On the Extraction of Vanadium (V) by Alkanols: A comparative Study of 1-pentanol and 3-methyl-1-butanol Isomers as Extractants for Vanadium (V) Extraction from Hydrochloric Acid Solutions

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Abstract

Further to the previous study on the extractive recovery of V(V) from HCl solution by some alkanols, the extraction of V(V) from HCl solutions by 1-pentanol and 3-methyl -1-butanol (isomers) dissolved in n-heptane were investigated in the present study, still propelled by the need to proffering viable alternatives to the scarce and relatively costly organophosphorous extractants, and also compare the extraction efficiency of straight- and branched-chain extractants. At the studied pH range (0.5-4.0) with 1-penttanol as extractant, the percentage V(V) extraction (extraction efficiency, E%) reached optimum E%= 40.0 at pH of 3.0 and for 3-methyl-1-butanol, optimum E% was 36.7 at pH 2.0. Increasing concentration of HCl at the studied range ($1.0 - 6.0 \text{ mol L}^{-1}$) was found to have an appreciable effect on percentage V(V) extraction. With both extractants, E% peaked at 5.0 and 6.0 mol L⁻¹HCl with values 70.0 and 66.7% for 1-pentanol and 3-methyl-1-butanol respectively.

With increase in the concentration of extractants, both extractants produced a trend of increased/decreased in E% for V(V), suggesting the extraction of two different species. The optimal percentage extraction values were 70.0 and 66.7% observed at log [1-pentanol] = 0.86 mol L⁻¹ and log[3-methyl-1-butanol]=0.86 mol L⁻¹, in that order. The determined enthalpy change. ΔH , for 1-pentanol and 3-methyl-1-butanol systems were -80.4/120 kJ mol⁻¹ and -203.0/254.7 kJ mol⁻¹ respectively, indicating exothermic/endothermic extraction processes. The isomer 3- methyl -1-butanol gave a higher extraction efficiency after a three-stage extraction (E% = 92.5) than 1-pentanol (E% = 83.4). Recycling experiment revealed both to be recyclable. From slope analysis, analytical and spectra data, the extracted complexes have been formulated as VO₂Cl.nROH and (VO₂Cl)_m.nROH (where m>> n) at low and high concentrations of extractant, respectively. On the whole, 3-methyl-1-butanol was assessed as fairly better as an extractant for vanadium (V) at lower temperature experimental conditions. The effect of methyl branched-chain played an insignificant role on the extraction.

Keywords: vanadium (V), extraction, 1-pentanol, 3-methyl-1-butanol, isomers, slope analysis, enthalpy

1. Introduction

Vanadium applications in various fields of human endeavours particularly modern technology, as well as its extraction in form of cationic vanadium (V) have been reported in recent work (Ojo and Oyegoke, 2013). The use of alkanols to extract valuable metals such as niobium (V) and tantalum (V), aluminium (III), gallium (III) and indium (III) was reviewed as well (Yamada, Hayashi & Yasui, 2006).

In the previous work reported by the current authors, vanadium (V) was extracted from HCl solutions by 1–butanol and 1–octanol with remarkable efficiencies, (Ojo & Oyegoke, 2013). It is a common knowledge that several alkanols such as 1–pentanol (Figure 1) and 3-methyl-1-butanol (Figure 2) share similar electronic and solvating properties with those previously investigated, and at least superficially, are expected to have the potentiality for V (V) extraction.

In addition to this, isomerization in compounds as in the case of 3-methyl-1-butanol, (branched-chain) and the straight-chain 1-pentanol expectedly should affect covalent and lipophillic properties of compounds, with the tendency to cause a disparity in solvating power and invariably the extraction efficiency. The presence of methyl branched-chain is also expected to bring a new dimension of disparity in steric hindrance of the extractants, and probably influence the extraction efficiency. It is in the light of this that 1-pentanol (straight-chain) and 3-methyl-1-butanol (branched-chain) were investigated as extractants for V (V) from HCl solutions.

