

A simple approach for the estimation of the mass transfer coefficients and diffusion coefficients of vanadium in Kuwait crude oils: an elution study

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ABSTRACT

One of the major concerns following the Iraqi invasion is the possibility of ground water contamination due to massive oil spillage. Nickel and vanadium are the major metals which exist in trace amounts in crude oils. The leaching of vanadium was studied on a laboratory scale using four different crude oils. Kuwait export (KE), Burgan (B), Ratawi (R) and Weathered (W). Nitric, sulphuric and acetic acids (pH 4 and 6) were the leaching medium. The role of a polar medium like de-ionized water was also checked. In the case of an acid medium, the rate of leaching was found to be significantly dependent on the type of crude and the pH value of the leaching medium.

The values for the mass transfer coefficient of vanadium are in the range $0.04\text{--}6.4 \times 10^{-3} \text{ cm}^3 \text{ min}^{-1}$. The diffusion coefficient values for vanadium are in the range $0.06\text{--}45.4 \times 10^{-7} \text{ cm}^2 \text{ day}^{-1}$. An overall study of these values suggests that nitric acid is the most effective leachant for vanadium in the four crude oils. The values for the mass transfer coefficient and diffusion coefficient using de-ionized water as the eluent stresses the importance of hydrogen ion concentration in elution studies. Time for 10% leaching vanadium in the oils is estimated. A novel feature stemming from this study is the values for the rate for 10% leaching. The time required for the leaching of 10% of vanadium ranged between 4–54.6 × 10 years.

An analysis of variance (ANOVA) of different experimental parameters is presented. The confidence interval for the concentration of vanadium was estimated. Parameters such as the type of acid, pH value of the acid and the type of crude were considered. The ANOVA tests indicated that the leaching ability of the acids varied with the type of crude. The tests showed that there exists a relationship between the initial pH value of the acid and the metal concentration. Better leaching results were obtained with acids having pH 4.

INTRODUCTION

The destruction of several oil fields in Kuwait has resulted in a massive oil spillage causing terrestrial pollution (Mackinnon & Peter 1991). The environmental damage which has been underpredicted is receiving increasing attention from the public and the scientific community.

Crude oils are known to contain appreciable amounts of nickel and vanadium. For

example, nickel and vanadium concentrations in the Saudi and Kuwaiti crudes varies between 1 and 29 mg kg⁻¹ and 1 and 60 mg kg⁻¹, respectively (HPI Consultants 1987; Sadiq & Zaidi 1984). The heavy metals, nickel and vanadium, have been proven to exist as chelates in crude oils. Structural elucidation of these compounds is not well established (Speight 1990; Tissot & Welte 1978).

The oil lakes in Kuwait contain water, either from the fire fighting efforts or rain. Due to weathering and atmospheric chemical reactions, rain water exhibits varying pH values (Hassan 1992). There exists a possibility that the water soluble hydrocarbons and heavy metals (nickel and vanadium) may seep into the ground water. The extent of seepage is dependent on the geological properties of the soil underlying the oil lake, the chemical nature of the crude oil, its specific gravity, and meteorological factors prevailing at that site. Therefore the rate at which the metals seep into the earth's surface is uncertain. (Simulation of a natural leaching process similar to that occurring in the open oil lakes in Kuwait is beyond the scope of this work.)

Jatin & Colin (1978) made an attempt to study the leaching of radium from contaminated soils. Carbon dioxide and sulphur dioxide distilled water (pH 5.5 and 3.5 respectively) were used as the leachants. The rate of leaching was dependent on the pH of the leachant and the soil texture. Diffusion coefficients based on Fick's law were established. The values were between 5×10^{-11} and 2×10^{-13} cm² day⁻¹. The time periods for long-time leaching were also established.

In another study, Baghdady & Sippola (1984) investigated the extraction of cadmium, chromium, nickel and lead from soil using aqua regia, 0.5 N HNO₃ and acid ammonium acetate-EDTA. The mean values of the metals extracted indicated that 0.5 N HNO₃ and acid ammonium acetate-EDTA could be used as a leachant in pollution studies.

Tyler (1978) studied the leachability of manganese, zinc, cadmium, nickel, vanadium, chromium, copper and lead from a control soil and polluted soil. Artificial rainwater (pH 4.2, 3.2, and 2.8) was used in the experiments. Ten percent residence time for manganese and lead in the control and polluted soil was determined. The residence time was found to decrease with decreasing pH values. The effect of spilled oil on terrestrial, aquatic animals and ecosystems has also been examined (McCown *et al.* 1973; Wein & Bliss 1973).

In this study, sulphuric, nitric and acetic acids (0.01, 0.001M) were used as eluents. Similar acids were used earlier by different researchers (Jatin & Colin 1978; Baghdady & Sippola 1984; Tyler 1978) for studying the leaching of different metals. The acids employed in the present work resembled rain water. The work was carried out with the following objectives: (i) to demonstrate the efficacy of nitric, sulfuric and acetic acids (pH 4 and 6) on four different crude oils, (ii) to study the leaching rate of vanadium, (iii) to estimate the mass transfer and diffusion coefficients, and (iv) to determine the time required for 10% leaching of the metal under simple experimental conditions.

The work is presented as follows: three different acids are employed to identify the best leaching acid and its optimal pH. An exchange mechanism for the reaction of an acid with the trace metals present in the crude oil is suggested. The effect of a polar medium, like water, in the elution of metals is also studied. Lastly, an analysis of the overall leaching data within the purview of the studied parameters is presented.

MATERIALS AND METHODS

Materials

The crude oil samples used in the present study were Kuwait Export (KE), Burgan (B), Ratawi (R), and Weathered (W). The virgin oil samples of the first three types were obtained from storage tanks at Kuwait Oil Company (KOC). The partially-weathered crude (W) was obtained directly from the oil lakes. Two litres of each oil were stored at 5°C in sealed glass containers. The characteristics of the oil used in the present study are shown in Table (1). The density of crude oil was used as a measure to check consistency in the sampling procedure.

