

Kinetic Study on Vanadium Recovery From Oil Fired Power Stations

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Abstract

In this work it has been attempted to study the kinetic of vanadium recovery from fly ash by leaching with NaOH solution from previous work of Mosa (1999).

The selected data were chosen at particle size of scale between 125 to 200 μm , temperature 343 to 373 K, reaction (leaching) time up to 8 hours and at about fixed NaOH solution molarity (3 to 4 M). The results show that vanadium recovery increased with time and temperature and mostly decreased with particle size. A second order polynomial mathematical correlation was employed to describe vanadium recovery response. The correlation coefficient of the obtained correlation was 0.9990 and variance of 0.9981 with confidence levels of 95 %. Kinetic study shows that the chemical reaction is the controlling step which has the highest resistance among other steps. A chemical reaction order was found equals to -3 with activation energy of 1080 J/ mole and frequency factor of about 2.6884.

Keywords: Vanadium, Heterogeneous reaction, Kinetics.

1.Introduction

Vanadium is widely distributed metal thorough out the earth crustal and this makes it more abundant than such important metal as copper, zinc, lead and tin. Vanadium occurs in carbon containing deposits such as crude oil, coal, oil shale and tar sands, it also could be found in soot ash that occurs as by product at the gasification of heavy petroleum fraction or crude oil residues at the production of synthetic gas. The burning of vanadium-containing fuel oil precedes a fly ash containing vanadium which can be extracted [Khorfan et al., 2002].

Vanadium can exist in different oxidation states from -2 to +5 but the forms found in natural environment are +3, +4 and +5. Oxidation state +4 is the most stable form.

During the burning of fuel oils in boilers and furnaces, the vanadium is left behind as vanadium pentoxide (V_2O_5) in the solid residues, soot, boiler

scale and fly ash. Vanadium pentoxide is used as the catalyst for a variety of gas-phase oxidation processes.

Vanadium pentoxide is the most common commercial form of vanadium that dissolves in water and acids forming vanadates with bases. Vanadium has a very low and non-accumulative toxicity; recovery plants can be operated in such manner to ensure no air pollution results [Kirk and Othmer, 1983].

In Iraq, the fuel oil used in electric power stations releases off combustion gases which contain vanadium that could constitute a good source of this metal [Jalhoom et al., 1992].

Recovery of vanadium can be achieved from scales and ashes by extraction with NaOH. Ammonium meta-vanadate was precipitated when using ammonia solution.

Two simple idealized models can be considered, the progressive-conversion model and the unreacted-core model [Levenspiel, 1999].

This study will discuss the explanation of previous experimental data of vanadium recovery by reaction of ash residue (fly ash) from South Baghdad Power Station (in Durah) by reaction with sodium hydroxide (NaOH) solution, determining of the controlling step and study the kinetic model to find reaction order and activation energy.

2.Unreacted-core Model for Spherical Particles of Unchanged Size

Heterogeneous reaction in which a gas or liquid contacts a solid, reacts with it and transforms it into product, such reaction may be represented by:



This model was first developed by Yagi and Kunii [1955, 1961], who visualized five steps occurring in succession during reaction (see Figure 1).

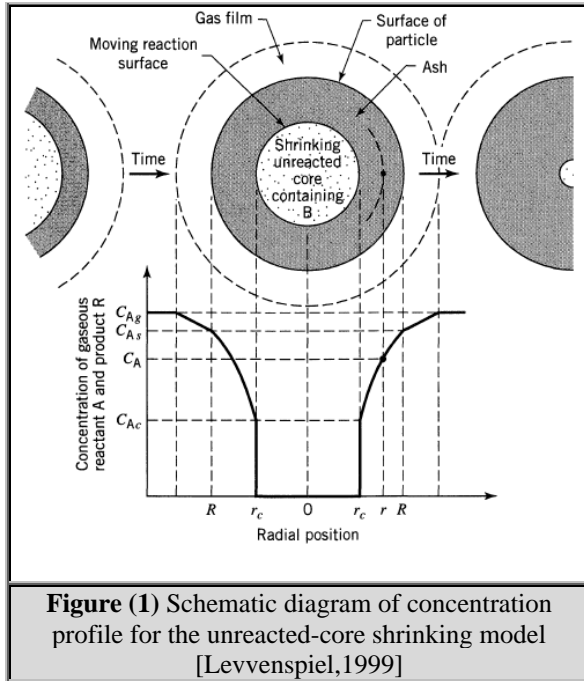
Step 1. Diffusion of gaseous reactant A through the film surrounding the particle to the surface of the solid.