 $CH_3 - CH_2 - CH_2 - CH_2 - CH_2 - OH$

Figure 1. The structure of 1 – pentanol

 CH_3 $CH_3 - CH_2 - CH_2 - CH_2 - OH$ Figure 2. The structure of 3– methyl – 1 – butanol

2. Materials and Methods

2.1 Materials Used and Their Sources

The chemicals NH_4VO_3 , KCl, $Na_2WO_4.2H_2O$, HCl, HNO_3 , H_3PO_4 , and the reagents 1-pentanol, 3-methyl-1-butanol and n – heptane were purchased from the British Drug House Limited (BDH). All the materials were of analytical grade except the reagents (solvents) and were used as purchased.

Ammonium metavanadate (3.51g, 0.03mol) with KCl (7.48g, 0.1mol) were dissolved in hydrochloric acid solutions of varied concentrations, and made up to the 1–litre mark as V (V) stock solution.

Solutions of 1–pentanol and 3-methyl-1-butanol of varied concentrations in n–heptane and of the ranges 5.43-9.05 mol L^{-1} and 5.47-9.11 mol L^{-1} respectively, were prepared by dissolving the pure alkanols in n–heptane and made up to the 1-litre mark with the diluent.

2.2 Extraction and Analytical Procedure

Extraction, separation and analysis (vanadium –phosphotungstate method) were carried out as previously reported except that pentanol and 3-methyl-1-butanol were used as extractants (Ojo & Oyegoke, 2013).

The effects of pH (in the range 0.5–4.0), concentration of HCl $(1.0 - 7.0 \text{ mol } \text{L}^{-1})$, variation in temperature between 301 and 321 K, recycling of pentanol and 3-methyl-1-butanol, batch extraction stages were investigated. The amount of vanadium extracted was determined by UV–visible spectrophotometry of the yellow phosphotungstate–vanadium complex at 400 nm (Vogel, 1998). The electronic spectra of the pure 1-pentanol and 3-methyl-1-butanol and their organic phases of V (V) were recorded on the Alpha spectrophotometer in the range 200–900 nm and 4000-650 cm⁻¹ at the Obafemi Awolowo University, Ile–Ife, and Bowen University, Iwo, Nigeria, respectively.

3. Results and Discussion

3.1 The Effect of pH

The effect of pH on percentage V (V) extraction (E%) from HCl solutions with 1–pentanol and 3–methyl – 1 – butanol is presented in Figure 3. The E% for V (V) extraction using 1–pentanol and 3-methyl-1-butanol follow an irregular trend characterized by increased/decreased, with optimal values attained at pH of 3.0 (E% = 40.0) and 2.0 (E% = 36.7) for pentanol and 3-methyl-1-butanol respectively. The observed trend was due to the prevalence of diverse kinds of extractable/unextractable V (V) species such as VO₂⁺, H₂VO₄ and H₂V₁₀O₂₈⁴⁻ which is consistent with what some authors reported in the organophosphorous extraction of V (V) from aqueous acids (Kurbatova & Kurbatov, 2006; Greenwood & Earnshaw, 1997; Ojo 2010; J. Ojo, Adebayo & I. Ojo, 2012), and also the alkanol extraction of V (V) by current authors (Ojo and Oyegoke, 2013). The unappreciable difference in the extraction efficiency (E%) of both extractants at this pH region might be due to the observed closeness in their hydrolytic stability(from preliminary work), implying that linear chain–length plays no role on E% under this experimental condition.



Figure 3. Effect of pH on the extraction of V(V) from HCl solutions with 1-pentanol and 3-methyl-1-butanol $[V(V)] = 0.03 \text{ mol } L^{-1}$; $[1 - \text{pentanol}] = 5.89 \text{ mol } L^{-1}$; $[3 - \text{methyl-1-butanol}] = 6.38 \text{ mol } L^{-1}$; Temperature = 301 K

3.2 The Effect of Concentration of HCl

The effect of concentration of HCl investigated at the range 1.0–7.0 mol L⁻¹ on V (V) extraction with 1–pentanol and 3-methyl-1-butanol is presented in Figure 4. The E% increased gradually from 30.0% at [HCl] = 1.0 mol L⁻¹ to 70.0% at [HCl] = 5.0 mol L⁻¹ followed by a decrease (E% = 46.7) with the use of 1–pentanol as extractant.While for 3-methyl-1-butanol, the trend was increased, decreased and increased. The least E% was 20.0% observed at [HCl] = 1.0 mol L⁻¹ and 66.7% at [HCl] = 6.0 mol L⁻¹.