The objective of this work was to study the rate of leaching under the present experimental conditions. The soil used in the experimental work were representative soil samples from near the burning oil wells. It had sand as the only constituent. The particle diameter of the sand was measured and its size distribution is shown in Fig. 1. The soil was dried at 55°C for a period of 24 hours prior to the preparation of the sand beds.

Leaching Apparatus

A glass column 100 cm in height and 2 cm internal diameter was used to hold the artificially contaminated soil bed (Fig. 2). The column had a stopcock with a bore of

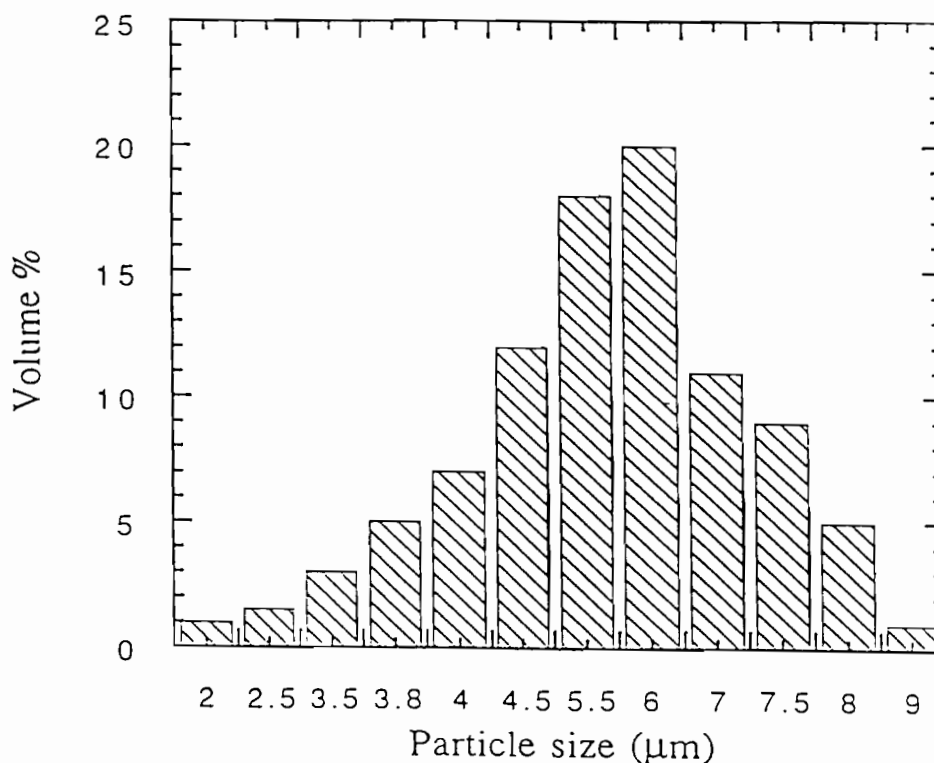


Fig. 1. Particle size distribution of sand.

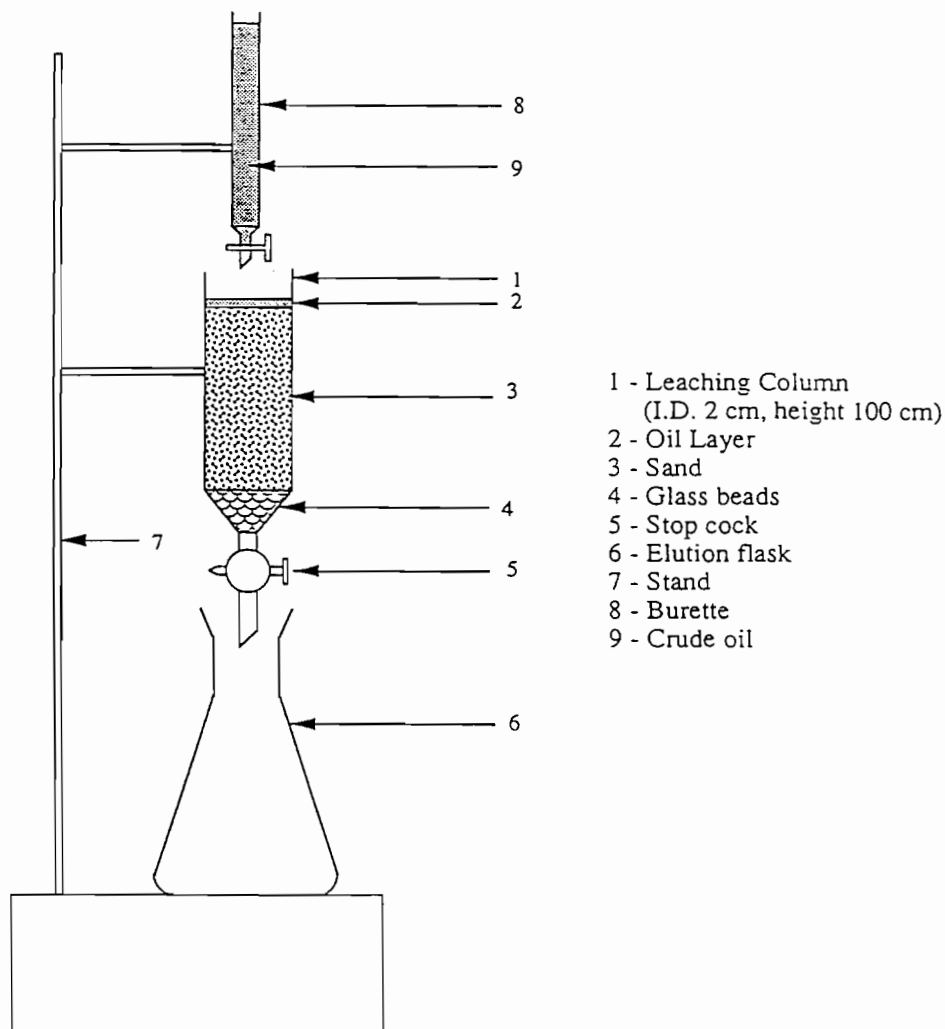


Fig. 2. Leaching apparatus.