Step 2. Penetration and diffusion of A through the blanket of ash to the surface of the unreacted core.
 Step 3. Reaction of gaseous A with solid at this reaction surface.

Step 4. Diffusion of gaseous products through the ash back to the exterior surface of the solid.

Step 5. Diffusion of gaseous products through the gas film back into the main body of fluid.

In heterogeneous reaction there are three resistances that may control the process namely; fluid film, ash layer and chemical reaction [Levenspiel,1999] as shown in Figure 1.



3. Diffusion through Fluid Film Controls [Levenspiel,1999]

Whenever the resistance of the gas film controls, the concentration profile for gaseous reactant A will be shown as in Figure 1. From this figure we see that no gaseous reactant is present at the particle surface; hence, the concentration driving force, $C_{Ag} - C_{As}$, becomes C_{Ag} and is constant at all times during reaction of the particle.

$$-\frac{1}{S_{ex}} \frac{dN_B}{dt} = -\frac{1}{4\pi R^2} \frac{dN_B}{dt} = -\frac{b}{4\pi R^2} \frac{dN_A}{dt} = bk_g(C_{Ao} - C_{As}) \quad 2$$

Where $bk_g(C_{Ao} - C_{As}) = bk_g C_{Ao} = \text{constan } t$

$$N_B = \rho_B V \quad 3$$

The decrease in volume or radius of unreacted core accompanying the disappearance of dN_B moles of solid reactant or dN_A moles of fluid reactant is then given by:

$$-dN_B = -bdN_A = -\rho_B dV = -\rho_B d\left(\frac{4}{3}\pi r_c^3\right) = -4\pi\rho_B r_c^2 dr_c \quad 4$$

Replacing Equation 4 in 2 gives the rate of reaction in term of shrinking radius of unreacted core, or:

$$-\frac{1}{S_{ex}} \frac{dN_B}{dt} = -\frac{\rho_B r_c^2}{R^2} \frac{dr_c}{dt} = bk_g C_{Ag} \quad 5$$

By integration:

$$t = \frac{\rho_B R}{3bk_g C_{Ao}} \left[1 - \left(\frac{r_c}{R}\right)^3 \right] \quad 6$$

From Equation 6, at complete reaction;

$$\tau = \frac{\rho_B R}{3bk_g C_{Ao}} \quad 7$$

The radius of unreacted core in terms of fractional time for complete conversion is obtained by combining Equations 6 and 7;

$$\frac{t}{\tau} = 1 - \left(\frac{r_c}{R}\right)^3 \quad 8$$

But:

$$1 - X_B = \left(\frac{\text{Volume of unreacted core}}{\text{Total volume of Particle}}\right) = \frac{\left(\frac{4}{3}\right)\pi r_c^3}{\left(\frac{4}{3}\right)\pi R^3} = \left(\frac{r_c}{R}\right)^3 \quad 9$$

Therefore;

$$\frac{t}{\tau} = 1 - \left(\frac{r_c}{R}\right)^3 = X_B \quad 10$$

Where x is a conversion

4. Diffusion through Ash Layer Controls [Levenspiel,1999]

The rate of reaction of A through ash layer at any instant is given by its rate of diffusion to the reaction surface, or:

$$-\frac{dN_B}{dt} = 4\pi r^2 Q = 4\pi R^2 Q_{As} = 4\pi r_c^2 Q_{Ao} = \text{const.} \quad 11$$

