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Research Article

Investigation into the possibility of using a novel ionic liquid leaching method to obtain vanadium from vanadium slag

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ABSTRACT

An environmentally friendly, novel method for extracting vanadium from slag with two imidazolium-based ionic liquids, namely1-butyl-3-methylimidazolium trifluoromethane sulfonate [$Bmim^+CF_3SO_3^-$], and 1-butyl-3-methyl-imidazolium hydrogen sulfate [$Bmim^+HSO_4^-$], before and after magnetic separation, was proposed in this study. The effect of the type of ionic liquid, the concentration of ionic liquid, temperature and liquid to solid ratio were examined. The optimum experimental condition after examining these influential factors was found to be: ionic liquid [$Bmim^+HSO_4^-$] 50% (v/v), a leaching temperature of 100 °C, and a liquid to solid ratio of 12 ml/g (12:1), at a leaching time of 90 min, with shaking speed of 250 rpm. The results indicated that a maximum vanadium extraction of 94.2% with 57.6% iron as an impurity could be achieved from the non-magnetic slag fraction. The kinetics of vanadium dissolution in 50% (v/v) [$Bmim^+HSO_4^-$] was governed by a surface chemical reaction with an estimated E_a= 65.5 kJ/mol.

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INTRODUCTION

Vanadium is one of the most important base metals and can be found in different primary and secondary resources worldwide [1]. The main raw material for the extraction of vanadium is titanomagnetite ore, which accounts for about 85% of vanadium production [2]. During the steelmaking process, vanadium is concentrated in a high-grade slag, which is a by-product of the process. The high-grade slag is widely used as a secondary raw material to extract vanadium [3]. The base metal is resistant to corrosion, has high fatigue resistance, and increases the hardness and tensile strength of steels to which it is added [4]. Therefore, vanadium has been used in the production of high-strength steels, as electrolytes in redox flow batteries, catalysts, and also in ferrous and non-ferrous alloys. In addition to these uses, vanadium as a critical raw material is used in electric mobility, defense and space applications. It also enables the transition to renewable energy sources by its use in long-duration energy storage (LDES) solutions [5].

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The commonly used process for the extraction of vanadium from both vanadium slag and titanomagnetite ore is the sodium roast-leach method. In some cases, a mixture of the three sodium salts (NaCl, Na_2CO_3, and Na_2SO_4) is added to the filtered cake and the mixture is fed to a rotary kiln to be roasted at a temperature range of 700–1200 °C. The roasted ore or slag is then leached using water to form the pregnant solution, from which vanadium can be precipitated as ammonium metavanadate (AMV), using ammonium sulphate ($(NH_4)_2SO_4$) [6]. This process is cost-effective, since the reagents used during the roasting stage are relatively cheap. Additionally, the sodium salt used in the process is selective towards vanadium in the ore or slag to form sodium metavanadate ($NaVO_3$), but some impurities i.e. Fe, S, K, Cr can also be extracted. Using water as a lixiviant gives the process the advantage of a relatively high vanadium extraction of 60–85% at a controlled pH of 5. To improve vanadium extraction during the leaching stage, water can be substituted by sulphuric acid [6].

Chen et al. [7] investigated roasting with NaOH molten salt for titaniferous magnetite ore. In the sodium roasting process, the sodium oxidation forms the vanadium oxides to water-soluble sodium vanadate and concurrently the reduction of iron oxides to metallic iron also occurred. Subsequently, water leaching achieved a satisfactory vanadium extraction of 84.5%, iron 89.4%, and titanium 95.6%. In another research, Zhang et al. [8] proposed substituting the sodium salts with calcium oxide for roasting at 850 °C for 2.5 h, followed by sulphuric acid leaching, precipitation, and calcination to recover vanadium pentoxide from the ore. The achieved vanadium extraction under these conditions was 93.3%.

