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Optimization and Dissolution Kinetics of Vanadium Recovery from LD Converter Slag in Alkaline Media1

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Abstract—Alkaline roasting-alkaline leaching process was used to recover vanadium from LD (Linz Donawitz) converter slag. The independent leaching parameters investigated were liquid to solid ratio (L/S) (10– 20 mL/g), temperature (40–60°C), NaOH concentration (1.0–3.0 M), and time (60–120 minutes). Response surface methodology (RSM) was utilized to optimize the leaching parameters and as a result, the most influencing parameter was found to be liquid to solid ratio. Based on the results, the optimum recovery condition (approx. 99%) was obtained with L/S ratio of 20, temperature of 40°C, NaOH concentration of 3.0 M, and leaching time of 100 minutes, respectively. Furthermore, the kinetics of alkaline leaching process was investigated using shrinking core model (SCM) equations. It was found that the rate of vanadium leaching is controlled by a mixed controlling mechanism which is comprised of chemical reaction and diffusion through the solid product layer.

Keywords: ld converter slag, vanadium, alkaline leaching, kinetics, shrinking core model **DOI:** 10.3103/S1067821216050126

INTRODUCTION

Extensive studies have been conducted on recovery and recycling of metal species from waste materials due to the need for waste disposal minimization in various industries [1]. As a result of the presence of 12– 25% vanadium pentoxide in steelmaking slag, vanadium-bearing slag is considered as one of the most important sources of vanadium which is extensively used as an alloying element to enhance mechanical properties such as tensile strength and fatigue resistance in ferrous and non-ferrous industries [2–5].

To recover vanadium from various wastes such as spent catalysts and steelmaking slag different processes have been proposed among which a class of pyrohydrometallurgical processes comprising alkaline roasting with NaCl or $Na₂CO₃$ followed by leaching is the most widely used method [2]. Xin-sheng et al. [3] utilized an oxidation process -in the presence of $Na₂CO₃$ —as a roasting step for a low grade vanadium slag to form water—soluble sodium vanadate compounds during the leaching process. Aarabi-Karsagani et al. [6] observed that a roasting stage is necessary to

convert insoluble calcium oxide compounds to

A multi-step process including roasting, acid leaching and solvent extraction for recovery of vanadium from steelmaking slag has been applied by Gupta and Krishnamurthy [5]. In their investigation, vanadium calcium oxide can be dissolved and converted to a soluble compound by means of sulfuric acid solution based on the following reaction.

$$
Ca_2V_2O_7 + 3H_2SO_4
$$

\n
$$
\rightarrow (VO_2)_2SO_2 + 2CaSO_4 + 3H_2O.
$$
 (1)

Instead of applying roasting step in the recovery process to increase the acid leaching efficiency, sodium chlorate or manganese dioxide as oxidants can be utilized [7]. Although acid leaching is an effective ¹ The article is published in the original. **1** The article is published in the original. **1** The article is published in the original.

sodium oxide compounds which can be dissolved by an acidic leaching process. Moskalyk and Alfantazi [4] reported a process which involved a roast/leach operation using multi-hearth furnaces. They demonstrated that during the roasting process, vanadium pentoxide was converted to sodium vanadate by adding sodium carbonate, sodium chloride, or sodium sulfate and finally, sodium vanadate was leached out by water.

Fig. 1. X-ray diffraction pattern of the slag.

presence of small amounts of impurities in the resulting solution would cause some problems in the later precipitation process for vanadium recovery in the form of vanadate salts. Using a carbonate bearing material as oxidant or an alkaline media for vanadium leaching from roasted slag can overcome the mentioned problem [5]. Chen et al. [8] and Mahdavian et al. [2] reported a roast/leach process including the alkaline leaching of roasted slag to recover vanadium from LD slag. They utilized sodium hydroxide and sodium carbonate as leachants and recovered more than 80% of vanadium from LD slag.