The observed general increase in E% at increased concentration of HCl for both extractants could be explained on the basis of increased desolvation of coordinated water from the V (V) hydration sphere, making the pervanadyl specie (VO_2^{+}) available for binding to the alkanolic oxygen, as similarly reported by the current authors on V (V) extraction by 1- butanol and 1-octanol and other workers (Ojo & Adeeyinwo, 2011; Thomas & Reddy, 2003; Ojo & Oyegoke, 2013).

It is worth noting that the E% values of 1- pentanol are generally higher (although minimally) than 3-methyl-1-butanol at the studied concentration range which could not be explained on the basis of solubility of both extractants in aqueous solutions, as they have nearly the same solubilities, 2.3 g /100 g and 2.0 g/100 g H₂O for 1- pentanol and 3-methyl-1-butanol respectively (Morrison and Boyd, 2009). And for the fact that both extractants showed good phase separation at nearly the same rate (from preliminary work), differential interfacial and coalescent properties could very scarcely account for the disparities in E%. The plausible reason that could be advanced is the steric hindrance presented by 3-methyl-1-butanol with branched methyl group, which may relatively hinder the binding of the VO₂⁺ specie to the alkanolic oxygen.



Figure 4. Effect of concentration of HCl solutions on percentage V(V) extraction $[V(V)] = 0.03 \text{ mol } L^{-1}$; $[1 - \text{pentanol}] = 5.89 \text{ mol } L^{-1}$; $[3 - \text{methyl-1-butanol}] = 6.38 \text{ mol } L^{-1}$; Temperature = 301 K

3.3 Effect of Concentration of Extractants

The effect of concentration of 1-pentanol and 3-methyl-1-butanol is depicted by Figure 5.From the slope analysis curves of the extractants, two linear portions were observed (at lower and higher concentrations). It was observed that increased extractant concentration produced an increased E% up to an optimum value, and subsequently decreased.

At log [1-pentanol] = 0.73, log D = 0 (E% = 50.0) which increased to an optimum of log D = 0.60 (E% = 86.5) at log [1-pentanol] = 0.86. As regards 3-methyl-1-butanol, at log [3-methyl-1-butanol] = 0.74, log D = 0.30 (E% = 66.7) which increased to an optimum of log D = 0.37 (E% = 70.0) at log [3-methyl-1-butanol] = 0.86 and subsequently decreased.

The difference in percentage extraction of V (V) by both extractants at lower concentration of extractants is minimal but becomes appreciable as the concentration increases.

This may be explained by the fact that angular VO_2^+ ion is not encountering much steric hinderance from the stericprone methyl branch of 3-methyl – 1 – butanol at lower extractant concentration (lower amount of molecules) but becomes hindered at higher concentration (higher amount of molecules). This observation is in agreement with our reported work where the linear nature of the vanadyl ion (VO^{2+}) was viewed to have played a vital role on its better extraction than the steric–prone angular pervanady ion, VO_2^+ from HCl solution by di (2–ethylhexyl) phosphoric acid. (J. Ojo, Adebayo and I. Ojo, 2012).

If assumed that VO_2^+ is the predominant extractable specie, its extraction by both alkanols can be generally represented by Equation 1 (Ojo and Oyegoke, 2013).

$$\operatorname{VO}_{2}^{+}_{(\mathrm{aq})} + \operatorname{Cl}_{(\mathrm{aq})}^{-} + \operatorname{nROH}_{(\mathrm{org})} = \operatorname{VO}_{2}\operatorname{Cl}^{-} \operatorname{nROH}_{(\mathrm{org})}$$
(1)

Then,

$$\mathbf{K}_{\text{ex}} = [\mathbf{VO}_2 \mathbf{CI}^{\text{-}} \mathbf{n} \mathbf{ROH}]_{\text{org}} / [\mathbf{VO}_2^{+}]_{\text{aq}} \cdot [\mathbf{CI}^{-}]_{\text{aq}} \cdot [\mathbf{ROH}]_{\text{org}}^{\text{n}}$$
(2)

$$D = [VO_2Cl nROH]_{org} / [VO_2^+]_{aq}$$
(3)

Where "aq", "org", K_{ex} and D represent aqueous and organic phases, and extraction equilibrium constant and distribution coefficient, respectively.

