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A Product Diffusion Model for the Extraction of Cerium and Yttrium from Magnetic Coal Fly Ash Using Citric Acid Solution

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Abstract. The recovery of rare earth elements from coal-related materials, primarily fly ash, has become an emerging topic for the past few years. The availability of fly ash as solid waste from coal combustion and its low radionuclide concentrations benefit its utilization as an alternative source of rare earth elements. Using organic substances like citric acid to extract the elements further helps the environmental aspect. The maximum recovery value of cerium and yttrium was determined by reacting magnetic fly ash of 5 grams with 0.5 M of a citric acid solution with an S/L ratio of 10 for 24 hours at various temperatures. A mathematical model is also suggested to elucidate the leaching phenomenon better. The mechanistic model is developed based on the metal complex's diffusion through the ash layer. The results show that the leaching capacity of either cerium or yttrium rises along with the temperature. The maximum recovery value for leaching at 363 K is 40.21% and 54.90% for cerium and yttrium, respectively. The product diffusion model presents befitting graphs to the experimental data quite well. The effective diffusion coefficient (D_e) for both cerium and yttrium rises exponentially with extraction temperature. It is found that the value of D_e's increases from the order of 10^{-10} at 298 K to 10^{-8} cm^2/s at 363 K. The diffusion activation energy for cerium and yttrium complexes appears to be 62.5 kJ/mole and 58.4 kJ/mole, respectively.

Keywords: Citric acid; Kinetics; Magnetic fly ash; Product diffusion model; Rare earth elements

1. Introduction

Rare earth elements consist of 15 lanthanides, yttrium, and scandium (Trisnawati et al., 2020). These elements possess unique catalytic, electronic, and magnetic properties, thus widespread utilization (Ascenzi et al., 2020). Rare earth is a vital element to be used in modern industries, including automobile catalytic converters, lasers, hybrid car batteries, and energy-efficient lighting (Charalampides et al., 2015; Tuan et al., 2019). The emergence of clean technologies increases the demand for the elements. Zhou et al. (2017) predicted that the demand for lanthanum, cerium, neodymium, europium, terbium, yttrium, and dysprosium oxide would increase to 33,600 tonnes in 2025 and reach 51,900 tonnes in 2030.

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The recovery of rare earth elements from coal-related materials, primarily fly ash, has gained much attention these past few years. The use of coal-related materials as an alternative resource of rare earth elements has its advantage compared to conventional ore as it has much lower radionuclide (uranium and thorium) concentrations (Zhang et al., 2020).

Since fly ash is available as solid waste, the utilization of fly ash as a rare earth source possesses no mining cost and is economically and environmentally beneficial (Pan et al., 2020).

Acid leaching is the most common method to extract rare earth elements from fly ash. Not only inorganic acids like H2SO4 and HCl, some researchers have also recovered rare earth elements by employing organic acids like acetic and citric acid (Manurung et al., 2020; Prihutami et al., 2020; Rosita et al., 2020a; Wen et al., 2020). Many have used citric acid as its utilization gives an excellent performance in extracting rare earth elements, prevents mineral acid leakage, and averts the release of toxic gasses generated by inorganic acid (Tang et al., 2016; Gergoric et al., 2018). Some studies have also reported the kinetics of organic acid in leaching rare earth elements from fly ash (Kashiwakura et al., 2013; Kim et al., 2017; Cao et al., 2018; Handoyo et al., 2019).

The leaching of rare earth elements from fly ash using a citric acid solution is a heterogeneous solid-liquid reaction. Generally, this reaction follows the shrinking core model and undergoes five steps of mechanisms (Levenspiel, 1999), namely: (1). Diffusion of reactant from bulk solution through the liquid film onto the solid surface, (2). Internal diffusion of reactant to the surface of unreacted solid, (3). Reaction at the surface of unreacted solid, (4). Internal diffusion of the product to the solid surface, and (5). Product diffusion through the liquid film to the bulk solution. The leaching kinetics is based on the slowest step as it has the most significant resistance. Even though the shrinking core model gives a good fit for most experimental data, there are times when the model needs a modification to fit specific leaching cases (Mirwan et al., 2017; Setiawan et al., 2019). This study proposed a modified shrinking core mathematical model to better explain the kinetics phenomenon of cerium and yttrium leaching from magnetic fly ash by citric acid.