Due to the need for applying the most efficient hydrometallurgical processes which are not obtainable by means of common experimental methods, statistical methods such as design of experiments (DOE), genetic algorithm, and neural network have been examined during recent years. By the mentioned means, operational time and expenses are decreased and efficiency will be enhanced [9, 10]. In comparison to both genetic algorithm and neural network methods which require a large number of experiments and consequently consume more energy and time, DOE methods using less energy and time provide more favorable results with a limited number of experiments [11, 12].

Furthermore, since interactions between parameters are ignored using current optimization methods such as Taguchi and factorial design, these methods are considered as low efficient methods and in order to overcome this problem, response surface methodology (RSM) has been explored recently [13–15]. This method is precise enough to obtain reliable results and also interactions between involved parameters can be quantified by applying RSM [15]. To build a second order (quadratic) model for the response variable in RSM method, central composite design (CCD) as an experimental design was applied to collect data for fitting the second order response [15]. Moreover, the necessity of a complete three-level factorial experi-

ment can be ignored by utilizing CCD method for an optimization process.

To the best of our knowledge, and in spite of the mentioned experimental studies, very few investigations have been published on the optimization of vanadium leaching from steelmaking slag in alkaline media. Also, there is a knowledge gap on kinetic aspects of vanadium alkaline leaching from steelmaking slag which would be vital for designing and applying an efficient recovery process for vanadium from various industrial wastes specially steelmaking slag. The main objective of the present investigation was to optimize vanadium alkaline leaching parameters such as liquid to solid ratio (L/S) , leaching temperature, NaOH concentration and leaching time from roasted LD converter slag by means of RSM method. Furthermore, kinetic aspects of alkaline leaching of vanadium were investigated and the controlling mechanisms of vanadium dissolution were identified using shrinking core model.

EXPERIMENTAL PROCEDURE

Slag Sample

The LD converter slag used in this study was obtained from disposal sites of waste materials of Esfahan Steel Company, Iran. The sample was ground and sieved to less than 0.850 mm in diameter as mentioned elsewhere [6]. Mineralogical and chemical analyses of the sample were carried out by X-ray diffraction (XRD) analyzer (Philips, Netherlands) and X-ray fluorescence (XRF) spectroscopy (S4explorer, Germany). Figure 1 reveals that vanadium presents in the form of $Ca_2V_2O_7$ and the other major phases in the slag sample composition are calcium hydroxide $(Ca(OH₂),$ calcium iron oxide $(CaFeO₂)$, and calcium silicate ($Ca₃SiO₅$). The XRF analysis of the slag sample is shown in Table 1 and it can be seen that the sample contains 1.12% of vanadium equivalent to 1.97% V_2O_5 .

Alkaline Roasting

To convert vanadium compound to a more soluble form, alkaline roasting process was applied using 20 wt % sodium carbonate (Na_2CO_3) as roasting agent at 1000°C for 2 hours [16]. Results of the XRD analysis after alkaline roasting process are shown in Fig. 2. According to Figs. 1 and 2, roasting process converts insoluble calcium vanadate $(Ca_2V_2O_7)$ to sodium vanadate ($NaVO₃$) as a more soluble compound in alkaline media [5]. The following reaction occurs in this step:

$$
Ca2V2O7 + Na2CO3
$$

\n
$$
\rightarrow 2NaVO3 + 2CaO + CO2.
$$
 (2)

Alkaline Leaching

Using a Pyrex reactor equipped with a reflux condenser, the leaching experiments were performed at atmospheric pressure. The reaction mixture was heated on a hot plate indirectly, through a water bath and it was agitated using a magnetic stirrer. For each leaching test, the roasted sample and predetermined concentrations of sodium hydroxide were charged into the reactor and the mixture was well mixed at 600 rpm at certain temperature, S/L ratio, and time after the leaching process, the slurry was filtered and analyzed by using the titration method to determine the vanadium contents. Also, some samples were analyzed by atomic absorption spectroscopy (Perkin-Elmer 2100—USA) to check the titration results. The leaching reaction is:

$$
\text{NaVO}_{3(s)} + \text{NaOH}_{(aq)} \rightarrow \text{Na}_2\text{VO}_3\text{OH}_{(aq)}. \tag{3}
$$