Li [9] examined a calcium roasting method, followed by ammonium carbonate $(NH_4)_2CO_3$ leaching. The method succeeded to be selective towards vanadium, hence, reducing the amount of impurities, and AMV is formed directly during the leaching stage. A maximum vanadium extraction of 96.0% was obtained with this process. Li et al. [10] substituted sodium salts with potassium salts, followed by sulphuric acid leaching. The conditions were as follows: potassium salt roasting at a temperature of 900 °C for 1 h, 10% (v/v) sulfuric acid concentration, liquid to solid ratio of 3 ml/g, leaching temperature 95 °C, and leaching time 1.5 h, which yielded an extraction efficiency of 71.4% for vanadium. Liu & Meng [4] decreased the amount of impurities leached when using non-salt roasting, followed by alkaline leaching.

However, these modifications do not properly address the disadvantages of the roast-leach process, such as (a) emission of toxic and corrosive gases, i.e. hydrogen chloride (HCl), chlorine (Cl_2), and sulfur dioxide (SO_2) [1], (b) high energy consumption in the roasting step, (c) extraction of impurities i.e. S, K, Cr, during leaching which increase capital costs in the refinery stage [6–8].

To overcome these problems, this investigation aimed to examine the feasibility of imidazolium-based ionic liquids (ILs), such as 1-butyl-3-methylimidazolium trifluoromethane sulfonate [$Bmim^+CF_3SO_3^-$], and 1-butyl-3-methyl-imidazolium hydrogen sulfate [$Bmim^+HSO_4^-$], to extract vanadium efficiently from vanadium slag with high iron content, without including a roasting step. Some reports suggested [11] that imidazolium-based ionic liquids can decrease the reaction time and enhance the recovery yield. While the extraction of other metals such as gold, silver, copper, titanium, and aluminium by using the imidazolium-based ionic liquid (IL) have been investigated, few studies could be found in the literature on vanadium extraction by ILs. Only Bell & Castleman [12] reported an unsuccessful attempt to leach vanadium oxide using 1-butyl-3-methylimidazolium trifluoromethane sulfonate [$Bmim^+CF_3SO_3^-$]. This investigation focused to identify which IL results in the maximum extraction of vanadium from V-bearing slag before and after magnetic separation and to establish under which conditions it could be achieved. To this end, two different ILs, their varying concentrations, various liquid to solid ratios and a number of leaching temperatures were investigated. These two ILs were selected to examine the effect of ionic liquids having the same cationic part as [$Bmim^+$] and different anionic parts as [$CF_3SO_3^-$] and [HSO_4^-] to extract the target metals.

MATERIALS AND EXPERIMENTAL PROCEDURE

Materials

The vanadium slag containing a high metallic iron content was supplied by Powder Tech, a company located in Brits in South Africa. The ILs 1-butyl-3-methylimida-zolium trifluoromethane sulfonate [$Bmim^+CF_3SO_3^-$] (IL-A), 1-butyl-3-methyl-imidazolium hydrogen sulfate [$Bmim^+HSO_4^-$] (IL-B), and sulphuric acid (98%) were bought from ACE Chemicals (Pty.) Ltd. These two IL have been selected since other researchers used them in their work also, and that facilitates comparisons. Moreover, the IL-B [$Bmim^+HSO_4^-$], is able to act as a Brønsted acid by releasing proton [H^+] into an aqueous solution owing acidic properties [13].

Experimental Procedure

The slag was crushed using a jaw crusher and then milled into a fine powder using a Zieb pulverizing mill. The initial particle size of the slag was ~20 mm, then after crushing in the jaw crusher become ~1mm. To make the crushed material finer, they were put in the Zieb pulverizing mill which milled them to a fine powder with a particle size of D_{80}= 45 µm. The bulk chemical composition of the slag is presented in Table 1, as measured by X-ray fluorescence (XRF). XRF powder method was carried out on a Rigaku-ZSX Primus II instrument using SQX analysis software. The instrument settings during operation was 60 kV and 150 mA.

