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A MODIFIED SHRINKING CORE MODEL FOR LEACHING OF ALUMINUM FROM SLUDGE SOLID WASTE OF DRINKING WATER TREATMENT

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ABSTRACT

The kinetics of aluminum leaching from sludge solid waste (SSW) using hydrochloric acid at different leaching temperatures (30-90 °C) was studied. A mathematical model was developed based on a shrinking core model by assuming first-order kinetics mechanisms for leaching and equilibrium linear at the solid-liquid interface. The proposed model is suited to fit experimental data with three fitting parameters and to simulate the leaching of aluminum from SSW, which was validated with mass transfer coefficient (k_c, cm/s), diffusion coefficient (D_e, cm^2/s), and reaction rate constants (k, cm/s). The evaluated k_c, D_e, and k are expected to follow an increasing trend with increasing temperature. The correlation coefficient $\geq$ 0.9795, the root mean square error $\leq$ 0.399, the mean relative deviation modulus $\leq$ 6.415%, and the value of activation energy is 13.27 kJmol^{-1}. The proposed model could describe the kinetics of aluminum leaching from the SSW DWT in accordance with test parameters and the relevant statistical criteria. Valuable information on the results of this work can be given for the purposes of the simulation, optimization, scaling-up and design of leaching process.

Keywords: Aluminum; Leaching; Recovery; Shrinking core model; Sludge solid waste

1. INTRODUCTION

Sludge solid waste of drinking water treatment (SSW DWT) is waste by-products of the drinking water treatment processes produced daily and largely produced during purification (e.g., coagulation-flocculation and solid-liquid separation with sedimentation process) while removing colloidal impurities from the raw water (Nair & Ahammed, 2015; Zhou et al., 2015). Its process is a fundamental process used a large amount of generally aluminum salt coagulants such as commercial aluminum sulfate and poly aluminum chloride (Nair & Ahammed, 2015). A large amount of coagulant used in DWT is proportional to the amount produced SSW. Global production of SSW is produced approximately 10,000 tons per day, in which the European countries such as Ireland, Germany, the Netherlands, the UK, and Portugal take up 10.38%, United States 72.6% and China Taiwan 0.003% (Babatunde & Zhao, 2007). In Indonesia, especially Banjarmasin, South Kalimantan, SSW is produced more than 6.91×10^{6} tons (70 wt.%) every year. At present the majority of SSW is still disposed to landfill, to sanitary sewers, and by land application, assuming that these SSW do not poisonous (Babatunde & Zhao, 2007). Given the high demand for the quantity and quality of drinking water, coupled with increasing generation of SSW and environmental restrictions on their disposal until recently, have prompted various studies to look for alternative options coagulant and reuse them (Zhou et al., 2015). The reduction and re-use of SSW have been considered, and providing varying achievement level.

The main constituents of SSW-DWT are SiO_2 and Al_2O_3, however, during the addition of aluminum salt in the water treatment process, the aluminum hydroxides [$Al(OH_3)$] precipitates have been formed (Cheng et al., 2012; Nair & Ahammed, 2015). Aluminum extraction from SSW is performed mainly by hydrometallurgical methods in which the SSW is first leached by a hydrochloric acid solvent. During the leaching process, the soluble aluminum hydroxides can be formed and stayed in solution. In the separation process, precipitates of aluminum hydroxides were transferred to the leaching filter cake.

Based on differential mass balance, mass transfer, and equilibrium relationships, the mathematical model will usually be proposed. Shrinking core (SC) model describes that irreversible desorption was followed by diffusion process through the pores inside the porous solid (Goto et al., 1996) which reacts with a solvent to leaving a reacted layer around the unreacted core. This model assumes that the solute is extracted located around the shrinking core. The SC model has been widely used in the hydrometallurgy for leaching process to obtain metal or any other material valuable (Safari et al., 2009; Baba & Adekola, 2012; Cheng et al., 2012) and successfully applied to leach of zinc from a zinc ore containing silica (Safari et al., 2007), to study of dissolution kinetics of a Nigerian galena ore (Baba & Adekola, 2012), to study the aluminum salt leaching from sludge (Cheng et al., 2012). Some studies described that the leaching process is dominated by three of the rate controlling step mechanisms i.e. diffusion through liquid film surrounding particle, diffusion through product layer, and surface chemical reaction at the unreacted core (Levenspiel, 1998; Baba & Adekola, 2012; Cheng et al., 2012). Most of them concentrate on the determination of rate controlling steps using three reaction mechanisms with whole leaching time process for removing and recovering some metals from different materials. In the development of the leaching process, the leaching kinetics of aluminum from SSW is necessary for process optimization and reactor design. In spite of many studies using different methods to remove and recover metals from sludge, however, an appropriate model for aluminum leaching kinetics is not available.