But

$$Q_A = \frac{dC_A}{dr} \quad 12$$

Combining Equation 11 and 12 gives;

$$-\frac{dN_B}{dt} = 4\pi r^2 D_e \frac{dC_A}{dr} = \text{const.} \quad 13$$

Integration Equation 13 gives;

$$-\frac{dN_B}{dt} \left(\frac{1}{r_c} - \frac{1}{R} \right) = 4\pi D_e C_A \quad 14$$

This expression represents the condition of reacting particle at any time. Eliminating NA by writing it in terms of rc as given by Equation 4 and replacing in Equation 14 gives;

$$t = \frac{\rho_B R^2}{6bD_e C_{Ao}} \left[1 - 3 \left(\frac{r_c}{R} \right)^2 + 2 \left(\frac{r_c}{R} \right)^3 \right] \quad 15$$

The time required for complete conversion of a particle (rc = 0) is;

$$\tau = \frac{\rho_B R^2}{6bD_e C_{Ao}} \quad 16$$

Dividing Equation 15 by Equation 16 gives;

$$\frac{t}{\tau} = 1 - 3 \left(\frac{r_c}{R} \right)^2 + 2 \left(\frac{r_c}{R} \right)^3 \quad 17$$

And;

$$\frac{t}{\tau} = 1 - 3(1 - X_B)^{2/3} + 2(1 - X_B) \quad 18$$

5. Chemical Reaction Controls [Levenspiel,1999]

When chemical reaction controls, the rate of reaction is given by;

$$-\frac{1}{4\pi r_c^2} \frac{dN_B}{dt} = -\frac{b}{4\pi r_c^2} \frac{dN_A}{dt} = bkC_{Ao} \quad 19$$

Writing NB in term of the shrinking radius, as in equation 4 gives;

$$-\frac{1}{4\pi r_c^2} \rho_B 4\pi r_c^2 \frac{dr_c}{dt} = -\rho_B \frac{dr_c}{dt} = bkC_{Ao} \quad 20$$

Or;

$$t = \frac{\rho_B}{bkC_{Ao}} (R - r_c) \quad 21$$

The time required for complete conversion is given when rc equal to zero;

$$\tau = \frac{\rho_B R}{bkC_{Ao}} \quad 22$$

Combining Equations 21 and 22 gives;

$$\frac{t}{\tau} = 1 - \frac{r_c}{R} = 1 - (1 - X_B)^{1/3} \quad 23$$

6. Results and Discussion

6.1 Mathematical Correlation

A second order polynomial mathematical correlation was employed in the range of the independent variables (temperature (X1), particle size (X2) and time (X3)). The general form of second order polynomial for these variables represented by the following equation:

$$Y = a_0 + a_1 X_1 + a_2 X_2 + a_3 X_3 + a_4 X_1^2 + a_5 X_2^2 + a_6 X_3^2 + a_7 X_1 X_2 + a_8 X_1 X_3 + a_9 X_2 X_3 \quad 24$$

Appendix A shows the experiments, which were extracted from Mosa [1999]. For postulating the best form of the above equation, the data of

Appendix A is first fitted with Equation 24 so that the analysis of variance of central composite design could be applied. By this analysis, each effect of the fitted polynomial equation could be tested for its significance.

The analysis of variance (F-test) is used for testing the significance of each effect in Equation 24 using the data given in Appendix A, the coefficients in Equation can be calculated simply by using least square method.

After the coefficient values of Equation 24 calculation, it is possible to compute values of vanadium conversion (Y_i) and the corresponding residual

$$e_i = Y - Y_i$$

As estimate of the experimental error variance S_r^2 is obtained by dividing the residual sum of square $\sum e_i^2$ by γ (number of degree of freedom).

$$\gamma = \text{No. of experiments} - \text{No. of coefficients in equation}$$

$$\gamma = 72 - 10 = 62. \text{ And,}$$

$$S_r^2 = \frac{\sum e_i^2}{\gamma} \quad 25$$

The estimated variance of coefficients S_a^2 is then calculated by Equation 26

$$S_a^2 = \frac{S_r^2}{\sum X^2} \quad 26$$

The significance of effects may be estimated by comparing the value of the ratio $\frac{a^2}{S_a^2}$ (F-calculated) with the critical value $F_{0.95}(1,62) = 4.00$ (the F-distribution at 95 % of confidence level with 1 and 68 degree of freedom).

