\[ \Delta c_p \] = amount of copper deposited by Faradaic current
\[ d \] = diameter of pellet [m]
\[ E \] = cell voltage [V]
\[ e \] = voltage difference per pellet [V]
\[ F \] = Faraday constant \([\text{C} \cdot \text{mol}^{-1}]\)
\[ f \] = ratio of copper-deposited area to surface area of each pellet
\[ i_p \] = Faradaic current flowing through a single pellet [A]
\[ j_b \] = bypass current density based on \(A_f\) \([\text{A} \cdot \text{m}^{-2}]\)
\[ j_p \] = Faradaic current density based on \(A_f\) \([\text{A} \cdot \text{m}^{-2}]\)
\[ j_t \] = total current density \([\text{A} \cdot \text{m}^{-2}]\)
\[ k \] = mass transfer coefficient of copper ions \([\text{m} \cdot \text{s}^{-1}]\)
\[ L \] = distance between two plate electrodes [m]
\[ n \] = number of electric charges
\[ n_p \] = number of cubic cells lined up per unit length [m\(^{-1}\)]
\[ Re_d \] = Reynolds number
\[ Sc \] = Schmidt number
\[ Sh_d \] = Sherwood number
\[ t \] = electrolysis period [s]
\[ U_g \] = superficial gas velocity \([\text{m} \cdot \text{s}^{-1}]\)
\[ \alpha \] = cell constant
\[ \varepsilon_s \] = solid holdup
\[ \kappa \] = electrical conductivity of electrolyte \([\text{S} \cdot \text{m}^{-1}]\)
\[ \eta \] = actual current efficiency [-]

\(<\text{Subscript}>m</\text{Subscript}>\) = mass transfer-controlling

Literature Cited

MODEL CALCULATION OF CHEMICAL REGENERATION OF SPENT CLINOPTILOLITE FROM AMMONIUM TREATMENT

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Institute of Industrial Science, University of Tokyo, Tokyo 106

Key Words: Ion Exchange, Adsorption, Environment, Mass Transfer

A mathematical model to describe regeneration of spent clinoptilolite by concentrated sodium chloride solution as regenerant was developed and compared with experimental observations. The model is based on solid-phase diffusion in clinoptilolite particles and the equilibrium relation of sodium and ammonium ions. The model predicted the trend observed for both the elution curve of ammonium and the fractional regeneration of the bed.

The results of the model calculation suggest that the concentration of regenerant is one of the controlling factors in determining the regenerant volume and the fractional regeneration. Particle size is also defined as an important design parameter in determining the fractional regeneration and average eluted ammonium concentration or regenerant volume.

Introduction

There have been several papers\(^2\A>6\) on removal of ammonium-nitrogen from wastewater by clinoptilolite in accordance with the recent stringent water quality standards in water environment. The most of those papers have only reported about adsorption operation of ammonium-nitrogen from wastewater by clinoptilolite.

Design of an ammonium-nitrogen removal facility from wastewater by clinoptilolite involves the problem of regeneration of spent clinoptilolite.
Therefore, the cost of ammonium removal can be influenced by operating conditions of adsorption as well as those of regeneration. Also, when the recovery of ammonium in high concentration is desired, the choice of design parameters and operating conditions is very important. For this reason there is a strong need for a simple, accurate calculation method for deciding design and operating parameters from the standpoint of regeneration.

It is not easy to find an analytical or approximate procedure to estimate the optical regeneration operating parameters since the process involves many complicated rate and equilibrium relations. B. W. Mercer et al.\textsuperscript{6) first reported on regeneration feasibility of spent clinoptilolite by using chemical regenerant. Koon and Kaufman\textsuperscript{4}) tried to find the optimum operation condition on regeneration of used zeolite for cyclic use. But these studies were performed mainly through pilot plant-scale experiments, and the results cannot be interpreted to have general conclusions.

The object of this paper is to present the formulation of a mathematical model for the chemical regeneration of spent clinoptilolite from ammonium treatment by sodium chloride solution as a regenerant in the fixed bed.