Table 1. Bulk chemical composition of vanadium slag (wt.% except V* in ppm)

Component	Fe_2O_3	SiO_2	TiO_2	MnO	Cr_2O_3	CaO	Al_2O_3	P_2O_5	V*	LOI
Content (wt.%)	46.03	17.94	13.38	10.91	1.61	1.53	1.25	0.12	163.04*	7.1

Table 2. Range of investigated experimental parameters

Parameters	Studied range				
Type of IL	[$Bmim^+CF_3SO_3^-$] (IL-A)			[$Bmim^+HSO_4^-$] (IL-B)	
IL concentration (v/v)	10%	20%	30%	40%	50%
Temperature (°C)	45	60	75	90	100
Liquid to solid ratio (ml/g)	5	6	8	10	12

Slag samples were treated in a magnetic separator to obtain a magnetic and non-magnetic fraction for further leaching. Sulphuric acid solutions with concentrations of 15%, 30%, 50% and 70% (w/w) were used for the initial leaching runs. A 10 ml volume of the different acid concentrations was mixed with 1 g of slag sample to ensure a liquid-to-solid ratio of 10 ml/g (10:1). These samples were leached under reflux condition with a condenser attached to the leaching flask stirring at a speed of 250 rpm, for 90 min at 100 °C. After 90 min, the mixture was filtered, and a 5 ml aliquot of the filtrate was pipetted into a 100 ml volumetric flask and made up to the mark. These diluted samples were analysed using atomic absorption spectrometry (AAS-Varian model A2000) to measure vanadium and iron content.

To investigate the effect of the IL concentration, a 10 ml IL/distilled water solution (IL-A and IL-B) with concentrations of 10%, 20%, 30%, 40% and 50% (v/v), was mixed with 1 g slag (magnetic and non-magnetic portion) with liquid to solid ratio (10:1). Then the samples were placed in the shaker with a speed of 250 rpm, at a temperature of 100 °C, for 90 min. The effect of varying temperatures on the leaching process was evaluated by using 10 ml 50% (v/v) solution of IL/distilled water mixed with 1 g slag sample to ensure a liquid to solid ratio of 10 ml/g. These samples were placed in the ultrasonic bath for 60 min at 60 °C, before they were leached at the following temperatures: 45, 60, 75, 90, 100 °C in the shaker with a speed of 250 rpm, for 90 min. To study the effect of the liquid to solid ratio on the V and Fe extraction, IL-A and IL-B with 50% (v/v) concentration was mixed with specific amount of the slag sample (magnetic and non-magnetic) to make different liquid to solid ratios of 5, 6, 8, 10, 12 ml/g. Table 2 listed the investigating parameters and their studied range. All experimental runs were carried out in duplicate to ensure good repeatability and average values are reported.

It was assumed that the total amount of vanadium and iron contained in the sample is the amount leached using 70% (w/w) sulphuric acid. Recoveries of each element were calculated as follows:

$$\%Vanadium\ Extraction = [V\ leached\ by\ IL/\ V\ leached\ by\ H_2SO_4] \times 100 \quad (1)$$

$$\%Iron\ Extraction = [\mathrm{Fe}\ leached\ by\ IL/\ \mathrm{Fe}\ leached\ by\ H_2SO_4] \times 100 \quad (2)$$

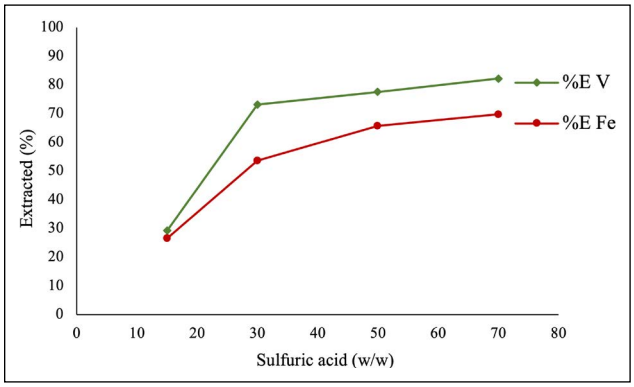


Figure 1. The effect of sulphuric acid concentration on V and Fe extracted from the vanadium slag sample. Conditions: liquid to solid ratio of 10 (mg/l), stirring speed 250 rpm, at 100 °C for 90 min.