Leaching Experimental Design

Design of experiments based on a central composite design (CCD) with five levels and response surface methodology (RSM) was used to obtain an empirical model and describing the optimum level of parameters and operating conditions. In the current study, $2⁴$ factorial design and six $(N_0 = 6)$ central replicates were employed in CCD and duplication of the center points was applied to determine the experimental error. In order to minimize the effect of systematic errors, the experiments were carried out in a random order. Thirty experiments were performed corresponding to the four variables of the central composite design (Tables 2 and 3) and the parameter levels and response values (V recovery) are given in Tables 2 and 3. In order to perform regression and graphical analysis of the obtained data, the DESIGN EXPERT 8.0.1 trial version software (State-Ease Inc., Minneapolis, MN, USA) was utilized.

KINETIC MODELING

Determining the kinetics is an essential step in designing a chemical process such as vanadium recovery from LD converter slag. Based on numerous studies on different systems such as oxidation, reduction, roasting and regeneration, it can be concluded that the shrinking core model (SCM) as a simple model can represent the majority of reacting fluid-solid systems [6, 17–19]. The reaction for which the kinetics was determined in this work is reaction (3). The ratio of stoichiometric coefficients of sodium vanadate to sodium hydroxide is 1 in this reaction and is shown by b in the followings. As described by Levenspiel [18], in a fluid-solid reaction system the leaching rate of spherical particles of unchanging size may be controlled by one of the following sequential steps, the slowest of which step is the rate controlling step [18]:

Table 1. XRF results of chemical composition of the slag Compound wt % $Na₂O$ 18.78 MgO 0.98 Al_2O_3 1.24 $SiO₂$ 6.88 P_2O_5 1.91 SO_3 0.79 K_2O 0.18

Table 2. Parameter levels used in the experimental design

 V_2O_5 1.97 MnO 1.52 $Fe₂O₃$ 17.57

 $*\sigma = 2.0$, $\sigma^* = (2^k - 9)^{1/4}$ for a central composite rotatable design, $k =$ number of factors studied, $-q =$ fraction of the number of factors (where $q = 0$ for full factorial design).

(a) Diffusion of the leaching agent through the liquid film surrounding the particle to the surface of the solid.

(b) Diffusion of the leaching agent through the produced solid layer at the surface of the unreacted core.

Fig. 2. X-ray diffraction pattern of the roasted slag at 1000°C (roasting time: 2 h, sodium carbonate: 20 wt %).

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	Table 3. Experimental design	
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(c) Chemical reaction of the leaching agent at the surface of the unreacted core.

If one of these steps is the limiting step (i.e., with a kinetic very slower than the other two), the end point of the process can be obtained from one the following formulas [18]:

Liquid film diffusion control

$$
\frac{t}{\tau_{\rm F}} = X,\tag{4}
$$

$$
\tau_{\rm F} = \frac{\rho R_0}{3bk_1C_{\rm N}},\tag{5}
$$

where *t* is the reaction time, τ_F is the time for complete dissolution by liquid film control, ρ is the density of vanadium ore, R_0 is the initial radius of particle, *b* is stoichiometric coefficient, $k₁$ is the mass transfer coefficient between fluid and particle and C_N is the concentration of the leaching agent in the bulk of fluid.

Reaction control

$$
\frac{t}{\tau_{\rm R}} = 1 - (1 - X)^{\frac{1}{3}},\tag{6}
$$

$$
\tau_{\rm R} = \frac{\rho R_0}{b k_{\rm s} C_{\rm N}},\tag{7}
$$

where τ_R is time for complete dissolution by chemical reaction control and k_s is the rate constant.

Solid product diffusion control

$$
\frac{t}{\tau_{\rm P}} = 1 - 3(1 - X)^{\frac{2}{3}} + 2(1 - X),\tag{8}
$$

$$
\tau_{\rm P} = \frac{\rho R_0^2}{6b D_0 C_{\rm N}},\qquad(9)
$$

where $\tau_{\rm P}$ is time for complete dissolution by product diffusion control and D_0 is the effective diffusion coefficient of NaOH in the product layer.