By using the model presented, sensitivities of operating or design parameters are examined here. The parameters involve flow rate and concentration of a regenerant, particle size of zeolite and length of the bed. Regeneration experiments were performed in a small column to verify the usefulness of the developed model.

From the results of the model calculation, the selection of design and operation parameters is discussed in connection with regenerant volumes, fractional regeneration and the concentration of eluted ammonium.

1. Model Development

The physical meaning of regeneration of spent clinoptilolite by concentrated sodium chloride solution is explained as the conversion of zeolite from ammonium form to sodium form as shown in Eq. (1), that is, the reverse reaction of ammonium adsorption:

\[
\text{Z-NH}_4^+ + \text{Na}^+ = \text{Z-Na}^+ + \text{NH}_4^+ \quad (1)
\]

The model is constructed for the regeneration of clinoptilolite saturated with ammonium ion by concentrated NaCl solution in the fixed bed on the basis of following assumptions:

1) Total cation concentration in the fixed bed consists of ammonium and sodium ions; that is, \( C_T = C_{Na^+} + C_{NH_4^+} \).

2) Cation exchange capacity of zeolite is constant and thus total cation in the particle is preserved; that is, \( q_0 = q_{Na^+} + q_{NH_4^+} \).

3) Intraparticle diffusion is expressed by solid-phase diffusion in a spherical particle.

4) The rate of ion exchange reaction is much faster than the rate of diffusion, and hence local equilibrium is maintained between the ammonium and sodium ions in the bed and at the surface of the particle.

Differential mass balance of total ion for the fluid phase in the fixed bed is described in the one-dimensional dispersion model.

\[
u \frac{\partial C_T}{\partial z} + \varepsilon \frac{\partial C_T}{\partial t} = D_e \frac{\partial^2 C_T}{\partial z^2} \quad (2)
\]

A differential mass balance of sodium ion in the bed is given by

\[
u \frac{\partial C_{Na^+}}{\partial z} + \varepsilon \frac{\partial C_{Na^+}}{\partial t} + k_{f_e} \varepsilon (C_{Na^+} - C_{Na^+}) = D_e \frac{\partial^2 C_{Na^+}}{\partial z^2} \quad (3)
\]

Sodium ion mass balance in the particle is written as

\[
\frac{\partial q_{Na^+}}{\partial t} = D_e \left( \frac{\partial^2 q_{Na^+}}{\partial r^2} + \frac{2}{r} \frac{\partial q_{Na^+}}{\partial r} \right) \quad (4)
\]

Equilibrium relation of sodium ion and ammonium ion at the surface of the particle is given in the previous paper\textsuperscript{10) as

\[
C_{Na^+}/C_{T,0} = \frac{K_e q_{Na^+}}{q_0} \left( 1 - q_{Na^+}/q_0 \right) \quad (5)
\]

Boundary conditions and initial conditions

\[
C_{Na^+} = q_{Na^+} = 0; \quad t > 0, \quad z = 0 \\
C_{T,0} = C_{Na^+}; \quad t = 0, \quad z = 0
\]

The solid-phase diffusion coefficient of sodium ion in clinoptilolite particle, also cited from previous paper, is \( 5.6 \times 10^{-12} \text{ m}^2/\text{s} \).

Basic Eqs. (2) and (7) are converted to nondimensional form as follows:

Total ion mass balance in fixed bed

\[
\frac{\partial X_T}{\partial Z} + \varepsilon \frac{\partial X_T}{\partial T} = \frac{\varepsilon}{P_e} \frac{\partial^2 X_T}{\partial Z^2} \quad (2')
\]

Sodium ion mass balance in fixed bed

\[
\frac{\partial X}{\partial Z} + \varepsilon \frac{\partial X}{\partial T} + a_1 (X - X_J) = \frac{\varepsilon}{P_e} \frac{\partial^2 X}{\partial Z^2} \quad (3')
\]
Mass balance in the particle
\[
\frac{\partial Y}{\partial T} = \alpha_2 \left( \frac{\partial^2 Y}{\partial \rho^2} + \frac{2}{\rho} \frac{\partial Y}{\partial \rho} \right)
\]
(4')

Equilibrium relation at the surface of the particle
\[
X_s = \frac{K_s Y_s}{1 - Y_s + K_s Y_s} \bigg|_{\rho = 1}
\]
(5')

Boundary conditions and initial conditions
\[
X = Y = 0; \quad T = 0, \quad Z > 0
\]
(6')
\[
X_f = X = 1; \quad T > 0, \quad Z = 0
\]
(7')

Since ammonium ion is exchanged with an equivalent amount of sodium ion in the zeolite particle as shown in Eq. (1), ammonium ion concentration can be determined from the mass balance of total cation and sodium ion in the same time step and position.