RESULTS AND DISCUSSION

Vanadium and Iron Extraction Before Magnetic Separation

To determine the vanadium and iron before magnetic separation, sulphuric acid solutions with concentrations of 15%, 30%, 50% and 70% (w/w) were used for the initial leaching runs. A 10 ml aliquot of the different acid concentrations was mixed with 1 g of slag sample to ensure a liquid to solid ratio of 10 ml/g (10:1). These samples were leached for 90 min in a shaker, at a temperature of 100 °C. Then the mixture was filtered, and a 5 ml aliquot of the filtrate was pipetted into a 100 ml volumetric flask and made up to the mark. These diluted samples were analysed using AAS to measure vanadium and iron content. Figure 1 displays the results obtained in terms of the concentration of vanadium and iron contained in the solution when the slag sample was leached using sulphuric acid at different concentrations. The maximum amount of vanadium and iron was 61 mg/l, and 384 mg/l, respectively from the slag sample before magnetic separation with 70% sulphuric acid. These values were considered as the total amount of vanadium and iron in the slag and were used when calculating percentage recoveries.

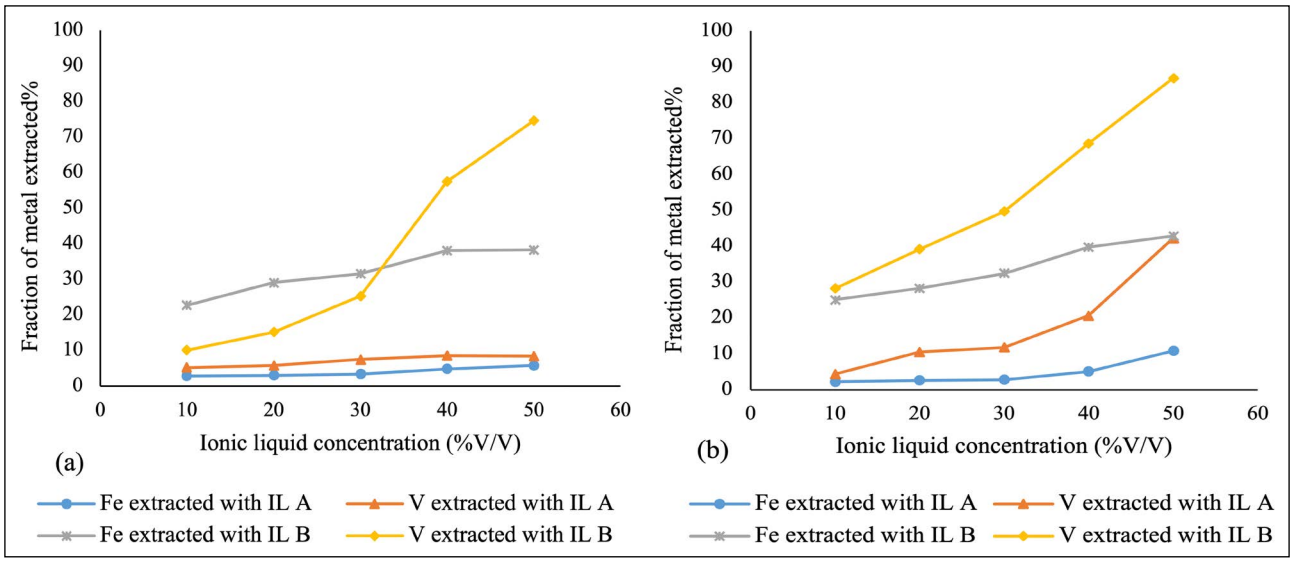


Figure 2. The effect of the IL (A & B) concentration on V and Fe extraction from **(a)** magnetic and **(b)** non-magnetic slag. Conditions: liquid to solid ratio of 10 (mg/l), stirring speed 250 rpm, at 100 °C for 90 min.