2. Methods

2.1. Materials

Less than 38 μm in diameter of magnetic fly ash was obtained from the Tanjung Awar-awar power plant, Tuban, Indonesia. X-ray diffraction analysis was performed on magnetic fly ash by using X'Pert 3 Powder from PANalytical, Netherland. Citric acid solution used as a leachant was prepared by diluting analytical grade of citric acid monohydrate (CASRN of 5949-29-1) bought from Merck in distilled water. The distilled water was obtained by processing raw water in a distillation instrument of 1032 from Köttermann, D-3162 Uetze-Hänigsen, Germany.

2.2. Leaching capacity test

Dried magnetic fly ash of 5 grams was put into Erlenmeyer and added with 50 mL of citric acid 0.5 M. The Erlenmeyer was then shaken in a water bath shaker at room temperature (298 K) for 24 hours. A sample of 4 mL was withdrawn at the end of the leaching process. The solid particles were separated from the liquid by centrifugation at 4,000 rpm for a minute. The liquid sample was then microfiltered, and a filtrate of 1 mL was diluted using aqua pro injection to 10 mL. The diluted sample was analyzed using Optima 8300 ICP-EOS from Perkin Elmer, USA, to determine the leached cerium and yttrium concentration. The leaching capacity test was also conducted at temperatures other than 298 K, namely at 318 K, 338 K, 348 K, and 363 K.

2.3. Model development

Cerium and yttrium can be leached from magnetic fly ash via the formation of soluble metal complexes with citric acid (Prihutami, 2020).

$$Ce^{3+} + jH^{+} + kC_6H_5O_7^{3-} \leftrightarrow CeH_j(C_6H_5O_7)_k^{(3+j-3k)} \tag{1}$$

$$Y^{3+} + jH^{+} + kC_6H_5O_7^{3-} \leftrightarrow YH_j(C_6H_5O_7)_k^{(3+j-3k)} \tag{2}$$

The complexes can take the forms of $MC_6H_5O_7$, $MHC_6H_5O_7^{+}$, $MH(C_6H_5O_7)_2^{2-}$, or $M(C_6H_5O_7)_2^{3}$, with M being either Ce or Y (Brown et al., 2014). The size of these metal complexes are predicted to be bigger than that of the reactants. The complexes need to diffuse through the ash layer and liquid film to reach the bulk solution. Meanwhile, film diffusion can be eliminated by adequate stirring in the system (Liu, 2013). In this sense, the internal diffusion of the complexes will be slower and thus is assumed to control the leaching rate. The illustration for this product diffusion model is presented in Figure 1.

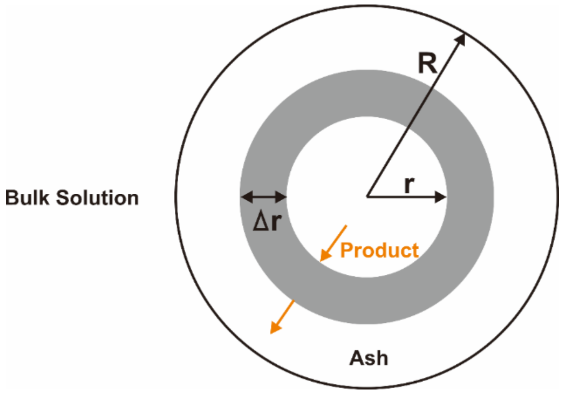

Figure 1 Leaching Model of Product Diffusion

A mathematical model for product diffusion can be suggested to describe the leaching kinetics of cerium and yttrium from magnetic fly ash by citric acid. The model is proposed based on the following assumptions.

1) Solid particles are spherical, the size of the particles is relatively uniform, and particle size remains constant throughout the leaching process
2) Film diffusion can be neglected as it is very likely to be much faster than internal diffusion, and hence the kinetics of leaching is controlled by product diffusion through the ash layer
3) Mass transfer of the product can be approached by Fick's Law (Chan et al., 2014):

$$N = -D_e\,\rho\frac{\partial X}{\partial r} \tag{3}$$

where N is mass transfer of the product (mg/min/cm^2), D_e is effective diffusivity of the product in the ash layer (cm^2/min), ρ is the density of the fly ash (g/cm^3), X is the concentration of rare earth element in fly ash (mg/g), and r is the radius of the transfer (cm)
4) The concentration of rare earth elements on the solid surface forms an equilibrium with those in the bulk liquid