Numerical calculation was executed as follows. First, equations of total mass balance and intraparticle diffusion were numerically solved by the Crank-Nicolson method and then the sodium ion mass balance equation in the bed was solved by the explicit method. In this work, \( \Delta Z = 1/40, \Delta T = 1/200 \) and \( \Delta \rho \) is 1/4.

The liquid-to-particle mass transfer coefficient in the fixed bed was calculated by Wilson's equation.\(^{11}\)

The dispersion coefficient in L-direction in the fixed bed was determined from a separate experiment in the same column. The pulse response was measured by introducing, in the water flowing through the same bed as was used for regeneration studies, a small pulse of 0.3% aqueous phenol solution, which is an inert solution to the clinoptilolite.

Moment analysis of the peak was adopted for deriving the axial dispersion coefficient. The second central moment was calculated by numerical integration and \( P_0 = \frac{u D_p}{E_s} = 0.40 \) and 0.20 were determined for \( D_p = 1.0 \times 10^{-3} \) m and \( 6.0 \times 10^{-4} \) m, respectively. The detailed procedure can be found elsewhere.\(^9\) These values were in reasonable agreement with the previous data for a fixed bed of small particles.\(^8\)

2. Experimental

2.1 Materials

Natural zeolite obtained from Kyuryongpo, Kyeongbuk, Korea was used. The sample was found to be mainly composed of clinoptilolite crystal. The physical properties of zeolite are given in Table 1. All cations contained in the original zeolite such as \( \text{Na}^+, \text{Ca}^{++}, \text{K}^+ \), and \( \text{Mg}^{++} \) were replaced by ammonium ion for the sake of simplicity. The cation exchange capacity of the sample was 1.90 mol/kg. The detailed preparation procedure of ammonium form zeolite can be found elsewhere.\(^{10}\)

As a chemical regenerant for spent clinoptilolite from ammonium treatment, sodium chloride, sodium hydroxide, and calcium chloride have been reported in the literature. In this work, sodium chloride solution is used because of the merits clarified by preliminary studies such as low cost, high selectivity against ammonium, stability on clinoptilolite, and higher exchange rate with ammonium ion.

2.2 Apparatus and procedure

A series of regeneration runs were carried out with a \( 1.05 \times 10^{-2} \) m (\( \phi \times 0.24 \) m) glass column. The conditions are listed in Table 2. 1.7 \( \times 10^{-2} \) kg of ammonium-form zeolite was filled in the column, which formed a bed volume of \( 2.1 \times 10^{-5} \) m\(^3\). The concentrated sodium chloride solution was introduced at SV of 5.5/h and the concentration of eluted ammonium ion in the effluent was analyzed for every effluent volume of \( 1 \times 10^{-6} \) m\(^3\) to obtain the ammonium elution curve and fractional regeneration of the bed.

Ammonium ion concentration in the effluent was analyzed by Nessler's method and total cation concentration was determined from the chloride ion concentration by an ion meter which is not influenced by cation ions composition.

3. Results and Discussion

3.1 Validity of the developed model

Typical experimental results of ammonium elution curve and fractional regeneration for Runs No. 1 and 2 as listed in Table 2 are plotted in Fig. 1. To verify the validity of the model developed here the results of model calculation in accordance with the experimental conditions are also shown in this figure by solid lines.