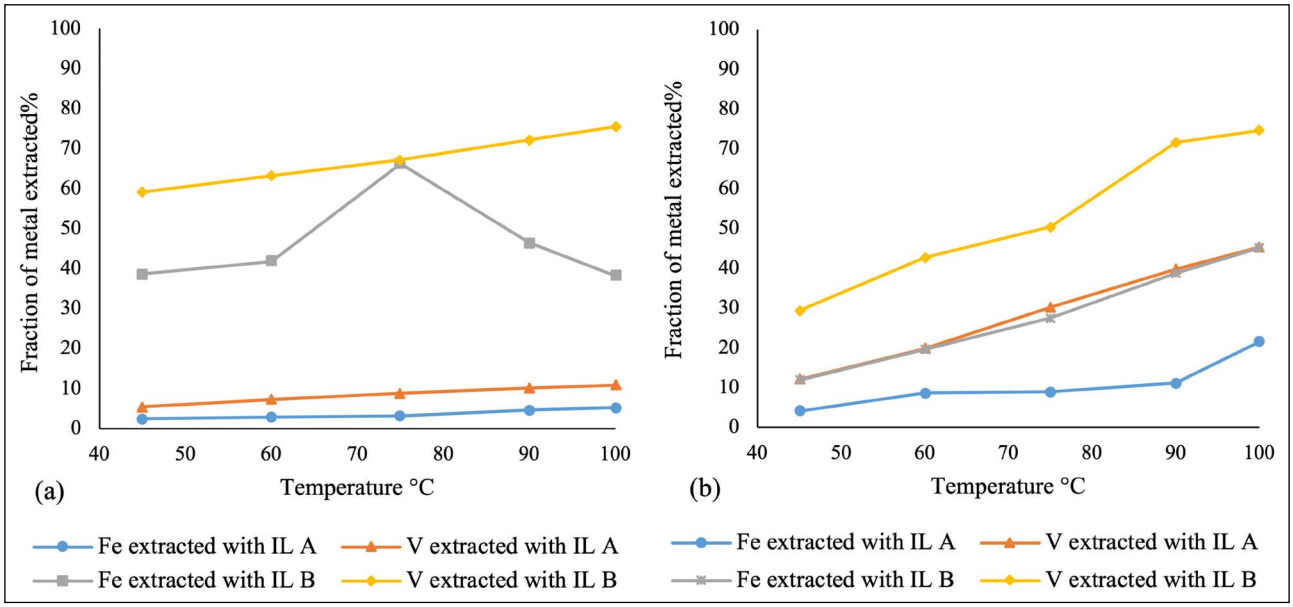


Figure 3. The effect of temperature from 45 to 100 °C on V and Fe extraction from **(a)** magnetic and **(b)** non-magnetic slag. Conditions: 10 ml IL (A & B) 50% (v/v) solution, liquid to solid ratio of 10 (mg/l), stirring speed 250 rpm, for 90 min.

Vanadium and Iron Extraction From the Magnetic and Non-Magnetic Slag Portions

Two different ILs were used to leach the magnetic and non-magnetic portions of the slag sample to investigate how the IL concentration, the liquid to solid ratio and the leaching temperature affect the recovery of vanadium and iron. Analyses indicated that the magnetic portion of the slag contained at least 10 times the amount (mass) of V compared to the non-magnetic portion, while the mass of Fe was at least twice as high in the magnetic portion as in the non-magnetic portion of the slag after separation.

The Effect of Ionic Liquid Concentration

Two different ILs [$Bmim^+CF_3SO_3^-$] (IL-A), and [$Bmim^+HSO_4^-$] (IL-B) were used to leach the magnetic and non-magnetic portions of the slag sample to investigate the effect of the IL concentration from 10% to 50% (v/v) on vanadium and iron extraction. The results are illustrated in Figure 2a, b. With 50% IL-B, 74.6% V and 38.7% Fe was extracted from the magnetic slag. It is evident that the magnetic separation increased the extraction of V to 86.9%, and Fe to 42.9% (Fig. 2b - non-magnetic slag). In comparison to IL-B, IL-A resulted in substantially less V and Fe with only 8.4% and 5.8% ex-