The mass balance of cerium and yttrium in the form of soluble complexes at a volume element of $4\pi r^2\Delta r$ is written as Equation 4.

$$-4\pi r^2 D_e\rho\frac{\partial X}{\partial r}\bigg|_r - \left(-4\pi(r+\Delta r)^2 D_e\rho\frac{\partial X}{\partial r}\bigg|_{r+\Delta r}\right) = \rho V\frac{\partial X}{\partial t} \tag{4}$$

By taking the limit of Δr to zero, Equation 5 can be obtained.

$$\frac{\partial^2 X}{\partial r^2} + \frac{2}{r}\frac{\partial X}{\partial r} = \frac{1}{D_e}\frac{\partial X}{\partial t} \tag{5}$$

where t is the specific leaching time (min). The boundary value problems (BVPs) of Equation 5 are as follows.

$$t = 0; r = r; X = X_0; C_f = 0 \tag{6}$$

$$t = t; r = 0; \frac{\partial X}{\partial r} = 0 \tag{7}$$

$$t = t; r = R; X = K_e C_f \tag{8}$$

where K_e is the phase equilibrium constant of rare earth elements on the particle surface and in bulk solution (L/g), while C_f is the concentration of rare earth elements in bulk solution at any time (mg/L).

The value of K_e can be calculated when the equilibrium concentration of rare earth in solid (X_e) and the equilibrium concentration of rare earth in the liquid body (C_e) are known. The value of C_e is obtained from the leaching capacity test.

$$K_e = \frac{X_e}{C_e} \tag{9}$$

X_e is calculated by assuming that the amount of rare earth in liquid at equilibrium is equal to the difference between the initial amount of rare earth in solid and its amount in solid at equilibrium.

$$\rho\, N_b \frac{4}{3}\pi R^3 X_0 - \rho\, N_b \frac{4}{3}\pi R^3 X_e = V\, C_e \tag{10}$$

$$X_e = X_0 - \frac{3V}{4\,\rho\, N_b \pi R^3}\, C_e \tag{11}$$

where X_0 is the initial rare earth concentration in solid (mg/g), V is the liquid volume (mL), N_b is the number of fly ash particles being leached, and R is the fly ash radius.

Meanwhile, the concentration of rare earth in the liquid body at any time (C_f) is calculated by assuming that the initial amount of rare earth in solid equals the total of rare earth in liquid and solid at every time.

$$\rho\, N_b \frac{4}{3}\pi R^3\, X_0 = V\, C_f + \int_0^R \rho\, N_b\, 4\pi r^2 X\, dr \tag{12}$$

$$C_f = \frac{4\,\rho\, N_b \pi\left(\frac{R^3}{3} X_0 - \int_0^R r^2 X\, dr\right)}{V} \tag{13}$$

where X is the rare earth concentration in solid at every time (mg/g).

2.4. Model solution and simulation

The numerical calculation for this model was performed using MATLAB®. The partial differential model (Equation 5) was converted into a set of ordinary differential equations by applying finite-difference approximation (FDA) on its LHS terms.The equations are solved using the method of lines. The value of D_e is obtained by minimizing sum-squared error (SSE), which is defined by Equation 14.

$$SSE = \sum\left[C_{f\,experiment} - C_{f\,calculated}\right]^2 \tag{14}$$

3. Results and Discussion

3.1. Magnetic Fly Ash Composition and Morphology

Magnetic fly ash from the Tanjung Awar-Awar power plant mainly consists of SiO_2, Fe_2O_3, and Al_2O_3 with fewer CaO, MgO, and others (Handoyo, 2019). The concentration of Fe_2O_3 in the magnetic fraction is much greater than in non-magnetic and raw fly ash, which only contain 5.58% and 9.04%, respectively (Manurung et al., 2020; Rosita et al., 2020b). It also has less SiO_2 and Al_2O_3 concentration (Prihutami, 2020). These elements include hematite, quartz, magnetite, mullite, and amorphous aluminosilicates.

