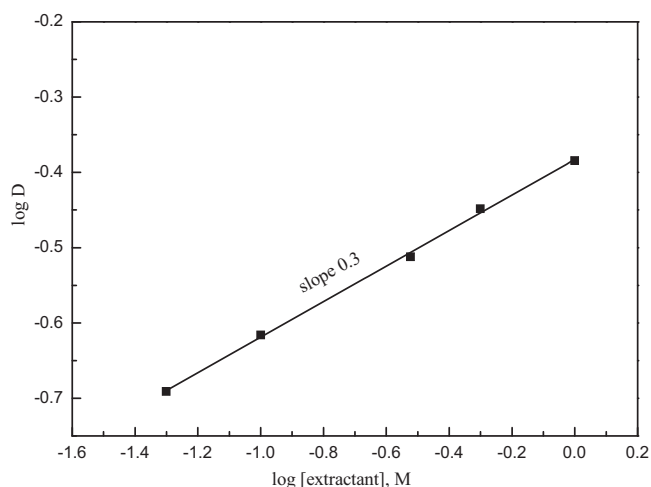
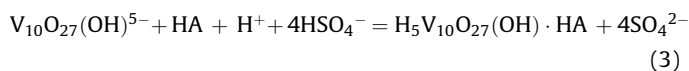
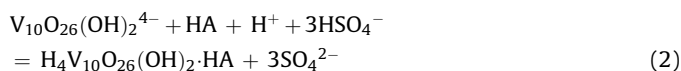


Fig. 7. Plot of $\log [\text{extractant}]$, M vs $\log D$. $[V(V)] = 200$ mg/L; Initial pH 4; $[LIX\ 63] = 0.05\text{--}1$ M; Diluent = Kerosene; O/A = 1:1.

400 mg/L. The plot of $\log [V(V)]_{aq}$ vs $\log [V(V)]_{org}$ by LIX 63 in these experiments is shown in Fig. 8. A straight line with a slope of 0.4 indicates that no polymerization of the extracted species of V(V) occurred in the LIX 63. The results shown in Figs. 6–8 indicate that the extraction of V(V) by LIX 63 at these pH ranges is supposed to occur by solvating reaction. The decrease of extraction percentage of V(V) in the equilibrium pH range of 2.5–3.7 (Fig. 2a) was due to a decrease in hydrogen ion concentration with the increase of the equilibrium pH. According to the distribution diagram (Fig. 1), anionic species including $V_{10}O_{27}(OH)^{5-}$ and $V_{10}O_{26}(OH)_2^{4-}$ are formed in the pH range of 2.5–3.7. Therefore, the extraction reaction of these species by LIX 63 can be represented as the following equations:



where HA is LIX 63.

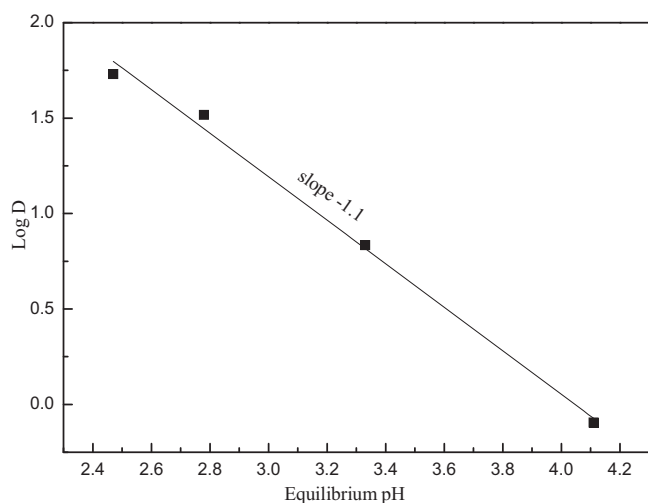


Fig. 6. Plot of \log equilibrium pH vs $\log D$. $[V(V)] = 200$ mg/L; $[LIX\ 63] = 0.1$ M; Diluent = Kerosene; O/A = 1:1; Initial pH 3–4.