The coefficient value (A) is used for estimation of F-calculated ($\frac{a^2}{S_a^2}$) and significance are shown in Table 1.

Table 1: Calculated values of Equation 4.1 coefficients

Coeff. No.	Effect of	Ai	Std. Error	F-calc.	Signif.
0	-	27.58	6.8400	16.26	S.
1	X1	-2.98	0.0990	906.07	S.
2	X2	1.262	0.0646	381.64	S.
3	X3	10.02	0.4000	627.50	S.
4	X12	0.0117	0.0006	380.25	S.
5	X22	-0.0076	0.0002	1444.00	S.
6	X32	-0.295	0.0237	154.93	S.
7	X1X2	0.0147	0.0020	5402.25	S.
8	X1X3	-0.045	0.0029	240.79	S.
9	X2X3	-0.0148	0.0015	97.39	S.

Correlation Coefficient R = 0.9990, Variance = 0.9981, F(1,62) = 4.00 Confidence level = 95 %

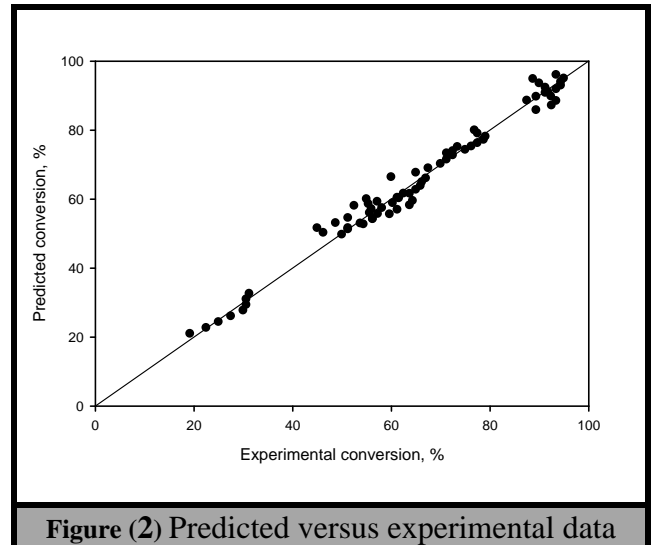


Figure (2) Predicted versus experimental data

6.2 Kinetic Model of Vanadium with NaOH

The reaction kinetics has been studied according to the differential method of analysis. This method deals directly with the differential rate equation to be tested, evaluating all terms in the equation including the derivative $d(\text{concentration})/d(\text{time})$, and testing the goodness of the fit of equation with experiments.

The rate of reaction of the present work can be expressed according to following:

$$r_A = \left(\frac{dC_A}{dt} \right) = kC_A^n \quad 27$$

Where k is the rate coefficient and n is the order of the reaction.

Differential analysis of the complete rate equation [Levenspiel, 1999] was used to calculate the change of conversion with time from a given data as shown in equations below.

$$C_A = C_{Ao}(1 - X_A) \quad 28$$

$$\frac{dC_A}{dt} = -C_{Ao} \frac{dX_A}{dt} \quad 29$$

Substitute in to Equation 27

$$r_A = \left(\frac{dC_A}{dt} \right) = -kC_{Ao}^n (1 - X_A)^n$$

$$-C_{Ao} \frac{dX_A}{dt} = -kC_{Ao}^n (1 - X_A)^n \quad 30$$

$$\frac{dX_A}{dt} = kC_{Ao}^{n-1} (1 - X_A)^n \quad 31$$

Let $k' = kC_{Ao}^{n-1}$, so that Equation 30 becomes

$$\frac{dX_A}{dt} = k'(1 - X_A)^n \quad 32$$

Where $\frac{dX_A}{dt}$ is the conversion change with time.