Fly ash used in this research is the by-product of Kalimantan coal combustion with a burning temperature of 1,673 K (Rosita et al., 2020b). Since quartz has a higher melting point, some quartz from coal remains unaltered in fly ash (Wang, 2014). However, the combustion temperature is sufficient to transform aluminosilicate materials into mullite and even promote its growth (Sultana et al., 2011). Meanwhile, magnetite and hematite in magnetic fly ash comes from the conversion of other iron minerals, mainly pyrite, in coal (Kukier et al., 2003).

Table 1 Oxide Composition (%) in Tanjung Awar-awar Magnetic Coal Fly Ash (Handoyo, 2019)

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	Cr_2O_3	TiO_2	MnO	P_2O_5	SrO	BaO	LOI
31.5	21.6	31.4	6.34	4.39	0.53	0.59	0.032	0.83	0.17	0.26	0.07	0.1	2.01

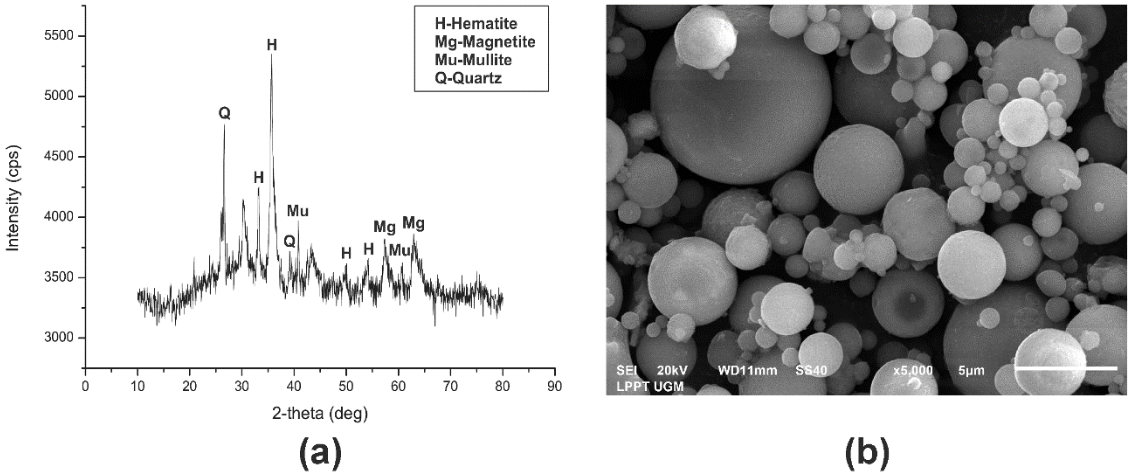

Figure 2 a) X-ray Diffraction Pattern of Magnetic Coal Fly Ash and b) Magnetic Coal Fly Ash with 5,000-Times Magnification

While non-magnetic fly ash usually has irregular shapes, magnetic fly ash is generally spheroidal (Vu et al., 2019). Scanning electron microscope analysis shows that magnetic fly ash from the Tanjung Awar-awar power plant corresponds to this finding since its shape tends to be spherical. These magnetic particles are formed by transforming and melting mineral matters in coal which condense into spheroidal particles and are followed by the deposition of Fe or Fe oxide from iron carbonate on the particle surface (Vu et al., 2019).

3.2. Leaching capacity

The leaching of rare earth elements from magnetic fly ash by citric acid reaches equilibrium after some time (Prihutami, 2020). The leaching capacity test is required to validate maximum rare earth recovery after infinity time at each operating condition. The leaching capacity data in Table 2 show a rise along with the increasing temperature.

In general, leaching capacity data show that the maximum recovery of either cerium or yttrium from magnetic fly ash is not too high. The recovery is approximately 40.21% and 54.90% at the highest leaching temperature for cerium and yttrium, respectively. The data indicate that there are unleached elements remain in the solid particles. The presence of acid extractable rare earth is greatly determined by the fly ash origin (Wang et al., 2019). Coal combustion at a temperature of more than 1,473 K causes vitrification, where glassy ash particles entrap some rare earth elements (Peterson et al., 2017). Vitrified rare earth cannot be leached as the glass is acid-resistant. Citric acid can only leach rare earth outside the glassy structure, thus lowering its leaching capacity.