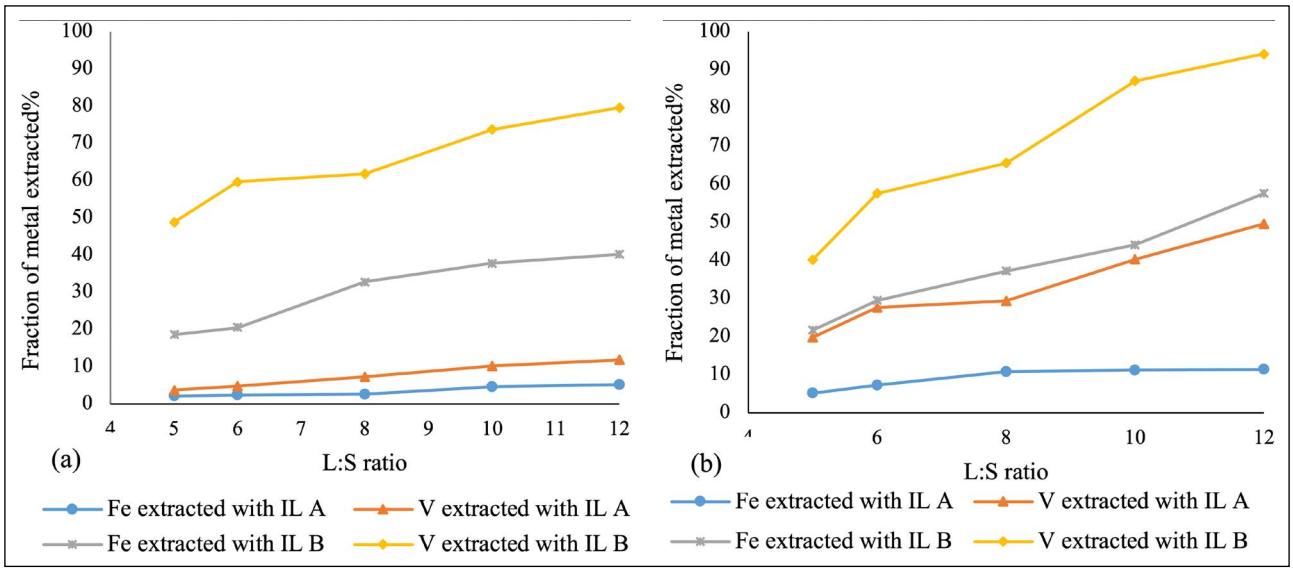


Figure 4. The effect of the solid-to-liquid ratio in the range of 5, 6, 8, 10, and 12 ml/g on V and Fe extraction from **(a)** magnetic and **(b)** non-magnetic slag. Conditions: 10 ml IL (A & B) 50% (v/v), stirring speed 250 rpm, at 100 °C for 90 min.

Table 3. Summary of V and Fe extraction with IL-B ($Bmim^+HSO_4^-$)

Studied factors	IL-B 50% (v/v)		Temperature 100 °C		L:S ratio (12:1)	
Extraction	%V	%Fe	%V	%Fe	%V	%Fe
Magnetic portion	74.6	38.3	75.5	38.2	79.6	40.2
Non-magnetic portion	86.9	42.9	74.6	45.1	94.2	57.6

traction, respectively from the magnetic slag portion. However, with IL-A, V and Fe extraction improved to 42.8% and 10.8%, respectively from the non-magnetic slag. Therefore, 50% (v/v) [$Bmim^+HSO_4^-$] (IL-B), was the better IL for V and Fe extraction from magnetic and non-magnetic slag portions.

The Effect of the Leaching Temperature

The effect of varying temperature on the leaching process was evaluated by using 10 ml of a IL at a concentration of 50% (v/v) with 1 g slag sample (magnetic and non-magnetic slag) to ensure a liquid to solid ratio of 10 ml/g. These samples were placed in the ultrasonic bath for 60 min at 60 °C, before they were leached at the following temperatures: 45, 60, 75, 90, 100 °C. As presented in Figure 3a, b, increasing the temperature generally leads to an increased V and Fe extraction for both the IL-A and IL-B. The reason is that, increasing the temperature improve the diffusion and the reaction rate leading to higher extraction. Thus, 100 °C was chosen as the optimum operating temperature. At 100 °C, IL-B extracted 75.5% V and 38.2% Fe from the magnetic portion of the slag. When considering the non-magnetic portion of the slag, the same IL-B achieved 74.6% V and 45.1% Fe at 100 °C. In contrast, IL-A extracted 10.8% V and 5.2% Fe from the magnetic slag, and 45.2% V and 21.5% Fe from the non-magnetic slag portions.