Plotting of conversion at given particle size versus time and this plot gives second order polynomial equation, deriving these equations gives differential equation of $\frac{dX_A}{dt}$ that can be calculate from given data.

Taking natural logarithm of Equation 32 gives:

$$\ln \frac{dX_A}{dt} = \ln k' + n \ln(1 - X_A) \quad 33$$

Table 2: Calculated values of k' and n versus various reaction conditions

Tem p. (K)	dp, μm	k'	N
333	200	0.91 0	- 3.3295
353	150	0.94 5	- 3.4266
373	125	0.96 8	- 3.9122
333	200	0.93 0	- 3.1324
353	150	0.96 6	- 3.1611
373	125	0.98 9	- 3.4403
333	200	0.94 9	- 3.1903
353	150	0.98 5	- 3.0303
373	125	1.00 9	- 3.3684

Figure 3 shows straight line of $\ln \frac{dX_A}{dt}$ versus $\ln(1 - X_A)$ with slope n and intercept $\ln k'$. The calculated values of n and k' are shown in Table 2.

The average value of the vanadium conversion reaction order (n) is minus 3.42 (about $-\frac{10}{3}$) and the values of rate coefficient appear to increase with temperature.

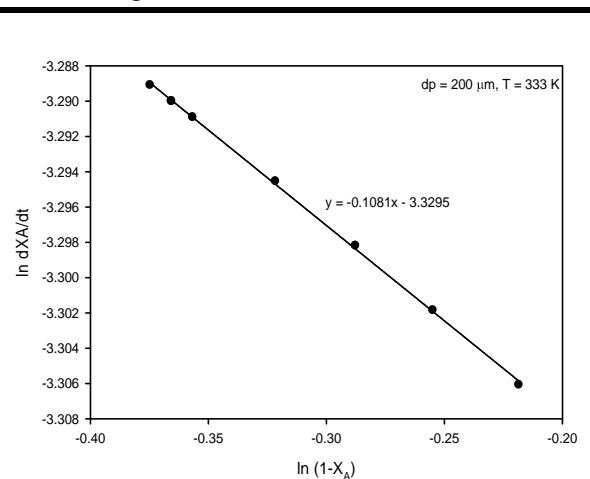


Figure (3) $\ln \frac{dX_A}{dt}$ versus $\ln(1 - X_A)$ at 333 K temperature and 200 μm particle size

The rate coefficients can be expressed by Arrhenius law, which is a function of temperature and particle size and assuming that the change of k versus dp at same temperature is done according to power law as in the below equation.

$$k = A_o \exp\left(-\frac{E}{RT}\right) \quad 34$$

Where:

Ao= pre-exponential constant (frequency factor)

E = activation energy of the reaction.

R = gas universal constant.

T = absolute temperature.

Table 3: Calculated values of, A_o and E	
$A_o, h^3(\text{wt. \%})^4$	2.6884
$E/R, -$	129.87
$E, \text{J/mole}$	1080

Non-linear estimation method has been used for solving Equation 35 to obtain the values of Ao and E. The calculated values are illustrated in Table 3.

7. Effect of Different Variables on the Conversion

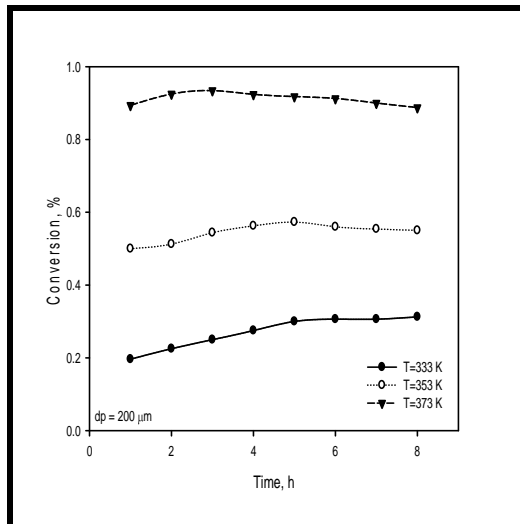


Fig. 4: Vanadium conversion versus time at different temperatures and 200µm particle size