5 mm internal diameter. The eluent was withdrawn through this outlet at predetermined time intervals. Glass beads having a diameter greater than the outlet of the leaching apparatus were used to support the sand bed.

Leaching Procedure and Analysis

All experiments were conducted at $20 \pm 2^\circ\text{C}$. Sand beds were made in the laboratory by loading 60 g of soil into the column. It was agitated for 15 minutes to promote settling. The final height of the sand bed was 25 cm. Artificially contaminated sand beds were prepared using 1.5 ml of the respective crude oil. A finely calibrated burette was used for transferring the oil into the bed. Although the time required for the oil to stop penetrating was only a few hours, the samples were kept for a period of 24 hours in order to maintain equilibrium. The acid solution was recycled every 30, 60, 90, 120,

150 and 180 minutes. The concentration of vanadium was estimated from 10 ml of the acid solution withdrawn at the end of the each time interval. The metal concentration was determined on a Philips 9100X atomic absorption spectrophotometer with a graphite furnace. The vanadium cathode tube operating at a wavelength of 318.5 nm was used in the studies.

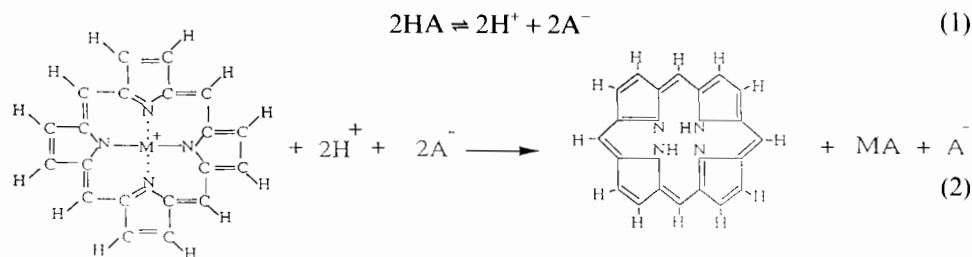
RESULTS AND DISCUSSION

The density, vanadium and sulfur content of the crude oil are presented in Table (1). These values confirm the fact that the chemical constituents of crude oil vary from source to source. Even in a particular source the chemical structures present might be different (Schobert 1990). The density of each oil was used as a selection criteria to prevent inconsistencies due to variation in molecular weight.

Table 1. Analysis results of the crude oil samples

Crude Type	Density (API) (ppm)	Vanadium Concentration	Sulfur Concentration (Wt%)
Kuwait Export (KE)	31.4	26.47	2.38
Burgan (B)	31.2	28.60	2.60
Ratawi (R)	23.9	40.80	3.99
Weathered (W)	19.2	44.8	3.44

Extraction of vanadium from mesotetraphenyl-porphyrin compounds was investigated by Donata *et al.* (1992) using the acids CF_3CO_2H and $C_7C_{15}CO_2H$. The leachability of vanadium was found to be dependent on the type of leachant used. Tyler (1978) studied the rate of leaching of metals such as zinc, cadmium, nickel, magnesium, vanadium, copper and chromium using acids of different pH values. The elution of these metals was dependent on the pH of the acid solution. The lower the pH of the extractant, the higher was the metal leaching rate. The atomic absorption data of our work showed an increase in the metal concentration with time. A typical plot illustrating this phenomena for vanadium using nitric acid (pH 6) as the eluent is shown in Fig. (3). It is therefore obvious that some reaction occurs between the acid solution and the metal. An exchange reaction between a porphyrin molecule and acid could be presumed to occur through the mechanism outlined below. If HA denotes a unionized acid, the following steps can plausibly occur in the exchange reaction:



where H^+ , MA and A^- denote the hydrogen ion, metal salt and anion, respectively.

At best this explanation is speculative, but nevertheless cannot be ruled out in our studies. An indirect confirmation was obtained by using $NaNO_3$ (5% W/V) as an eluent instead of acid. The metal ion concentration was not affected by the time of contact, thus ruling out the possibility that metal leachability is a result of counter ion effect. This experiment were conducted identically to those using acid as eluent. The atomic absorption data (Fig. 4) showed no increase in metal concentration, indicating the importance of an acid medium. (Although the conditions in this experimental work do not simulate the natural leaching conditions on site, this work does give an idea about a simple leaching process).

The mass transfer coefficient for nickel and vanadium can be calculated using the following expression (Geankoplis 1983):

$$\frac{C_0 - C}{C_0 - C_s} = \exp(-K_L A / V) \cdot T \quad (3)$$

where C_0 , C , C_s , K_L , A , V and T denote the total initial concentration of the sample in ppb, total leached concentration ppb of the metals at time T , saturation solubility in ppb, mass transfer coefficient in $cm^3 \text{ min}^{-1}$, the exposed area of the samples in cm^2 , volume of the solutions in cm^3 and time in minutes, respectively. The mass transfer coefficients for vanadium is shown in Table 2. The values of the mass transfer coefficient are in the range of $0.04-6.4 \times 10^{-3} \text{ cm}^3 \text{ min}^{-1}$ for vanadium. At pH 4 and 6, sulphuric and acetic acids are effective eluents for vanadium. Typical plots obtained through regression analysis (Figs. 5, 6 & 7) show that straight lines are obtained on plotting the experimental values of vanadium using sulphuric, acetic and nitric acids