Aarabi-Karsagani et al. [6] investigated the kinetics of vanadium leaching from LD converter slag. They reported that the rate of vanadium leaching from the slag in sulfuric acid follows a two-stage mechanism and concluded that the whole leaching period cannot be described by a single mechanism. Consequently, they suggested different controlling steps in each stage. Based on the SCM, they reported that the kinetics of leaching at low temperature is controlled by chemical reaction for both short period (less than 15 minutes) and long period (longer than 30 minutes) of leaching. They also showed that at high temperatures, the leaching kinetics is controlled by diffusion through the solid product layer for both short and long periods. Liu et al. [20] studied the kinetics of vanadium dissolution in alkaline media in the range of 30–90°C and introduced the chemical reaction as the controlling mechanism in the leaching process. Li et al. [19] presented the dissolution kinetics of vanadium from black shale in sulfuric acid between 110–150°C in which both external mass transfer and diffusion through the product layer controlled the leaching rate. Moreover, Amer [21] showed that at short times, vanadium leaching using acidic hydrometallurgical processing is controlled by chemical reaction, followed by diffusion through the product layer at longer times. In a similar manner, Liu et al. [22] studied the kinetics of vanadium dissolution by water and concluded that in the short time, kinetics of the leaching process follows the SCM and the rate is controlled by diffusion through the liquid film during the long time. In spite of the mentioned investigations, Li et al. [23] observed that when the vanadium-bearing mineral is dissolved in sulfuric acid solution, only diffusion through the product layer is the rate controlling step. Based on the above survey, it can be concluded that several models can be applied to kinetics of leaching of different samples and there is no agreement on the rate controlling step in vanadium leaching process.

When none of the above mechanisms is the rate controlling step, all three steps can be taken into account for describing the overall kinetics of leaching by the SCM [18]:

$$
t = \tau_{F} X + \tau_{P} \left[1 - 3(1 - X)^{\frac{2}{3}} + 2(1 - X) \right] + \tau_{R} \left[1 - (1 - X)^{\frac{1}{3}} \right].
$$
 (10)

In this case, the overall rate of leaching processes determined from Eq. (10) and the total time of complete leaching can be determined from summation of constants of this equation (i.e., τ_F , τ_P and τ_R). Share of each of the above mentioned steps in the kinetics of leaching can be revealed by fitting the experimental data to Eq. (10) and evaluating the constants of this equation. For this purpose, the method proposed by Nazemi et al. [17] was used in this study.

RESULTS AND DISCUSSION *Model Fitting*

The results of analysis of variance (ANOVA) which were performed by DX8 program are shown in Table 4. In order to estimate the model coefficients and the response for the quadratic polynomial, multiple regression analysis was employed and the following equation was obtained:

$$
Recovery = +61.98 + 8.88A + 10.12B + 2.96C
$$

+ 6.96D + 4.19AB + 3.94BC (11)
- 3.94BD + 5.93A² + 6.18C²,

where *A*, *B*, *C* and *D* are NaOH concentration in M, L/S in mL/g , temperature in \degree C and time in minutes, respectively. The correlation coefficient (R^2) for this model was calculated to be 0.8982 which shows that the model fits adequately to the experimental data. The perturbation diagram and the above equation reveal that the liquid to solid ratio (L/S) (*B* has the highest coefficient) is the most effective and temperature (*C* has the lowest coefficient) is the least influencing parameters, respectively (data is not shown). In Eq. (11), the negative sign for parameters of L/S and temperature (or $-BC$) means that the interaction between *B* and *C* (which is not a mathematical multiplying *B* by *C* or $(B \times C)$ but has a physical meaning) reduces the recovery. This phenomenon is independent of the effect of *B* or *C* separately.