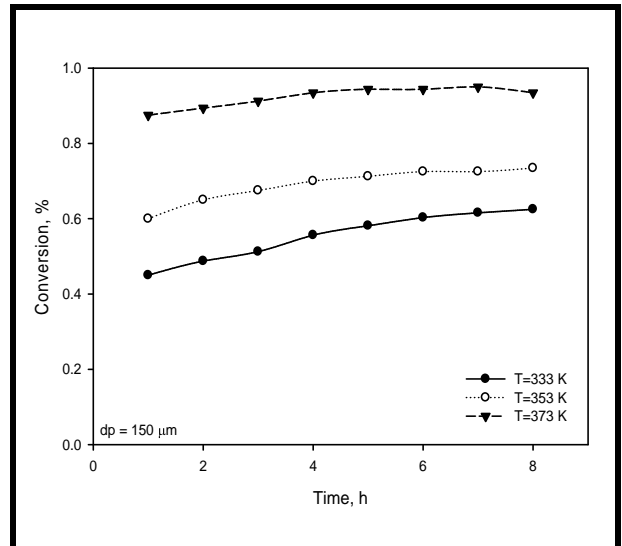


Fig.5: Vanadium conversion versus time at different temperatures and 150µm particle size

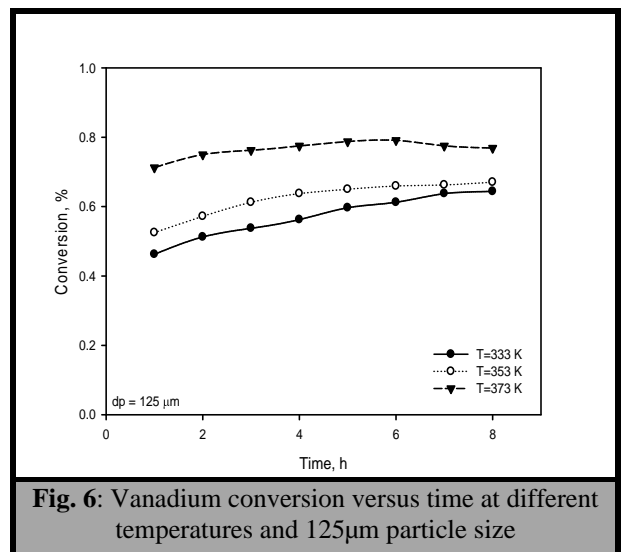


Fig. 6: Vanadium conversion versus time at different temperatures and 125µm particle size

From these figures(using equation 24) it can be seen that the vanadium conversion increase as the time increases, also it is obvious that best time which leads to maximum conversion of vanadium is greatly dependent on the specific condition of the test i.e. particle size and temperature of the test.

Examining these figures, it can be shown that increasing the temperature will increase the conversion. The temperature influences the conversion in number of ways. Firstly as the temperature increases, the mobility of the molecules will increase due to more energy gain. Secondly, the effective diffusivity increases with the increase in the temperature (this is coincided with the definition of the diffusivity). Referring to these figures, which clearly illustrate the interaction between temperature and particle size.

8. Time for Complete Conversion and Controlling Step

The time for complete conversion was calculated from given data of time and conversion according to constant spherical particle shape of shrinking-core model [Levenspiel, 1999] for constant particle size in heterogeneous reaction in which a gas or liquid contact a solid, reacts and transforms it into product.

For film diffusion controls Equation 10 is used

$$\frac{t}{\tau} = X_B \quad 10$$

For ash diffusion controls Equation 18 is used

$$\frac{t}{\tau} = 1 - 3(1 - X_B)^{2/3} + 2(1 - X_B) \quad 18$$

And for chemical reaction controls Equation 23 is used.

$$\frac{t}{\tau} = 1 - (1 - X_B)^{1/3} \quad 23$$

Where t is time of reaction (h), X_B is conversion of vanadium and τ is time for complete conversion.