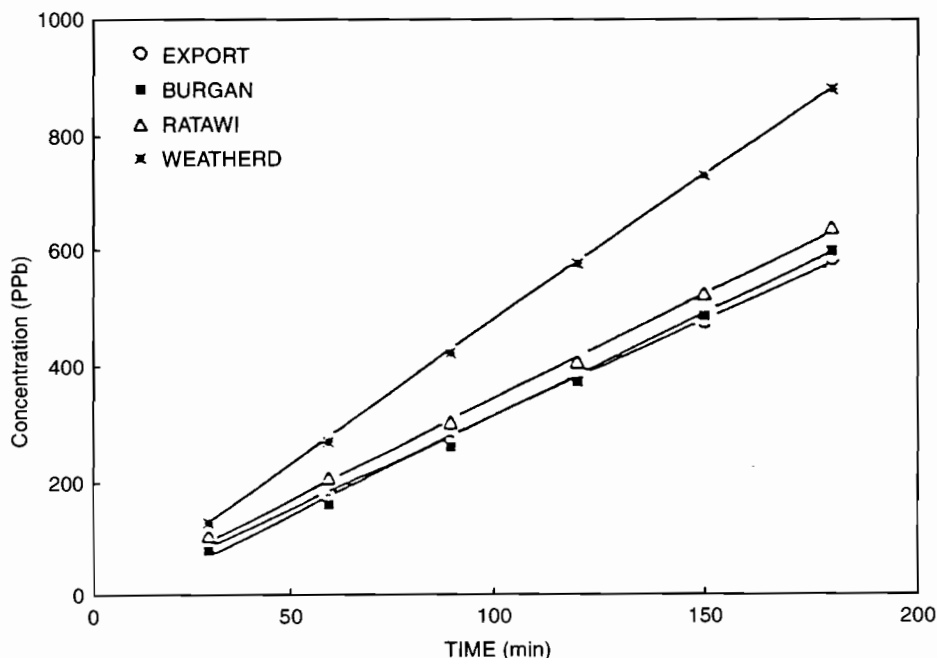


Fig. 3. Nickel concentration (ppb) versus time using nitric acid (pH = 6) as eluent.

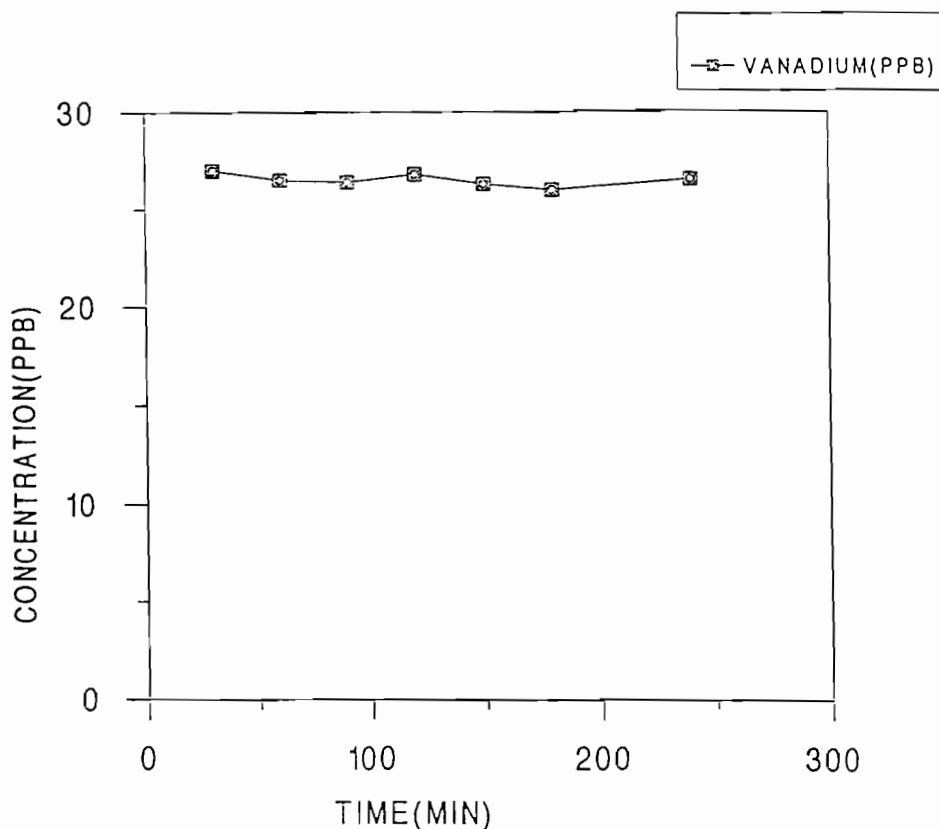


Fig. 4. Vanadium concentration (ppb) versus time using nitric acid (pH = 6) as eluent.

as a leachant. All plots have a negative slope. Slopes with more negative values are obtained for lighter crudes (Kuwait Export in the case of vanadium); vanadium has a high mass transfer coefficient in the light crudes and lower mass transfer coefficients in the heavy crudes. Table 2 also reveals that in a few cases the mass transfer coefficient values obtained are less significantly affected by the type of leachant used. This is plausibly related to the chemical structural variation in the different crude oils (Speight 1990). The more complex the structural environment around a metal, the less would be its interaction with the acid. Another factor which has an effect on the mass transfer rates is the particle size distribution of the sand bed. Since the ratio of surface area to volume is smallest for the largest pores, the potential for attenuation in larger pores is the least. The mobility of a metal ion in such a case would be the highest.

Leaching experiments with immobilized wastes have shown that elution of ions from a solid matrix can be approximated by Fick's law of diffusion (Godbee *et al.* 1969; De Johnzhe *et al.* 1965). When the leaching process is described by Fick's diffusion equation, it can be shown that in the case of a semi-infinite slab, a plot of C/C_0 versus $T^{1/2}$ is a straight line (Crank 1956).

The diffusion coefficients were calculated from the equation

Table 2. Mass transfer coefficient values of the metal vanadium in crude oil samples.