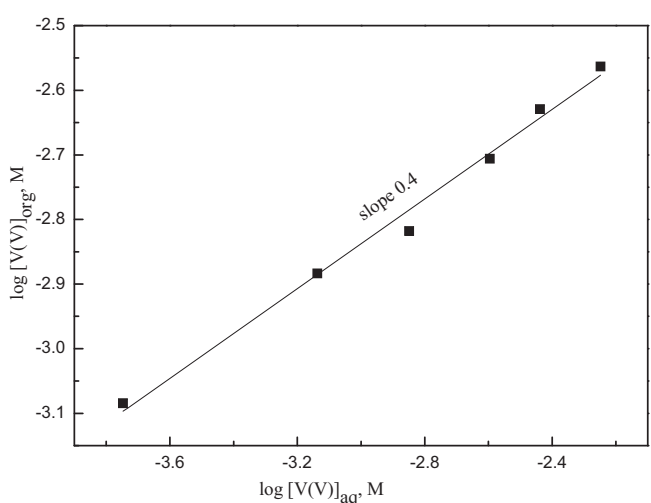


Fig. 8. Plot of $\log [V(V)]_{aq}$, M vs $\log [V(V)]_{org}$, M. $[V(V)] = 50\text{--}400$ mg/L; Initial pH 4; $[LIX\ 63] = 0.1$ M; Diluent = Kerosene; O/A = 1:1.

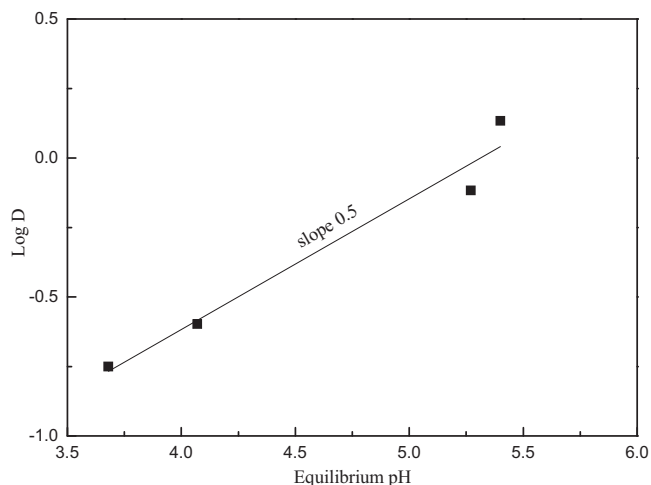


Fig. 9. Plot of log equilibrium pH vs log D . $[V(V)] = 200$ mg/L; $[LIX\ 63] = 0.1$ M; Diluent = Kerosene; O/A = 1:1; Initial pH 4–6.

The structure of $V_{10}O_{27}(OH)^{5-}$ and $V_{10}O_{26}(OH)_2^{4-}$ in organic phase has been reported in the literatures by using Burker/Siemens CCD method [22–24]. In order to elucidate the structure of $V(V)$ extracted at the equilibrium pH range of 2.5–3.7 by LIX 63, more fundamental works are necessary.

Extraction reaction at pH > 3.7

The distribution ratio of $V(V)$ increased with the increase of the equilibrium pH value from 3.7 to 5.4 with a slope of 0.5, indicating that 1 mol of H^+ ion was exchanged with 1 mol of $V(V)$ (Fig. 9). The influence of LIX 63 concentration on the extraction of 200 mg/L $V(V)$ was also studied by varying LIX 63 concentration from 0.05 to 1 M, keeping initial pH at 6. The solution pH decreased from 7.3 to 3.7 as LIX 63 concentration increased from 0.05 to 1 M. Fig. 10 shows that the plot of log [extractant] vs log D was linear with a slope value of 0.8, indicating that 1 mol of LIX 63 was associated with 1 mol of the extracted species. The plot of log $[V(V)]_{aq}$ vs log $[V(V)]_{org}$ for these experiments is shown in Fig. 11. A straight line with a slope of 0.5 indicates that no polymerization of extracted species occurred in the organic phase. Since anionic species, $V_{10}O_{27}(OH)^{5-}$, is predominant at pH > 3.7 (Fig. 1), LIX 63 may still extract $V(V)$ as a solvating extractant [25]. Based on the obtained results, the extraction reaction between the extractable species of