The aim of this study was to determine the influence of different temperature from 30 to 90 °C on the aluminum leaching rate from the SSW and to develop a mathematical model based on SC model used in supercritical fluid extraction and the enforceability of the model for the aluminum leaching kinetics with temperature dependent in which the mass transfer coefficient, reaction rate constant, and diffusion coefficients were used as fitting parameter.

2. METHODOLOGY/ EXPERIMENTAL

2.1. Material

SSW samples were collected from the sludge ponds of DWT Banjarmasin, Indonesia, and washed and dried under direct sunlight for 24 hours, and then oven-dried at 105°C for 3 hours. They were milled in a grinder, sieved to select particles 0.074-0.044 mm in with particle size of 0.0585 mm before leaching process was carried out.

2.2. Leaching procedure

About 5 g (measured exactly) of grinded and classified SSW was extracted with 250 ml of 4 M hydrochloric acid (HCl) (prepared from Sigma-Aldrich with a purity of ≥ 32%) in an Iwaki Pyrex boiling flask. The flask had one neck for a thermometer and for the inlet or withdrawal of samples at regular time intervals (3-60 min). The reaction mixture was stirred at 300 r.min-1 using a magnetic stirrer and heated (Dragon Lab, MS-H-Pro, People's Republic of China) indirectly through a water bath at different temperatures (30, 50, 70 and 90 ºC). At selected time interval (5, 10, 20, 30, 40, 50 and 60 minute), all samples were collected using a syringe and filtered for analysis determine aluminum content in solution using inductively coupled plasma cluster optical emission spectrometer (ICP-OES) (9060-D Teledyne Leeman Labs. the USA). Each analysis was repeated three times and deputized with average values.

The total of aluminum can be leached out in the acid condition and calculated based on the standard methods (Cheng et al., 2012). SSW was added in nitric acid with a ratio of 1:1 and filtered. The filtrate will be analyzed using ICP-OES to know the amount of soluble aluminum. The result indicates that approximately 61.8 mg of aluminum ions can leach from 1 g of WTS, and the aluminum leaching recovery (x) can be stated as $x = (X/X_0)\times 100$, where X_0 denotes total aluminum obtained through acid leaching process and X is the amount of aluminum obtained at different conditions (mg/g).

2.3. Model development

According to Cheng et al., (2012), a lower pH between 1 and 3 in the aluminum dissolution would be obtained the higher aluminum recovery ratio approximately 70-90%. Furthermore, at normal temperature Al_2O_3 can not react with acid so that the solid phase amorphous aluminum hydroxide contained in sludge can be leached out by the acidification process. Chemical reactions that occur in the SSW and hydrochloric acid, are stated as follows:

$$Al(OH)_3\ (s) + 3HCl\ (aq) \rightarrow AlCl_3\ (aq) + 3H_2O \qquad (1)$$

The aluminum ions were formed by appending acid ions to dissolve aluminum hydroxide from SSW, and by a dispersion mechanism, the aluminum ions can be dissolved and leached out from SSW (Cheng et al., 2012). The hydrochloric acid ions first were diffused through the film surrounding the SSW particles to the surface of the solid. Furthermore, the acid ions will be continued to penetrate and to diffuse through the blanket of a product layer to the surface of the unreacted core and reacted with the aluminum precipitates. And ultimately aluminum ions would be diffused out of the SSW particles to the surrounding fluid. The common SC model in leaching process is illustrated in Fig. 1.