Acid	Crude	Initial pH	$K_L \times 10^3$ $\text{Cm}^2 \text{min}^{-1}$ (Vanadium)
NITRIC ACID	KE	4	$0.138 \pm (.2 \times 10^{-4})$
		6	$0.97 \pm (.8 \times 10^{-5})$
	B	4	$0.09 \pm (.17 \times 10^{-4})$
		6	$0.78 \pm (.23 \times 10^{-4})$
	R	4	$0.31 \pm (.11 \times 10^{-4})$
		6	$0.3 \pm (.67 \times 10^{-5})$
SULPHURIC ACID	W	4	$0.1 \pm (.48 \times 10^{-5})$
		6	$0.3 \pm (.11 \times 10^{-4})$
	KE	4	$0.48 \pm (.22 \times 10^{-6})$
		6	$6.4 \pm (.16 \times 10^{-4})$
	B	4	$1.23 \pm (.23 \times 10^{-5})$
		6	$0.23 \pm (.17 \times 10^{-5})$
ACETIC ACID	R	4	$0.37 \pm (.16 \times 10^{-4})$
		6	$0.12 \pm (.17 \times 10^{-4})$
	KE	4	$1.45 \pm (.45 \times 10^{-4})$
		6	$0.42 \pm (.24 \times 10^{-4})$
	B	4	$1.33 \pm (.47 \times 10^{-5})$
		6	$0.99 \pm (.34 \times 10^{-4})$
DEIONIZED WATER	R	4	$0.82 \pm (.12 \times 10^{-4})$
		6	$0.51 \pm (.1 \times 10^{-5})$
	W	4	$0.04 \pm (.17 \times 10^{-4})$
		6	$0.4 \pm (.22 \times 10^{-4})$
	KE	7	$0.02 \pm (.8 \times 10^{-5})$
		B	7
R	7	$0.01 \pm (.3 \times 10^{-4})$	
W	7	—	

$$\frac{C}{C_o} = \frac{A}{V} \frac{2D^2}{\pi} T^{-1} \quad (4)$$

where C , C_o , A , V and T are as defined above and D represents diffusion coefficient in $\text{cm}^2 \text{day}^{-1}$. Table (3) shows the diffusion coefficient values of vanadium.

Figures 8 and 9 are typical plots obtained through regression analysis for the estimation of diffusion coefficients. Nitric and sulfuric acids are good eluents for weathered crude oil, while acetic acid is good for Ratawi. Globally, nitric acid exhibits the maximum leaching rate for all the crude oils.

The role of a polar medium (de-ionized water) was studied. The experiments were conducted in an identical manner to experiments using acid as eluent. The metal concentration was not affected with time, thus ruling out the possibility of leaching using water. Figure (10) is a typical plot used for estimation of the mass transfer and diffusion coefficient of vanadium using de-ionized water. (The plot is almost parallel to the x-axis.) The mass transfer and diffusion coefficient values for the metal which is too low is shown in Tables 2 and 3.

The diffusion values were used for estimating the leaching behaviour over long

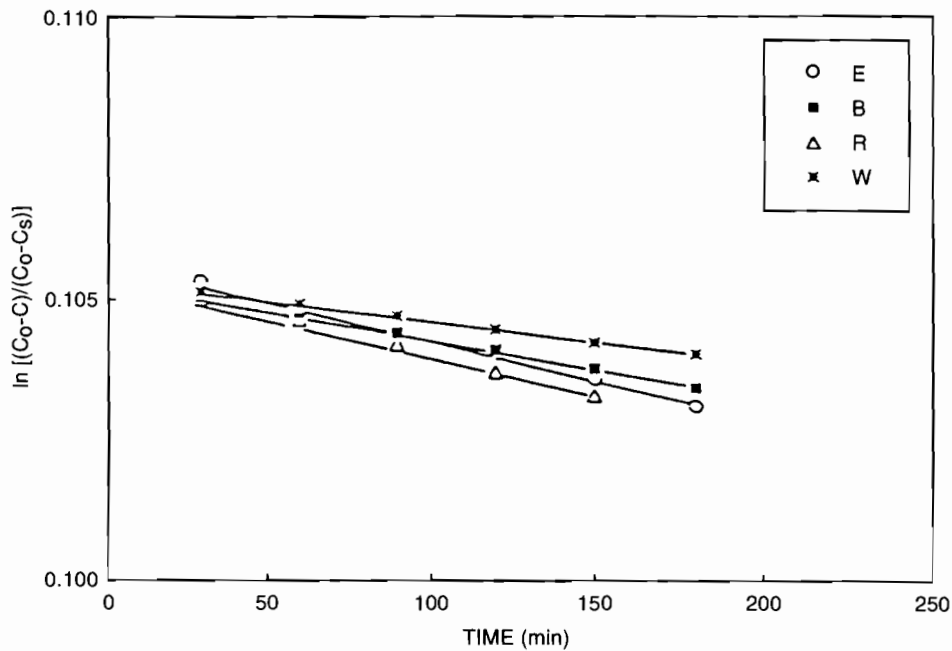


Fig. 5. Nickel and vanadium concentration (ppb) versus time using NaNO_3 (5% W/V) as eluent.