Figure 7 show the calculation of τ for given temperature and particle size for the shrinking-core model for spherical particles of unchanging size where this model was first developed by Yagi [1955] and Kunii [1961].

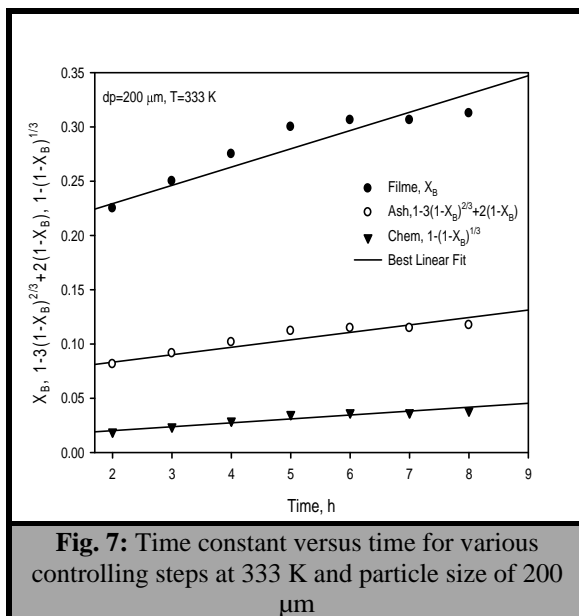


Fig. 7: Time constant versus time for various controlling steps at 333 K and particle size of 200 μm

(τ) of the different steps usually vary greatly one from the other. In such cases, it may consider that step with the highest τ to be the rate-controlling step [Levenspiel, 1999]. It is obvious from above figure that the chemical reaction is the controlling step.

The rate controlling step of a fluid-solid reaction is deduced by noting how the progressive conversion of particles is influenced by particle size and operating temperature. This information can be obtained in various ways depending on the facilities available and the materials at hand. The following observations are a guide to experimentation and to the interpretation of experimental data.

The chemical step is usually much more temperature-sensitive than physical steps; hence, experiment at different temperatures should easily distinguish between ash or film diffusion on one hand and chemical reaction on the other as the controlling step, so it's clear from fig. 4 to 6 that the chemical reaction is the controlling step.

When a hard solid ash forms during reaction, the resistance of gas-phase reactant through this chemical reaction is usually much greater than through the gas film surrounding the particle. Hence, in the presence of non-flaking ash layer, film resistance can safely be ignored. In addition, ash resistance is unaffected by changes in gas velocity.

9. Conclusions

1. It was found that the chemical reaction step has the highest resistance among other steps, which could consider as the controlling step for vanadium recovery by treatment with caustic soda.
2. A maximum vanadium conversion achieved at 5 hours, 125 μm paratical size of ash at 373 K, while 7 hours was required to get such conversion for the 125 μm particle size ash at 37

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Appendix A Experimental data and predicted vanadium conversion				
Temperature (X_1), K	Particle size (X_2), μm	Time (X_3), h	Experimental conversion (Y_{exp}), %	Predicted conversion (Y_{pre}), %
333	200	1	19.23	20.94
333	200	2	22.50	22.65
333	200	3	25.00	24.34
333	200	4	27.50	26.01
333	200	5	30.00	27.67
333	200	6	30.63	29.31
333	200	7	30.63	30.94
333	200	8	31.25	32.56
353	200	1	50.00	49.66
353	200	2	51.25	51.18
353	200	3	54.38	52.68
353	200	4	56.25	54.17
353	200	5	57.30	55.65
353	200	6	56.00	57.10
353	200	7	55.38	58.55
353	200	8	55.00	59.98
373	200	1	89.38	85.79
373	200	2	92.5	87.13
373	200	3	93.44	88.44
373	200	4	92.40	89.75
373	200	5	91.80	91.03
373	200	6	91.25	92.31
373	200	7	90.00	93.57
373	200	8	88.75	94.81
333	150	1	45.00	51.56
333	150	2	48.75	53.04
333	150	3	51.25	54.50
333	150	4	55.63	55.95
333	150	5	58.13	57.39
333	150	6	60.31	58.81
333	150	7	61.56	60.21
333	150	8	62.50	61.60
353	150	1	60.00	66.35
353	150	2	65.00	67.64
353	150	3	67.50	68.92
353	150	4	70.00	70.18
353	150	5	71.25	71.43
353	150	6	72.50	72.67
353	150	7	72.50	73.89
353	150	8	73.44	75.09
373	150	1	87.5	88.55
373	150	2	89.38	89.66
373	150	3	91.25	90.75
373	150	4	93.44	91.83