Substituting, we have

$$\mathbf{K}_{\mathrm{ex}} = \mathbf{D} / [\mathbf{ROH}]^{\mathrm{n}} \cdot [\mathbf{CI}^{\mathrm{n}}] \tag{4}$$

Taking logarithm of both sides, while keeping [Cl⁻] constant, we have

$$logD = 10gK_{ex} + nlog[ROH]$$
(5)

A plot of log D against log [ROH] should expectedly yield a straight line of slope equal to 'n' the number of solvated molecules. However, the two linear portions observed on the plots of both 1–pentanol and 3–methyl-1-butanol at lower and higher extractant concentrations imply that two different species were extracted.

With 1-pentanol, at the ascending portion of the curve (lower concentration), the determined slope, 'n' was equal to six and for the descending portion of the curve, it was -4.6, which implies that six molecules of 1-pentanol were coordinated at lower concentration, while polymeric V (V) specie is coordinated to one molecule of 1-pentanol at high concentration which does not agree with Equation (1).

With respect to 3–methyl-1-butanol, the determined slope 'n' was equal to 1.0 and -0.8 at lower and higher extractant concentrations, respectively, implying that one molecule of 3–methyl-1-butanol was coordinated to V (V) at lower concentrations while polymeric V (V) was coordinated to one molecule of 3–methyl-1-butanol at higher concentrations, similar to observations made in Mo(VI)/HCI/TBP and V (V)/HCI/ROH systems (Ojo, Ipinmoroti & Adeeyinwo, 2008.) and these could not be rationalized by Equation (1).

The extraction at higher concentration of alkanols could therefore be represented by the Equation (6).

$$mVO_{2}^{+} + mCI^{-} + nROH \qquad (VO_{2}CI)_{m}.nROH \qquad (6)$$





Figure 5. A plot of log D versus log [1-pentanol] or log [3-methyl-1-butanol] for the extraction of V(V) from HCl solutions.

 $[V(V)] = 0.03 \text{ mol } L^{-1}; [HCl]_{optimum} : 5 \text{ mol } L^{-1} (1-\text{pentanol}), 6 \text{ mol } L^{-1} (3-\text{methyl-1-butanol});$

Temperature = 301 K

3.4 Effect of Temperature

The effect of temperature on the distribution coefficient of V(V) extraction from 5.0 and 6.0 mol L⁻¹ HCl by 1-pentanol and 3-methyl-1-butanol respectively at the temperature range 301-321K is shown in Figures 6 and 7. Both extractants show a characteristic ascending and descending orders as the temperature increased.TheVan't Hoff equations is

$$\log D = -\Delta H/2.303RT + C \tag{7}$$

where D, Δ H and C represent distribution coefficient, enthalpy change and constant for the extractive reaction respectively (Talla, Gaikward & Pawar, 2010). It could be explained (from fundamentals) that the two linear portions described by equation of a straight line are characteristic of mathematical step functions of positive and negative linear regressions of defined 1/T (that is 'x') limits ($3.22 \leq x < \infty$ to $-\infty < x < 3.22$ where x = 1/T). The plot of log D against 1/T x 1000 yielded two linear ascending /descending straight lines of the slopes equal to 4.2 and -6.3 and Δ H was determined to be -80.4 and 120 kJ mol⁻¹ indicating exothermic and endothermic reaction at lower and higher temperatures with 1-pentanol. As regards 3-methyl-1-butanol the plot of log D versus 1/T x 1000 yielded slopes of 10.3 and -13.3 and ΔH values of -203.0 and 254.7 kJ mol⁻¹, the extraction could similarly be referred to as exothermic / endothermic.