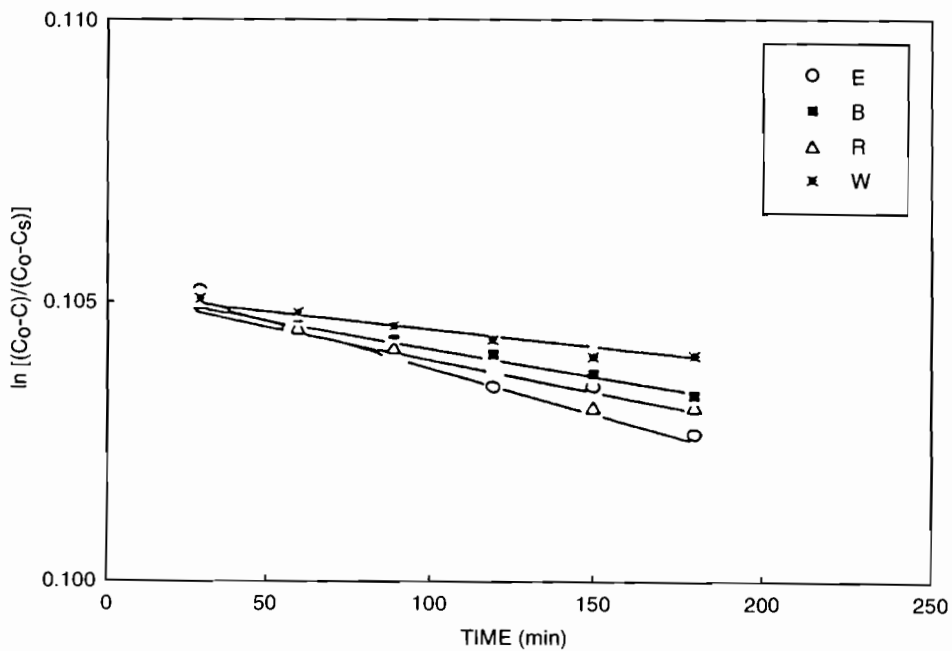


Fig. 6. Mass transfer of vanadium by sulphuric acid (pH = 4) from oil contaminated soil.

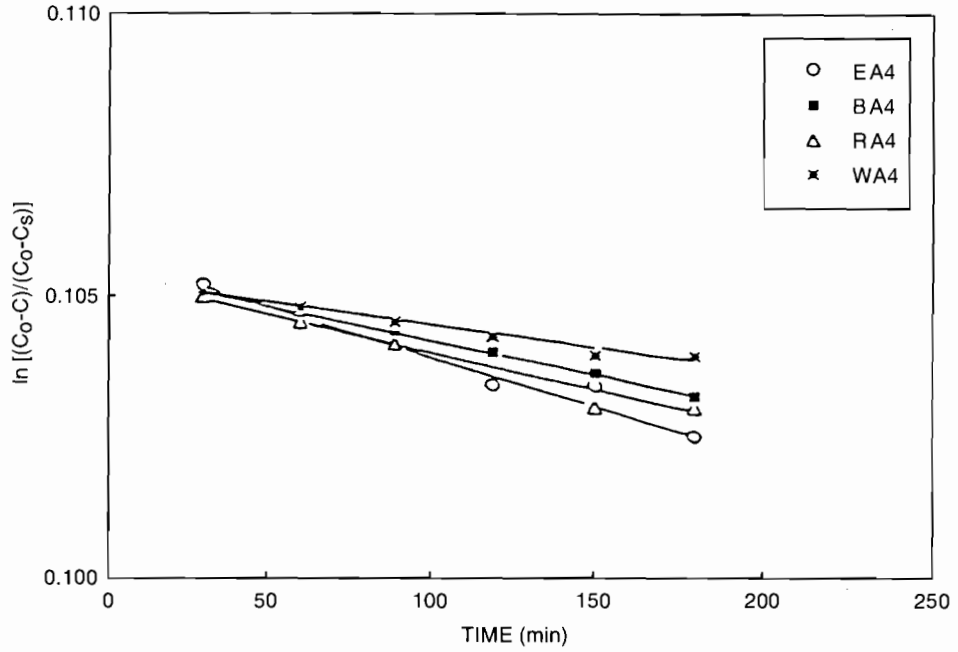


Fig. 7. Mass transfer of vanadium by acetic acid (pH = 4) from oil contaminated soil.

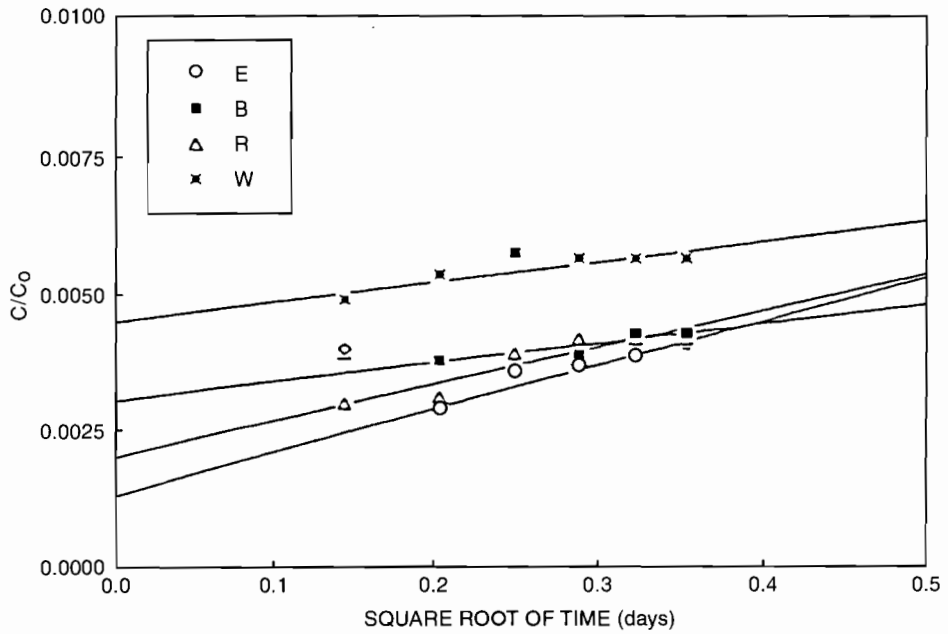


Fig. 8. Mass transfer of vanadium by nitric acid (pH = 6) from oil contaminated soil.

Table 3. Diffusion coefficient values of the metal vanadium in crude oil samples.