Response Surface Plotting

Interactions between influential parameters (e.g., time, temperature, and L/S ratio) and vanadium recovery are presented using three-dimensional plots in Figs. 3 and 4, respectively. Interaction terms with *p*-values <0.05 and the largest absolute coefficients in the fitted model (*AB* (NaOH concentration \times L/S), *BC* (L/S \times temperature) and *BD* (L/S \times time),

Source	Sum of Squares	$D_{\rm f}$	Mean Square	F Value	<i>p</i> -value Prob > F
Model	8377.47	9	930.83	19.61	< 0.0001
A-NaOH	1890.37	$\mathbf{1}$	1890.37	39.83	< 0.0001
$B-L/S$	2460.37	1	2460.37	51.84	< 0.0001
C-Temp	210.04	1	210.04	4.43	0.0483
D-time	1162.04	1	1162.04	24.48	< 0.0001
AB	280.56	$\mathbf{1}$	280.56	5.91	0.02246
BC	248.06	1	248.06	5.23	0.0333
BD	248.06	1	248.06	5.23	0.0333
A^2	1000.14	1	1000.14	21.07	0.0002
C^2	1086.25	$\mathbf{1}$	1086.25	21.07	0.0001
Residual	949.19	20	47.46		
Lack of Fit	825.86	15	55.06	2.23	0.1918
Pure Error	123.33	5	24.67		
Cor Total	9326.67	29			

Table 4. Analysis of variance (ANOVA)

respectively) were selected for the axes of the response surface plots to account for curvature of the surfaces. The interaction implies that the effect produced by changing one variable depends on the level of other variables which is discussed below.

Figure 3 reveals the interactions between the leaching parameters. It can clearly be seen that increasing the L/S ratio greatly affects the efficiency of vanadium recovery at high NaOH concentrations. Since at low NaOH concentrations, the leaching agent (NaOH) is less than which is required for dissolving the vanadium and by increasing the L/S; the amount of leachate available for reacting with the slag will be increased, at lower NaOH concentrations, raising the L/S will increase the efficiency with less intensity. However, the amount of NaOH concentration is not enough, therefore, the vanadium recovery could not be impressionable by increasing the L/S effectively. At higher NaOH concentrations, the leaching agent is enough to recover vanadium and increasing the L/S has an intensive effect (Figs. 3a and 4a).

Interaction between the L/S ratio and temperature is shown in Figs. 3b and 4b. Based on these figures, at low L/S ratio, temperature rising can even inversely decrease the recovery of vanadium which can be explained by the side reactions occurring in the system and this can be a confirmation for the fact that temperature has lesser impact compared to other parameters, as discussed before (Eq. (11)).

As seen in Figs. 3c and 4c, at shorter leaching intervals, increasing the L/S ratio improved vanadium extraction, while at longer intervals, the extraction increased slightly. The reason could be the fact that at longer leaching intervals, vanadium was extracted during the leaching process and increasing the L/S ratio could not be very effective to increase the amount of vanadium recovery.

Process Optimization

A multiple response method or optimization of any combination of four goals, namely L/S, temperature, NaOH concentration and time was applied. The best result was obtained from 30 optimum points via numerical optimization which were derived from solving the model. The maximum vanadium recovery of 99% can be achieved using the following levels of parameters; L/S ratio of 20, temperature of 40°C, NaOH concentration of 3 M, and time of 101 minutes.

Comparing these optimum levels with previous studies shows that with modeling the process and considering various interactions between different parameters, optimum condition is obtainable. For example, Mahdavian et al. [2] used a salt roasting-basic leaching process which was applied to extract vanadium from steel making slag. Sodium carbonate was found to be a successful leaching agent and to recover 80% of vanadium, the optimum conditions for leaching were 80°C, S/L: 1/20, sodium carbonate to sodium hydroxide mass ratio of 40–50 : 10, and leaching time: 60 min. Furthermore, using the same chemical analysis of LD slag and alkaline roasting process as current investigation, Aarabi-Karsagani et al. [6] reported the maximum vanadium recovery of 95% at the optimum

Fig. 3. Vanadium recovery dependency on the leaching parameters and interactions between (a) L/S and NaOH concentration, (b) temperature and L/S and (c) L/S and time.

leaching condition of 70°C, S/L: 1/15, acid concentration: 3 M, and leaching time: 150.