These values of (Δ H) were much higher than were obtained for 1-butanol and 1-octanol extraction of V(V) reported in the previous work (Ojo and Oyegoke, 2013). The presence of dual energy profiles (exothermic/endothermic) as temperature changes in the extraction of V(V) by these alkanols are peculiar and exciting, which could be exploited to an advantage in making a choice of operating temperature if it were to be commercially exploited. This dual characteristic feature is similar to the presence of two activation energies (Arrhenius plots at low &high temperatures) observed in the kinetic study of Mo(VI) extraction from HNO₃ by tri-n—butyl phosphate reported by the current author (Ojo, 2007)



Figure 6. A plot of log D versus 1/T x 1000 for the extraction of V(V) from HCl solutions with 1 - pentanol[V(V)] = 0.03 mol L^{-1} ; [1 - pentanol] = 5.89 mol L^{-1} ; [HCl] = 5 mol L^{-1} .



Figure 7. A plot of log D versus 1/T x 1000 for the extraction of V(V) from HCl solutions with 3 - methyl-1-butanol $[V(V)] = 0.03 \text{ mol } L^{-1}$; [3-methyl-1-butanol] = 6.38 mol L^{-1} ; [HCl} = 6.0 mol L^{-1}

3.5 Extraction Stages

T.I.I. 1 TL.

The determination of number of extraction stages required for quantitative extraction of V(V) from 5.0 and 6.0 mol L⁻¹ HCl by 1-pentanol and 3-methyl-1-butanol respectively is presented in Table 1. With the use of 1-pentanol the percentage of V(V) extracted after a three-stage extraction was 83.4% while for 3-methyl-1-butanol was 92.5% at likewise extraction stages implying the later was more efficient.

Table	1.	Ine	effect	OI	number	01	extraction	stages	on	percentage	$\mathbf{v}(\mathbf{v})$	extraction	with	1-pentanoi	and
3-meth	yl-1	-buta	nol.												

N/N

Stage	ge 1-pentanol		3-methyl-1-butanol	
	$V(V)conc/(g L^{-1})$	E(%)	V(V)conc/(g L ⁻¹) E(%)	
1	0.458	70.0	0.509 67.3	
2	0.356	76.7	0.229 85.0	
3	0.254	83.4	0.115 92.5	

Initial V(V) concentration = 1.529 g L^{-1}

3.6 Recycling

The effect of the number of recycling stages of 1-pentanol and 3-methyl-1-butanol on percentage extraction and recycling coefficient of V(V) in 5.0 and 6.0 mol L⁻¹HCl medium respectively is presented in Table 2.With 1-pentanol the distribution coefficient (D) and percentage extraction (E%) decrease minimally with three consecutive extractions from E%= 70,0 (at D=2.3) to 68.2(at D=2.1).In order to quantify the recycling effect, the recycling coefficient (R.C) was defined as "D" of nth extraction/D of initial extraction (or 1st stage). If R.C \geq 1.0 the extractant is recyclable and if R.C< 1.0, it is weakly recyclable. The determined R.C values were greater than one (R.C. = 1.1) using 1-pentanol.

While for 3-methyl-1-butanol the E% =70.0 (at D = 2.3) was also greater than one (R.C. =1.2), hence, the extractants could be said to be recyclable (the latter being slightly higher) which was supported by the insignificant losses in volumes during equilibrations.

1-pentanol					3-methyl-1-butanol			
Recycling s	tage							
	D	E%	R.C ^b	D	E%	R.C ^b		
1 ^c	2.3	70.0	-	2.0	66.7	-		
2	2.1	68.2	1.1	2.2	68.3	1.1		
3	2.3	70.0	1.0	2.3	70.0	1.2		

Table 2. Effect of number of recycling stages on distribution coefficient, percentage and recycling coefficient on V(V) extraction

b =Recycling coefficient, $1^{c} =$ initial extraction

3.7 Electronic Spectra

The electronic spectra bands of 1-pentanol dissolved in n-heptane, aqueous and organic phases of 1-pentanol-V(V) are presented in Table 3.