Acid	Crude	Initial pH	Diffusion Coefficient of Vanadium $D \times 10^6$ $\text{Cm}^2 \text{ day}^{-1}$	Time for 10% leaching of Vanadium ($\times 10$ years)
NITRIC ACID	KE	4	$22.29 \pm (.2 \times 10^{-4})$	0.42
		6	$11.8 \pm (.5 \times 10^{-11})$	0.79
	B	4	$1.8 \pm (.13 \times 10^{-11})$	5.1
		6	$8.3 \pm (.2 \times 10^{-12})$	1.1
	R	4	$2.95 \pm (.2 \times 10^{-9})$	3.1
		6	$2.4 \pm (.2 \times 10^{-12})$	3.9
W	4	$0.17 \pm (.6 \times 10^{-12})$	54.6	
	6	$2.5 \pm (.12 \times 10^{-11})$	3.7	
SULPHURIC ACID	KE	4	$1.97 \pm (.2 \times 10^{-12})$	4.7
		6	$0.56 \pm (.11 \times 10^{-12})$	16.5
	B	4	$11.2 \pm (.17 \times 10^{-4})$	0.83
		6	$0.45 \pm (.2 \times 10^{-10})$	20.6
	R	4	$1.7 \pm (.14 \times 10^{-3})$	5.5
		6	$0.56 \pm (1.0 \times 10^{-11})$	16.5
W	4	$0.63 \pm (0.7 \times 10^{-12})$	14.7	
	6	—	—	
ACETIC ACID	KE	4	$0.2 \pm (.8 \times 10^{-11})$	46.4
		6	$2.4 \pm (.2 \times 10^{-12})$	3.8
	B	4	$12.7 \pm (.5 \times 10^{-12})$	07.3
		6	$12.9 \pm (.3 \times 10^{-11})$	0.72
	R	4	$15.8 \pm (.9 \times 10^{-12})$	0.58
		6	$7.5 \pm (.16 \times 10^{-11})$	1.2
W	4	$9.7 \pm (.3 \times 10^{-12})$	0.95	
	6	$5.8 \pm (.3 \times 10^{-11})$	1.6	
DEIONIZED WATER	KE	7	$0.6 \pm (0.8 \times 10^{-11})$	--
		7	—	—
	B	7	$0.075 \pm (0.6 \times 10^{-12})$	--
		7	$0.08 \pm (0.4 \times 10^{-11})$	--
R	7	—	—	
	7	—	—	

periods of time. The estimated time for a specific leaching fraction can be obtained from Eq. (4), where the notations have their usual significance. Estimates of the time required for 10% leaching of the original concentrations of the metal under the present experimental conditions are shown in Table (3). It should be noted that many factors such as the pH of rain water, ambient temperature, water content of soil and geological factors would have to be considered in utilizing the data for extrapolation purposes.

Using the analysis of variance (ANOVA) we tried to explore the significance of different experimental parameters in our leaching studies. The results showed that there exists a significant effect of the type of crude on metal concentration. The initial pH values of the leaching acids also have a significant effect on vanadium concentration. The leaching rates were enhanced as the hydrogen ion concentration increased. In our studies pH 4 indicated the highest mean concentration. The leaching time and the three solvents did not vary significantly in their effect on vanadium concentration.

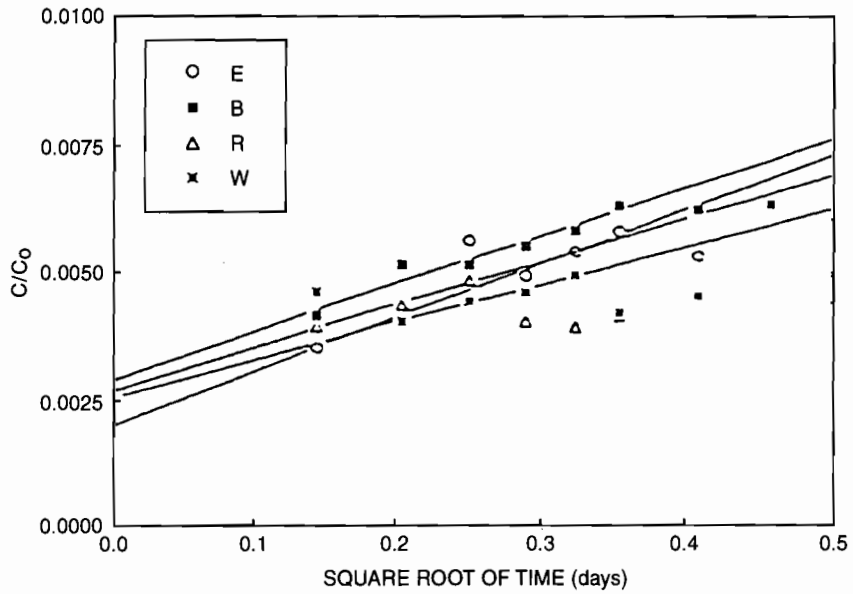


Fig. 9. Mass transfer of Vanadium by sulphuric acid (pH = 6) from oil contaminated soil.