Table 2 Leaching Capacity of Cerium and Yttrium at Various Temperatures

Temperature (K)	Leaching Capacity (%)	
	Cerium	Yttrium
298	36.83	47.52
318	38.60	50.06
338	39.05	51.74
348	39.75	53.00
363	40.21	54.90

3.3. Kinetics model

The kinetics model is essential for the scale-up process and predicting recovery at any arbitrary condition. Besides, it can also be used to explain the leaching phenomenon. In this model, rare earth complexes undergo a diffusion through the ash layer before reaching the bulk liquid, controlling the leaching rate. At the solid surface, rare earth elements form a phase equilibrium where the value of its constant can be calculated using Equations 11 and 9.

Table 3 Phase Equilibrium Constant of Cerium and Yttrium at Various Temperature

Temperature (K)	K_e (L/g)	
	Cerium	Yttrium
298	0.0172	0.0110
318	0.0159	0.0100
338	0.0156	0.0093
348	0.0151	0.0088
363	0.0148	0.0082

The equilibrium constant is the ratio of rare earth concentration in fly ash to its concentration in the liquid. Table 3 presents the equilibrium constants of cerium and yttrium, which decrease as temperature increases. It shows that the equilibrium shifts to the bulk liquid at higher temperatures and causes rare earth concentrations in liquid to rise.

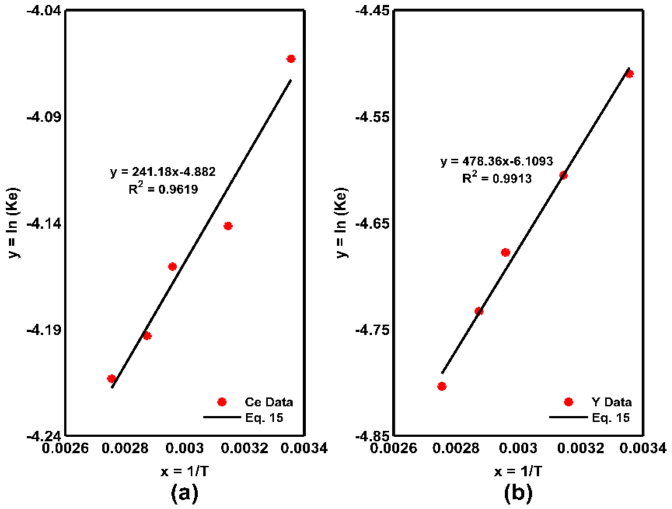

Figure 3 Relation between ln (K_e) of a) Cerium and b) Yttrium to 1/T

Smith et al. (2001) described the value of equilibrium constant as a function of temperature in Equation 15.

$$ln\frac{K_e}{K_e^{'}} = -\frac{\Delta H^o}{R}\left(\frac{1}{T}-\frac{1}{T^{'}}\right) \tag{15}$$

where K_e' is the equilibrium constant at standard condition, ΔH° is the enthalpy of leaching at standard condition, R is the ideal gas constant, T is equilibrium temperature, and T' is standard temperature. Figure 3 shows the plot between ln K_e and 1/T for cerium and yttrium. The linear regression for both data gives satisfying R^2 values, which are 0.9619 and 0.9913. The R^2 values are very close to one, meaning that the relation between K_e and T follows Equation 15 very well. According to Equation 15, the value of ΔH° is found to be -2.005 kJ/mole for cerium and -3.977 kJ/mole for yttrium.