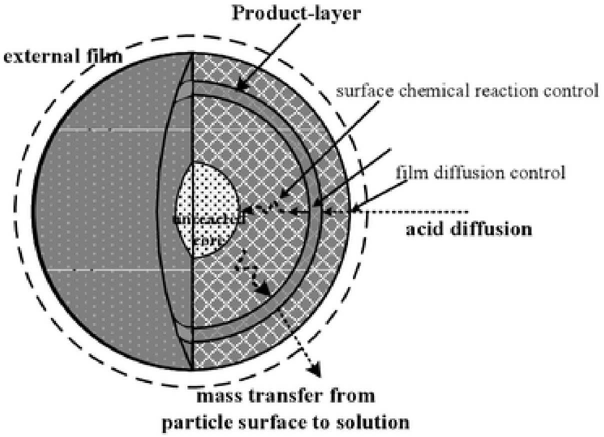

Figure 1. Schematic drawing of the SC model

2.4. Mathematical modeling of leaching kinetics

SC model assumptions as follows: SSW spherical particles; completely mixed particles so that its spherical shape can be maintained; fixed temperature during the leaching process; other substances in the SSW are not a significant effect on the kinetics (Safari et al., 2009).

Total mass balance equation for leaching with the following

$$\frac{dC_{AL}}{dt} = \frac{k_c}{R}\frac{(1-\varepsilon_L)}{\varepsilon_L}(C_{A(R)} - C_{AL}) \qquad (2)$$

Variations in time from the solid phase (aluminum concentration in a particle of SSW) are related to the mass transfer rate of solute as follows

$$\frac{d\overline{q}}{dt} = \frac{4\pi R^2 k_c \rho_s}{b \times solids\ mass}(C_{A(R)} - C_{AL}) \qquad (3)$$

The diffusion in outer particle is given by

$$-\frac{d}{dr}\left(-D_e 4\pi r^2 \frac{dC_{A(R)}}{dr}\right) = 0 \qquad (4)$$

The average value of the solute concentration as follows

$$\frac{\bar{q}}{q_0} = \left(\frac{r_c}{R}\right)^3 \tag{5}$$

Boundary conditions and initial conditions are given as follows:

$$r = r_c \rightarrow D_e \frac{dC_{A(R)}}{dr} = kC_{A(R)} \tag{6}$$

$$R = r \rightarrow -D_e \frac{dC_{A(R)}}{dr} = k_c(C_{A(R)} - C_{AL}) \tag{7}$$

$$r_c = R \text{ and } \bar{q} = q_0 \text{ at } t = 0 \tag{8}$$

The model parameters, k_c, k, and D_e can be obtained from fitting the experimental data to Equation 6 to 8. By integration of Equation 4 obtains solution

$$C_{A(R)} = -\frac{k_1}{r} + k_2 \tag{9}$$

By substitution of Equation 6 to Equation 9 is obtained

$$k_1 = \frac{kk_2 r_c^2}{(D_e + kr_c)} \tag{10}$$

The mass of solute of the aluminum leaching recovery is calculated combining Equation 7, 9, and Equation 10. Furthermore, solving Equation 9 for finding k_1 and k_2 would result in obtaining $C_{A(R)}$. Before that, the Equation 11 substituted into Equation 10.

$$k_2 = \frac{k_c C_{AL}(D_e + kr_c)R^2}{D_e(kr_c^2 + R^2 k_c) + k_c k(r_c R^2 - r_c^2 R)} \tag{11}$$

$$C_{A(R)} = -\frac{kk_c C_{AL} R^2 r_c^2}{D_e(kr_c^2 + R^2 k_c) + k_c k(r_c R^2 - r_c^2 R)r} + \frac{k_c C_{AL}(D_e + kr_c)R^2}{D_e(kr_c^2 + R^2 k_c) + k_c k(r_c R^2 - r_c^2 R)} \tag{12}$$