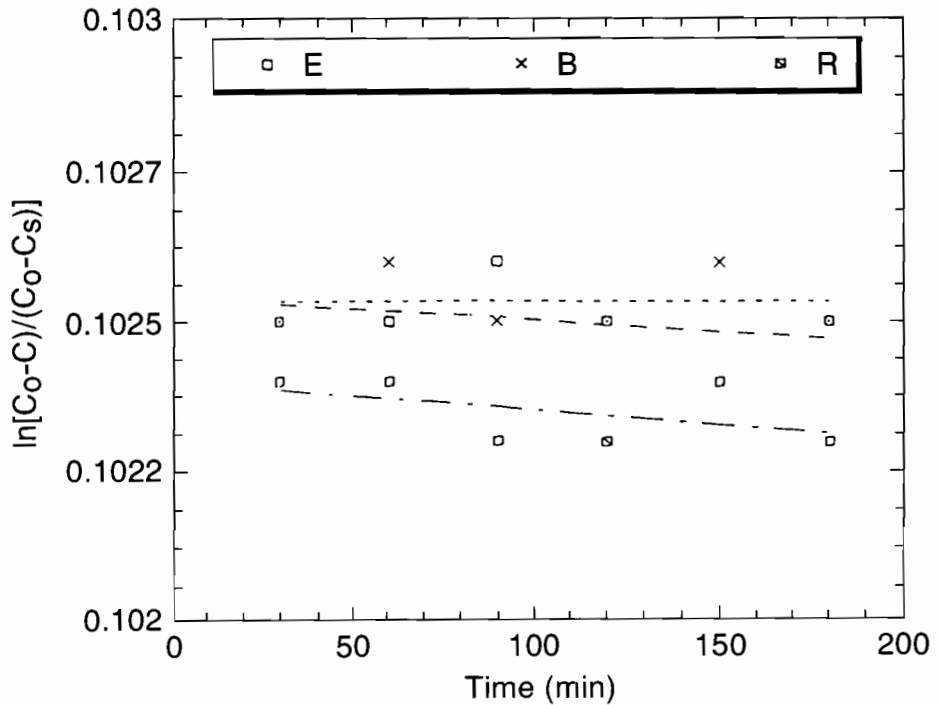


Fig. 10. Mass transfer of Vanadium by acetic acid (pH = 4) from oil contaminated soil.

Table 4. Ninety five percent confidence interval (CI) of vanadium concentration in different treatments.

Source		Number of Readings (N)	95% C.I. of Vanadium Concentration (ppb)
ACID:	NITRIC	49	129.90 ± 21.9
	SULPHURIC	51	110.90 ± 10.37
	ACETIC	61	127.90 ± 9.13
CRUDE:	K.E	46	116.57 ± 8.89
	B	48	129.23 ± 7.08
	R	46	109.39 ± 8.31
	W	45	137.3 ± 26.3
INITIAL pH:	4	82	137.39 ± 13.22
	6	79	115.03 ± 9.15

Nitric acid leached the highest amount of vanadium. The 95% confidence intervals (C.I.) of vanadium are indicated in Table 4.

CONCLUSIONS

The leaching behaviour of 4 different crude oils was investigated. Three different acids (pH 4 and 6) were employed. It appears that an exchange reaction occurs between an acid and a metaloporphyrin complex. The mass transfer coefficient values of vanadium have been calculated. The mass transfer coefficient of vanadium are in the range of $0.04\text{--}6.4 \times 10^{-3} \text{ cm}^3 \text{ min}^{-1}$. The values reveal that sulphuric and acetic acids are effective eluents for vanadium at pH 4 and 6.

The diffusion coefficients were estimated using Fick's law of diffusion. The values for vanadium are in the range of $0.06\text{--}6.4 \times 10^{-7} \text{ cm}^2 \text{ day}^{-1}$. The diffusion coefficient values do not vary consistently with the pH of the leaching medium. The rates for 10% leaching of the heavy metals indicate that possibilities of ground water contamination do exist, under the present experimental conditions.

Analysis of variance of the experimental data indicated that the hydrogen ion concentration plays a crucial role in the metal-extraction studies. Time and the nature of the solvent did not have a pronounced effect on the concentration of the different metals.

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اسلوب مبسط لتقدير معاملات انتقال المادة والانتشار للفانيدوم في النفط الخام الكويتي: دراسة الانتزاع الاختياري

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خلاصة

إن أحد الاهتمامات البيئية الرئيسية التي عقيبت الغزو العراقي على دولة الكويت هي إحتفال تلوث المياه الجوفية نتيجة السكب الهائل للنفط. وتعتبر مادتي النيكل والفانيدوم من المعادن الرئيسية التي توجد في النفط الخام بكميات ضئيلة. ولقد تم عمل دراسة مخبرية لانتزاع (أو ترشيح) النيكل والفانيدوم من أربعة زيوت نفط خام مختلفة هي نفط الكويت للتصدير (KE)، وبرقان (B)، ورطوة (R) و نفط خام تغير بفعل الطقس الجوي (W). واستخدمت محاليل أحماض النيتريك، والكبريتيك، والاستيك (بتراكيز 4-6 PH) كأوساط للانتزاع. كما تم دراسة دور الوسط القطبي مثل الماء المقطر الغير مؤين. وبينت الدراسة انه في حالة الوسط الحمضي، فإن معدل الانتزاع يعتمد اعتماداً فاعلاً على نوعية النفط الخام، ودرجة تركيز وسط الانتزاع.

وقد تراوحت قيم معامل انتقال المادة للفانيدوم ما بين 0.04×10^{-3} سم³/دقيقة. وتراوحت قيم معامل الانتشار للفانيدوم ما بين 0.06×10^{-7} سم²/اليوم. وتبين الدراسة الشاملة لهذه المعاملات ان حمض النيتريك هو أكثر هذه الاوساط الحمضية فاعلية في انتزاع الفانيدوم من هذه النفوط الأربعة. كما ساعد استخدام الماء المقطر الغير مؤين كوسط قطبي هذه الدراسة ايضاً تقديراً للفترة الزمنية المطلوبة لانتزاع 10% من كمية الفانيدوم من النفط ومن النتائج الرائدة لهذه الدراسة هو تحديد الزمن لانتزاع 10% من التركيز الأولي والذي تبين أنه يتراوح ما بين 40-546 سنة.

وتقدم الدراسة تحليلاً للمتغيرات لأهم المعاملات المختلفة في هذه التجربة المخبرية، وتقديراً لمستوى الثقة لقياسات التركيز، وقد اعتبرت متغيرات اساسية مثل نوع الحمض، درجة تركيزه، ونوع النفط الخام. وقد بينت الدراسة الاحصائية ان مقدرة المحاليل الحمضية على انتزاع المعادن تتغير مع نوع النفط الخام، وان هناك علاقة بين التركيز الأولي للأحماض ودرجة تركيز المعادن في المحاليل. وكانت افضل النتائج لانتزاع المعادن باستخدام المحاليل الحمضية ذات تركيز يعادل (PH=4).