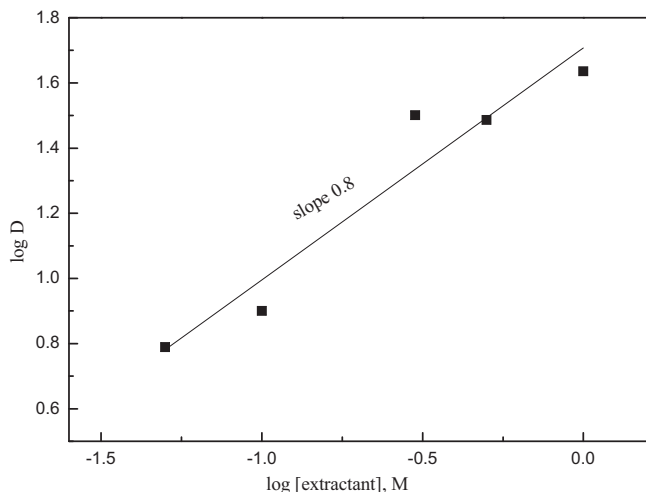


Fig. 10. Plot of log [extractant], M vs log D . $[V(V)] = 200$ mg/L; Initial pH 6; $[LIX\ 63] = 0.05$ –1 M; Diluent = Kerosene; O/A = 1:1.

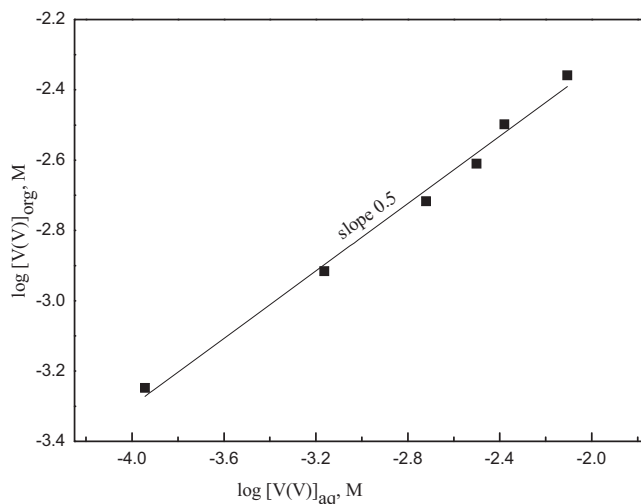
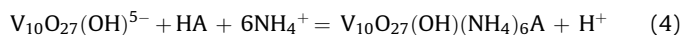


Fig. 11. Plot of log $[V(V)]_{aq}$, M vs log $[V(V)]_{org}$, M. $[V(V)] = 50$ –400 mg/L; Initial pH 6; $[LIX\ 63] = 0.1$ M; Diluent = Kerosene; O/A = 1:1.

$V(V)$ and LIX 63 can be represented as the following equation:



Since ammonium ion is a weak acid, it can take part in the solvent extraction reaction at relatively moderate pH.

Conclusion

Solvent extraction of $V(V)$ from sulfate solutions has been investigated using LIX 63 and PC 88A as a function of equilibrium pH, extractant and metal concentration. The extraction of $V(V)$ depended strongly on the equilibrium pH and the extraction behavior of $V(V)$ by LIX 63 was different from that by PC 88A. The extraction of $V(V)$ by PC 88A occurred via a cationic exchange mechanism in the initial pH range from 1.2 to 6. While one reaction is responsible for the extraction of $V(V)$ by PC 88A, several reactions can occur during the extraction of $V(V)$ by LIX 63 from sulfate solutions. Cationic exchange mechanism between $V(V)$ and LIX 63 occurred in the equilibrium pH range of 1.6–2.5 and the extraction reaction was the same as PC 88A. When the equilibrium pH was higher than 2.5, the extraction of anionic species of $V(V)$ by LIX 63 occurred via a solvating mechanism. The dependence of the extraction of $V(V)$ on the hydrogen concentration at the equilibrium pH > 2.5 indicated that two different reactions can occur as the equilibrium pH increases.

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