Statistical analysis

According to Bucić-Kojić et al., (2013) the proposed model parameters determined by three statistical criteria, namely c_{coef}, *RMSE*, and *E* were calculated by the following:

$$c_{coef} = \sqrt{1 - \frac{\sum_{i=1}^{N}\left(C_{pred,i} - C_{\exp,i}\right)^2}{\sum_{i=1}^{N}\left(C_{\exp,i} - C_{\exp,sr}\right)^2}} \tag{13}$$

$$RMSE = \sqrt{\frac{1}{N} \cdot \sum_{i=1}^{N}\left(C_{pred,i} - C_{\exp,i}\right)^2} \tag{14}$$

$$E = \frac{100}{N}\sum_{i=1}^{N}\left|\frac{C_{\exp,i} - C_{pred,i}}{C_{\exp,i}}\right| [\%] \tag{15}$$

The suitability of data and models was good when *RMSE* → 0, c_{coef} → 1, and $E < 10\%$ (Bucić-Kojić et al., 2013).

3. RESULTS AND DISCUSSION

Model parameters adjusted to aluminum content in untreated leaching (q_0 = 61.8 mg of aluminum ions for all investigated temperatures), the particle radius *R* was fixed at 0.0295 cm (262.5 mesh), the solid to solvent ratio was 0.02 g/mL and the temperature-dependent the mass transfer coefficient, reaction rate constant, and diffusion coefficients, which values are listed in Table 1 together with the results of statistical analysis. Mass transfer coefficient, the reaction rate constant, and the diffusion coefficient were evaluated by minimizing the *RMSE* criteria which expected to follow an increasing trend as the temperature increases (Bucić-Kojić et al., 2013) causing a mass transfer from the solid to the solvent occurs higher. According to Geankoplis (1993), diffusion coefficients of solid are 10^{-30}-10^{-4} m^2/s.

Figure 2 shows that the aluminum leaching recovery increased with increasing temperature as the function of time. Equation (12) was adapted to experiment data by minimizing the difference of *RMSE*. The shape of the curve obtained from the calculation model has followed the curve of leaching experiment data. Based on Figure 2 and Table 1, this model is appropriate to estimate the leaching kinetics of aluminum from SSW which confirmed by $c_{coef} \geq 0.995$, $RMSE \leq 0.399$ mg/g, and *E* value lower than 6.415% for all temperatures.

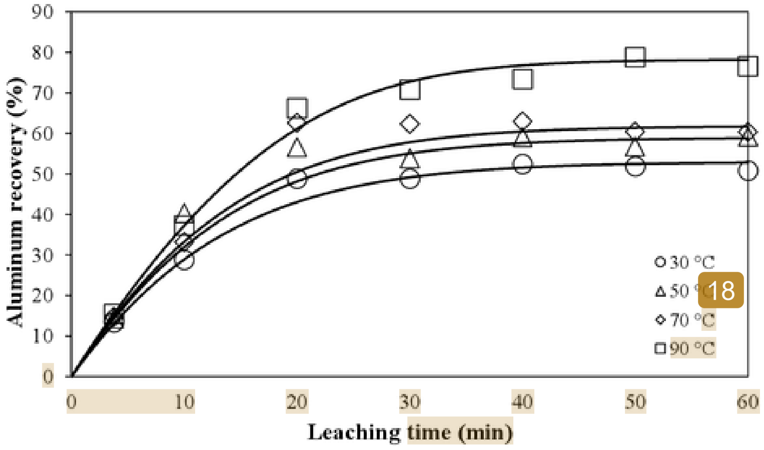

Figure 2. Aluminum leaching recovery from experimental data and model curves according to Equation (12)

Table 1 Fitting model parameters (k, k_c and D_e) and statical criteria (c_{coef}, $RMSE$, E) at different leaching temperatures (T)

***T* (°C)**	**30**	**50**	**70**	**90**
***k* (cm/s)**	10.50	16.40	20.60	25.40
k_c (cm/s)	0.0041	0.0045	0.0047	0.0049
D_e.10^{-5} (cm^2/s)	6.80	8.50	9.35	9.85
c_{coef}	0.999	0.995	0.997	0.999
RMSE	0.399	0.394	0.368	0.384
***E* (%)**	2.793	6.415	4.975	2.865

The relationship between the mass transfer coefficient, the reaction rate constants, the diffusion coefficient and leaching temperature was characterized by the activation energy from Arrhenius equation (Bucić-Kojić et al., 2013).