The Effect of Liquid-to-Solid Ratio

To examine the effect of the liquid-to-solid ratio, the IL-A and IL-B with 50% (v/v) concentration were mixed with a specific amount of the slag sample (magnetic and non-magnetic) to make different liquid-to-solid ratios of 5, 6, 8, 10, 12 ml/g. Figure 4a, b illustrates the effect of the solid-to-liquid ratio on V and Fe extraction from magnetic and non-magnetic slag. Generally, vanadium extraction increases with increasing the volume of the IL used for leaching. This is to be expected and in line with the mass transfer theory, which states that an increasing concentration gradient with an increasing liquid-to-solid ratio is the driving force for mass transfer [14]. A liquid-to-solid ratio of 12 ml/g (12:1) was therefore chosen as the optimum practical ratio. At this liquid-to-solid ratio IL-B yielded V and Fe of 94.2% and 57.6%, respectively from the non-magnetic portion of the slag (Fig. 4b), and slightly less from the magnetic portion (Fig. 4a) as 79.6% V and 40.2% Fe. With IL-A, 11.8% V and 5.2% Fe extraction were obtained from the magnetic slag, and it improved in the non-magnetic slag fraction to 49.6% V and 11.4% Fe.

A summary of the results on the optimum point for the studied factors with IL-B [$Bmim^+HSO_4^-$] which was the better IL for the magnetic and non-magnetic portion for V and Fe extraction is summarised in Table 3.

Kinetic Model

Leaching is a reaction between solid particles and liquid which is defined as a heterogeneous reaction. The reaction occurs at the interface of the leaching agent and a solid phase. The general leaching reaction can be defined as follow:

$$A_{(l)} + bB_{(S)} \rightarrow cC$$

Table 4. Shrinking core model equations as limiting step

	Limiting step	Equation	R^2	Rate constant
1	Diffusion through the product layer (SP)	$1 - 3(1 - X)^{2/3} + 2(1 - X) = k.t$	0.94	0.003
2	Diffusion through the product layer (CP)	$X + (1 - X)\ln(1 - X) = k.t$	0.95	0.004
3	Diffusion through the liquid film (SP)	$X = k.t$	0.99	0.009
4	Surface chemical reactions (CP)	$1 - (1 - X)^{1/2} = k.t$	0.99	0.006
5	Surface chemical reactions (SP)	$1 - (1 - X)^{1/3} = k.t$	0.98	0.004
6	New shrinking core model	$1/3\ln(1 - X) + [(1 - X)^{-1/3} - 1] = k.t$	0.87	0.001

*[SP = Spherical Particle & CP = Cylinder Particle]

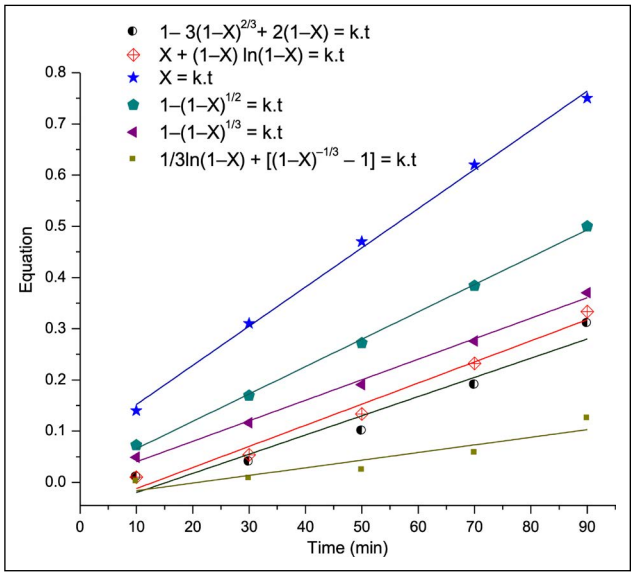


Figure 5. Different SCM model equation fittings.