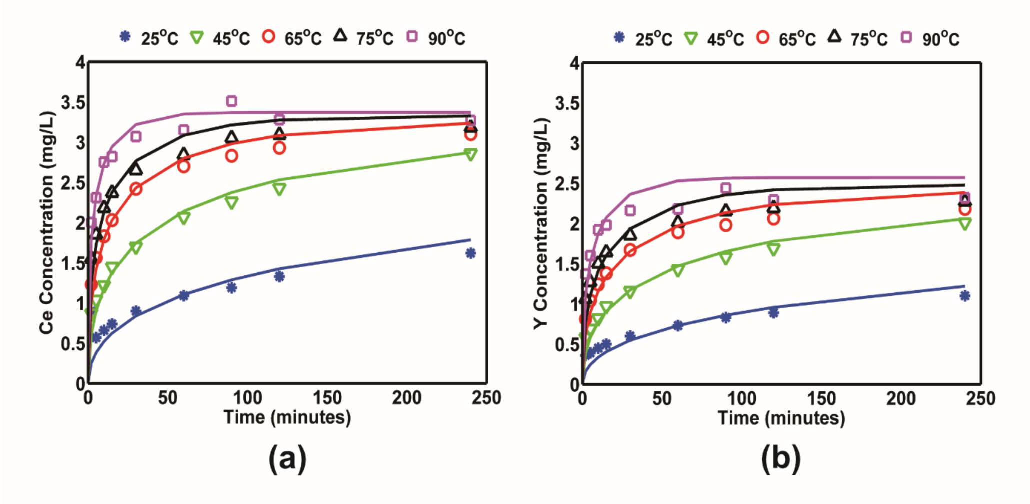

Figure 4 Product Diffusion Model for the Leaching of a) Cerium and b) Yttrium

Table 4 Parameters for Product Diffusion Model at Various Temperatures

Temperature (K)	Cerium		Yttrium	
	D_e (cm^2/min)	SSE	D_e (cm^2/min)	SSE
298	1.5250×10^{-10}	0.5392	1.9250×10^{-10}	0.1064
318	1.0350×10^{-9}	0.1265	1.2550×10^{-9}	0.0743
338	3.3750×10^{-9}	0.1497	3.5750×10^{-9}	0.1721
348	5.8550×10^{-9}	0.2650	6.0950×10^{-9}	0.3108
363	1.5775×10^{-8}	0.1649	1.4815×10^{-8}	0.4053

The plot between rare-earth concentration data from the experiment and the product diffusion model is shown in Figure 4a and Figure 4b. Parameters of the model, namely effective diffusivity of the products (D_e) and SSE, are presented in Table 4. Both graphs show the product diffusion model lines that coincided with the data. The SSE values close to zero, indicating that the product diffusion model gives a good result.

The value of D_e for both cerium and yttrium increases with the elevation of leaching temperature as the kinetic energy also increases. The plot between ln D_e and 1/T, shown in Figure 5, gives an R^2 value of 0.9947 for cerium and 0.9920 for yttrium. These values suggest that the diffusion of rare earth complexes is affected by temperature according to the Arrhenius equation (Atmani et al., 2016).

$$D_e = D_0\, e^{-\frac{E_d}{RT}} \tag{16}$$

where D_0 is a pre-exponential factor, and E_d is the diffusion activation energy. The diffusion activation energy for cerium complexes is 62.5 kJ/mole, while for yttrium is 58.4 kJ/mole. Diffusion activation energy is the minimum energy needed by the complexes to start moving. The calculation shows that yttrium complexes diffuse more quickly as it requires less energy than cerium complexes.

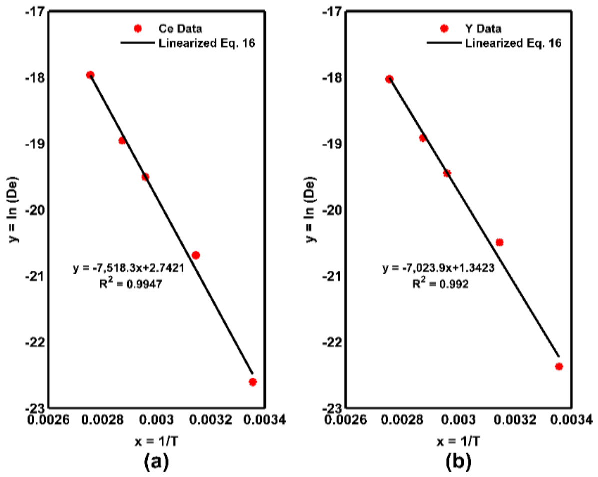

Figure 5 Relation between ln (D_e) of a) Cerium and b) Yttrium to 1/T

The product diffusion model is proposed to provide a more flexible kinetics model. This model is readily developed for cases involving different-sized particles, non-isothermal processes, or others by adding equations relevant to their process conditions. The derivation of Ke and De into temperature functions also creates the a more generic model.

4. Conclusions

The rate of diffusion can be enhanced by increasing the kinetic energy of molecules via heating. As cerium and yttrium leaching is controlled mainly by diffusion, their leaching capacity depends on temperature. Besides, the leaching capacity is also greatly influenced by the fly ash origin. The value of leaching capacity is an important variable in calculating the kinetics model. The proposed product diffusion model fits very well for cerium and yttrium leaching. The diffusion activation energy calculated from the model shows that cerium complexes have a higher value than yttrium. The result explains the lower recovery value of cerium as it needs more energy to leach.

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