$$k = Ae^{-E_a/RT} \tag{16}$$

In the form of a logarithm, it becomes Equation 17

$$\ln k = \ln A - \frac{E_a}{RT} \tag{17}$$

where A is the pre-exponential factor, E_a is the activation energy (J/mol), R is the global gas constant (8.314 J/K mol), and T is the absolute temperature (K). The activation energy was calculated from the Figure 3. k value is inversely proportional to the temperature (Levenspiel, 1998). Figure 3 shows that graph slope and intercept were obtained 1.5967 and 7.6666, respectively, which using the data presented in Table 1. Activation energy can be obtained by multiplying slope value to a global gas constant and the result is 13.27 kJ/mol.

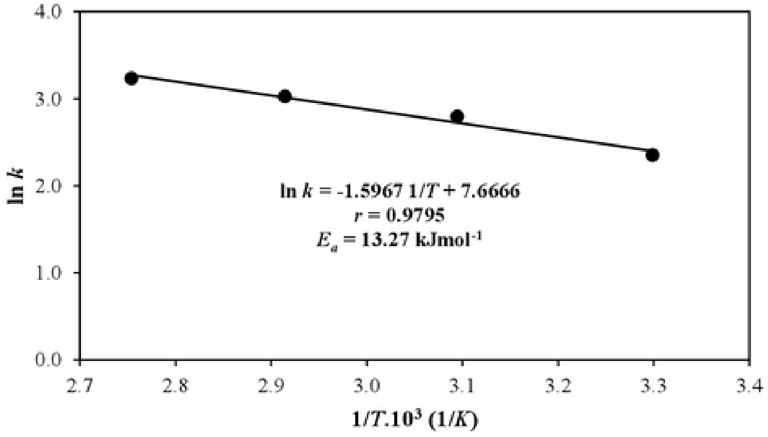

Figure 3. Arrhenius plot: ln *k* is the reaction rate constant, cm/s, and *T* is absolute leaching temperature, K.

4. CONCLUSION

The temperature had an influence on the leaching of aluminum recovery. The maximum recovery of aluminum after 50 min of agitated leaching at 90 ºC was 78.8%. The reaction rate constants and the mass transfer coefficient increased with increasing temperature from 10.50 to 25.40 cm/s and from 0.0041 to 0.0049 cm/s, respectively. The effective diffusivity ranged between $6.80 \cdot 10^{-5}$ and $9.85 \cdot 10^{-5}$ m^2/s and also indicated a good trend with temperature.

The proposed model could describe the kinetics of aluminum leaching from the SSW DWT in accordance with test parameters. It is evidenced by the relevant statistical criteria. Consequently, Valuable information on the results of this work can be given for the purposes of development of leaching process (simulation, optimization, scaling-up and design).

5. ACKNOWLEDGEMENT

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6. NOMENCLATURE

b	stoichiometric coefficient (–)
c_{coef}	correlation coefficient (–)
$C_{exp,i}$	experimental value of aluminum recovery, particle core (%)
$C_{exp,sr}$	experimental value of aluminum recovery, solid phase (%)
$C_{pred,i}$	predicted aluminum recovery calculated with the model, particle core (%)
C_{AL}	concentration in the solvent (kmol/m^3)

$C_{A(R)}$	concentration of aluminum leaching (kmol/m^3)
D_e	effective diffusivity (m^2/s)
E	mean relative deviation modulus (–)
k	reaction rate constant (cm/s)
k_c	mass transfer coefficient (cm/s)
k_1	first partition constant (kg/m^3)
k_2	second partition constant (kg/m^3)
N	number of experimental points (–)
$\bar{q}$	average value of q, mol/m^3
q_o	initial solid-phase concentration, mol/m^3
r	radial coordinate
r_c	critical radius of core (m)
R	particle radius (m)
$RMSE$	root mean square error (–)
t	leaching time (s)
T	leaching temperature (°C)

Greek letters

ε_L	void fraction
ρ_s	sludge solid waste density (kg/m^3)
π	the ratio of a circle's (3.14159)

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