The electronic absorption spectra of 1-pentanol in n-heptane showed absorption bands at 29940 ($\mathcal{E} < 1.0 \text{ M}^{-1} \text{ cm}^{-1}$) ascribable to $n \rightarrow \sigma^*$ transition. This band shifted positively (shorter wavelength) by about 3173 and 3617 cm⁻¹ in the aqueous and organic phases of 1-pentanol-V(V) system respectively, suggesting a positive involvement of ligand-to-metal charge transfer (LMCT) between the bound Cl⁻ and V(V) lend credence to by the observed and typically high molar absorptivity ($\mathcal{E} = 246 - 2590 \text{ M}^{-1} \text{ cm}^{-1}$). With 3-methyl-1-butanol dissolved in n-heptane, its absorption band was observed at 30674 ($\mathcal{E} < 1.0 \text{ M}^{-1} \text{ cm}^{-1}$) ascribable to $n \rightarrow \sigma^*$ transition. The positive shift in absorption frequency (hypsochromic) was about 2659 cm⁻¹ for both the aqueous and organic phases of 3-methyl-1-butanol- V(V) system and molar absorptivities were 3429 and 805 M⁻¹ cm⁻¹ in that order , similarly, charge transfer was advanced (Ojo, Ipinmoroti & Adeeyinwo, 2007; J. Ojo, Adebayo, & I. Ojo, 2012; Ojo, 2013; Ojo & Oyegoke, 2013).

Table 3. Electronic spectra data for the extractants, aqueous and organic phases of V(V) in 1-pentanol and 3-methyl-1-butanol

Compound	Band (cm-1)	ε / M ⁻¹ cm ⁻¹	Assignment
1-pentanol	29940	<1	$n \rightarrow \sigma^*$
Aqueous phase of V(V)	33113	2590	Charge transfer
Organic phase of V(V)	33557	246	Charge transfer
3-methyl-1-butanol	30674	< 1	$n \rightarrow \sigma^*$
Aqueous phase of V(V)	33333	3429	Charge transfer
Organic phase of V(V)	33333	805	Charge transfer

3.8 Infrared Spectra

The infrared spectra data for the extractant and organic phases of 1-pentanol and 3-methyl -1-butanol are presented in Table 4. With 1-pentanol extractant, infrared absorption bands are observed at 3338 and 2958 cm⁻¹ ascribable to U (O-H) and U(C-H) vibrations, respectively.

In the organic phases, the U (O-H) vibration shifted downwards by 72 cm⁻¹ indicating the involvement of the oxygen atom of the hydroxyl group in bonding to the VO_2^+ ion.

A new band observed at 790 cm⁻¹ assigned to U (V = O) is indicative of coordination of the pervanadyl ion (VO₂⁺) to the oxygen atom of the hydroxyl group.

As regards 3-methyl-1-butanol extractant, the observed bands are 3341 and 2958 cm⁻¹ assigned to U (O-H) and U (C-H) vibrations. In the organic phase, the U (O-H) shifted downwards by 72 cm⁻¹ indicating that the hydroxyl group was involved in bonding to the VO_2^+ ion. In addition, a new band observed at 790 cm⁻¹ which was absent in the exctractant has been assigned to U (V=O) is a confirmation of the coordination of the pervanadyl ion to the alkanolic hydroxyl group (Ojo, 2010; Ojo, & Oyegoke, 2013; Kurbatova, Kurbatov, & Medvedeva, 2004; Kurbatova & Kurbatov, 2006; Lamber, Sharvell, Lightner, & Cooks, 1987).

Table 4. Infrared spectra data for the extractant and organic phase of V(V) in 1-pentanol and 3-methyl-1-butanol

Compound	Band (cm ⁻¹)	Assignment			
1-pentanol	3338	U (O-H)			
	2958	U (C-H)			
Organic phase of V(V)	3266	U (O-H)			
	2958	U (C-H)			
	790	U (V=O)			
3-methyl-1-butanol	3341	U (O-H)			
	2958	U (C-H)			
Organic phase of V(V)	3265	U (O-H)			
	2960	U (C-H)			
	790	U (V=O)			

4. Conclusion

From the current study, the extraction efficiency of 1-pentanol and its isomer, 3-methyl-1-butanol are at par, with the presence of branched methyl chain exhibiting an insignificant effect on the overall efficiency of the extractant. However, at lower temperatures, 3-methyl-1-butanol has a slight edge in terms of extraction efficiency and recycling coefficient over the straight-chain analog. But on the whole, either could be selected for the extraction of V(V) from HCl solutions with high prospects.

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