373	150	5	94.38	92.89
373	150	6	94.38	93.94
373	150	7	95.00	94.97
373	150	8	93.44	95.99
333	125	1	46.25	50.21
333	125	2	51.25	51.58
333	125	3	53.75	52.93
333	125	4	56.25	54.26
333	125	5	59.68	55.59
333	125	6	61.25	56.89
333	125	7	63.75	58.19
333	125	8	64.38	59.46
353	125	1	52.50	58.03
353	125	2	57.19	59.21
353	125	3	61.25	60.38
353	125	4	63.75	61.53
353	125	5	65.00	62.67
353	125	6	65.94	63.79
353	125	7	66.25	64.90
353	125	8	67.03	65.99
373	125	1	71.25	73.27
373	125	2	75.00	74.26
373	125	3	76.25	75.24
373	125	4	77.50	76.21
373	125	5	78.75	77.16
373	125	6	79.10	78.10
373	125	7	77.50	79.02
373	125	8	76.88	79.92

Variable Notation

Variable	Notation
A_0	Frequency factor
C_A, C_B	Concentration of reactant, mole/m ³
C_{A0}	Initial concentration of component A, mole/m ³
D_e	Effective diffusivity, m ² /s
E	Activation energy, J/mole
k	Reaction rate constant, s
k_g	Mass transfer coefficient, m/s
N_A, N_B	Number of moles of component A and B at any time
Q	Rate of mass transfer, mole/m ³ s
Q_A	Flux of A through the surface at any radius (r), mole/m ³ s
Q_{AC}	Flux of A through the reaction surface, mole/m ² s
Q_{AS}	Flux of A through the exterior surface of particle, mole/m ² s
R	Radius, μm
r_c	Radius of unreacted core, μm
ρ_B	Molar density, mole/m ³

دراسة حركية التفاعلات لاسترداد الفناديوم من مخلفات حرق الوقود في محطات الطاقة الكهربائية

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كلية الهندسة / جامعة النهريين

الخلاصة:

اهتم هذا البحث بدراسة حركية استعادة الفناديوم من مخلفات حرق الوقود في محطات الطاقة الكهربائية بعملية الاستخلاص بواسطة هيدروكسيد الصوديوم من نتائج البحث السابق لموسى (١٩٩٩). البيانات المحددة تم اختيارها عند حجم حبيبي يتراوح من ١٢٥ إلى ٢٠٠ مايكرو متر ودرجة حرارة من ٣٤٣-٣٧٣ كلفن و وقت زمن الهضم من ١-٨ ساعات وتركيز مولاري ثابت تراوح من ٣-٤ مولاري . تشير النتائج إلى أن نسبة استعادة الفناديوم تزداد بزيادة درجة الحرارة وزمن الهضم ونقصان الحجم الحبيبي . استخدمت معادلة رياضية من الدرجة الثانية لوصف استجابة استعادة الفناديوم و وجد إن معامل الارتباط كان ٠.٩٩٩٠ والتباين ٠.٩٩٨١ لمستوى ثقة ٩٥ % . دراسة الحركية أظهرت إن الخطوة المسيطرة على العملية هي التفاعل الكيمياء والتي تمتلك أكبر مقاومة من بين باقي الخطوات. كانت درجة حركية التفاعل هي -٣ و طاقة تنشيط ١٠٨٠ جول لكل مول وعامل تردد ٢.٦٨٨٤.

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