Kinetics

The vanadium in the slag can react with NaOH and the SCM for spherical particles of unchanging size can be used to describe the kinetic aspects of this reaction during the leaching process. Recovery of vanadium against time of leaching at various temperatures is shown in Fig. 5. It can be seen in this figure that, regardless of the leaching temperature, the vanadium recovery increases as a function of time up to 40 min. After 40 min of leaching, the recovery remains almost constant (or increases slightly). Therefore, in order to investigate the kinetics of the leaching process in this study, only recovery processes performed at less than

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40 min were taken into account. Standard deviation is also shown in this figure.

Table 5 represents the SCM parameters (dissolution times) calculated by fitting the experimental data shown in Fig. 5 (up to 40 min) to Eq. (10) by the

Table 5. SCM parameters obtained by fitting experimental data to Eq. (10)

Temperature, $\rm ^{\circ}C$	τ_F , min	$\tau_{\rm p}$, min	τ_R , min	R^2
20	0	164.1	133.7	0.997
40	0	111.9	49.7	0.989
50	0	81.1	21.1	0.956
60		68.2	16.7	0.981
70		40.7	30.3	0.998

Fig. 4. Three-dimensional plots for V recovery as a function of (a) L/S and NaOH concentration (b) L/S and temperature (c) time and L/S.

method proposed by Nazemi et al. [17]. It can be seen in this table that the coefficient related to mass transfer through the liquid film was found to be zero at all temperatures while coefficients associated with diffusion through the product layer and the chemical reaction are non-zero and of the same order of magnitude. In other words, mass transfer through the liquid film surrounding particles is very fast compared to the other two mechanisms such that it does not affect the rate of leaching. On the other hand, diffusion through the product layer and chemical reaction are equally important in determining the rate of leaching and both mechanisms are significant in the process. It is worth noting that if the traditional method for detecting the rate controlling step (considering only one of these mechanisms at a time) were used, it would have not been possible to find the rate controlling mechanism since plots related to diffusion through product layer

and chemical reaction (Eqs. (6) and (8)) would show the same quality if fit to the experimental data.

CONCLUSIONS

Based on the results obtained from the current research, the following conclusions were made:

—The LD slag contains 1.97% of V_2O_5 equivalent to 1.12% of vanadium.

—From XRD results, alkaline—roasting converted calcium vanadate $(Ca_2V_2O_7)$ to sodium vanadate $(NaVO₃)$ which is more soluble in alkaline media.

—RSM was used to optimize the vanadium leaching parameters. Based on the results, optimum leaching condition (approx. 99%) was obtained with L/S ratio of 20, temperature of 40°C, NaOH concentration of 3.0 M and leaching time of 100 minutes, respectively.

Fig. 5. Effect of temperature on vanadium recovery as a function of time (particle size 0.850 mm, 3 M NaOH and S/L of 1 : 15).

—The most influencing leaching parameter was found to be the liquid to solid ratio and the less effective was the temperature.

—At all temperatures applied during the experiments and based on the equations in the SCM, the rate of vanadium leaching is controlled by a mixed controlling mechanism during short time periods which is comprised of chemical reaction and diffusion through the solid product layer.

NOMENCLATURE

- *B* Liquid to solid ratio, mL/g
- *b* stoichiometric coefficient
- *C* temperature, °C
- C_N concentration of NaOH in the bulk of fluid, M
- *D* time, min
- D_0 diffusion coefficient, m²/s
- D_f degrees of freedom
- *F* Fischer variation ratio
- *k* number of studied factors
- k_1 mass transfer coefficient between fluid and particle, m/s
- k_s reaction rate constant, s^{-1}
- *q* fraction of the number of factors
- *R*² correlation coefficient
- R_0 initial radius of particle, m

X fraction of reacted vanadium

Greek symbols

- σ parameter in central composite design
- $ρ$ density of vanadium ore, mol/m³
- τ_F time for complete dissolution by liquid film control, s
- $\tau_{\rm P}$ time for complete dissolution by product diffusion control, s
- τ_R time for complete dissolution by chemical reaction control, s

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