Where A is the leaching agent (liquid), B is the solid phase (the particles), and C is the produced product(s). Typically, during leaching the following steps happen sequentially: diffusion of the leaching agent through the thin liquid film surrounding the solid particle, chemical reaction on the surface of the solid particle, and diffusion of the leaching agent through the product. The slowest step acts as a limiting step and controls the kinetics of dissolution [15].

To determine the kinetics of vanadium dissolution in 50% (v/v) IL-B ($Bmim^+HSO_4^-$), the experimental results at time intervals of 10, 30, 50, 70, and 90 min at 100 °C were evaluated using the shrinking core model. Considering the solid particles can be spherical and/or cylinder, the fitness of the different equations for diffusion, chemical reaction, and the new equation (mixed model) as a limiting step controlling the kinetics of vanadium dissolution were assessed.

To do so, the function of each equation versus time was plotted to obtain the correlation coefficient (R^2). Table 3 tabulated the list of the evaluated equations as a limiting step, the obtained correlation coefficient (R^2), and the rate constant. The trendline with R^2 close to one indicates the most suitable equation fitting to the experimental results. In this case, the surface chemical reaction (spherical and/or cylinder particles) controls the kinetics of vanadium dissolution in 50% (v/v) IL-B ($Bmim^+HSO_4^-$). Figure 5 illustrates the function and the fitness of each equation versus time.

Since the surface chemical reaction indicates the best fit of the results, the slope of the straight line presenting the functionality of the chemical reaction (Δ) in Figure 5, was taken as the rate constant (k) to estimate the activation energy as 65.5 kJ/mol. The value of the obtained activation energy is >40 kJ/mol, which is another indication that the surface chemical reaction is controlling the kinetics of the dissolution reaction [13].

CONCLUSIONS

The following conclusions can be drawn from this investigation:

- Of the two ILs which were used in this investigation, the aqueous solutions of the IL-B [$Bmim^+HSO_4^-$] emerged as a better extractant than the IL-A [$Bmim^+CF_3SO_3^-$].
- Magnetic separation concentrates the amount of vanadium and iron in the magnetic portion of the sample, leading to improved vanadium extraction, even though the recovery percentages of the non-magnetic fraction were higher than in the magnetic fraction.
- Under the investigated parameters, the optimum conditions for leaching were: IL-B [$Bmim^+HSO_4^-$] with 50% (v/v) concentration, a temperature of 100 °C, and L:S of 12:1.
- Maximum extraction of 94.2% V and 57.6% Fe was achieved from the non-magnetic portion of the slag sample.
- A surface chemical reaction controls the kinetics of vanadium dissolution in 50% (v/v) IL-B [$Bmim^+HSO_4^-$]
- The effectiveness of the different ionic liquids under different conditions to extract various amounts of V and Fe, coupled with magnetic separation of the material in two different fractions, offers the opportunity to design a process to separate the V and Fe in the slag for further recovery and purification.

- This study demonstrated the viability of a direct leaching method using imidazolium-based ILs to extract vanadium from the slag in yields comparable to traditional pyrometallurgical and hydrometallurgical means, with no emission of toxic gases, and considerably lower energy requirement compared to the conventional roast-leach method.

The study is incomplete in that it still needs to illustrate that:

- Recycling the ionic liquid to be reused in the extraction process would lead to less consumption than conventional acids in the leaching solution.
- Ionic liquids have large electrochemical "windows/ranges" which offer the potential for metal recovery and recycling after electrochemical recovery of V and/or Fe.

Follow-up work currently underway involves investigating the final separation of the V and Fe from the leach solution in such a way that the ionic liquid can be recycled. We also plan future work with other types of ionic liquids.

Acknowledgements
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DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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