This figure shows the concentration of ammonium ion...
Table 3. Parameters of model calculation

<table>
<thead>
<tr>
<th></th>
<th>C</th>
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<th>(D_p)</th>
<th>(L)</th>
<th>(u)</th>
<th>(SV)</th>
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<td></td>
<td>[\times 10^3 \text{mol/m}^3]</td>
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<td>[\times 10^{-2} \text{m}]</td>
<td>[\times 10^{-2} \text{m}]</td>
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<td>3.70</td>
<td>5.5</td>
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<td>Case 3</td>
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<tr>
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<td>48.0</td>
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<td>0.06</td>
<td>12.0</td>
<td>3.70</td>
<td>11.0</td>
</tr>
</tbody>
</table>

Fig. 1. Comparison of model with experimental results for Runs No. 1 and 2.

eluted in the effluent and the fractional regeneration against the regenerant volumes. Fractional regeneration was calculated from the integration of the elution curve.

The predicted lines of ammonium elution and fractional regeneration in Fig. 1 agree with the observed values as a whole. This fact suggests that the model developed in this work is valid for the analysis of chemical regeneration of spent clinoptilolite from ammonium treatment.

3.2 Effects of parameters

Effects of the parameters in the model were examined by changing the values assigned in the standard case (case 1). Examinations were performed for the concentration and flow rate of regenerant, particle size, and bed length as listed in Table 3.

1) Regenerant concentration The effect of regenerant concentration on the peak height of ammonium concentration eluted in the effluent and fractional regeneration are shown in Fig. 2 for cases 1, 2, 3 and 4. The concentration range of regenerant adopted here, \(5 \times 10^2\) to \(5 \times 10^3 \text{mol NaCl/m}^3\), covers the sodium concentration of seawater.

As the concentration of regenerant increased, the peak concentration of ammonium became higher, from 380 to 1310 \text{mol/m}^3, resulting in less regenerant volume needed for the regeneration of spent clinoptilolite. This result is caused by the increase of the driving force of intraparticle mass transfer because the increase of total cation concentration in fluid phase decreases \(X_s\) while the cation exchange capacity of clinoptilolite is constant.

2) Flow rate of regenerant Figure 3 illustrates the results of calculation obtained for cases 1, 5 and 6 where S.V. are 2.5, 5.5 and 11.0, respectively. From this figure, it can be concluded that the higher flow rate requires a much greater volume of regenerant to reach a desired extent of regeneration than does a lower flow rate.

The exchange rate in clinoptilolite tends to be controlled by the solid-phase diffusion of cation. Therefore, it may be expected that the shape of the elution curve is not affected drastically by flow rate. As the flow rate increases, however, the time needed for regeneration becomes shorter while a greater volume of regenerant is needed, as shown in Fig. 3.

From this result, selection of the flow rate should be considered one of the important operating parameters that influences regeneration results.

3) Particle size Figure 4 shows the effect of particle size on the effluent curve of ammonium. When particle size was changed from \(3.2 \times 10^{-4} \text{m}\) to \(1.0 \times 10^{-3} \text{m}\) in diameter, the peak of the elution curve and fractional regeneration at the same regenerant volume decreases considerably.
In the case of ammonium removal from wastewater by Na-clinoptilolite, that is, in the case of favorable equilibrium relations, particle size is not an effective design parameter when it is below $1.0 \times 10^{-3} \text{ m}$ as shown in Fig. 5, but in regeneration operation it is regarded as one of the important design parameters in determining the volumes of regenerant and eluted ammonium concentration.

4) Bed length Calculation results for longer (case 9) and shorter bed length (case 10) are given in Fig. 6. It was found that relatively less regenerant volume is required to reach the desired regeneration ratio with a longer bed.

3.3 Selection of parameters

Selection of the column design and operation parameters are discussed by using the model developed in this work from two viewpoints. One is the selection of regenerant concentration in connection with the regenerant volumes and the regenerated fraction of exhausted zeolite and the other is the selection of particle size from the average ammonium concentration in the effluent, which is directly related to the recovery of desorbed ammonium in high concentration.

1) Selection of regenerant concentration Figure 7 gives the bed volumes of regenerant against moles of NaCl introduced per kg of zeolite as a parameter of fractional regeneration for each regenerant concentration.

It was found from the results of this figure that the relation between the volumes of regenerant and fractional regeneration was

$$\log B_R = AR_f + B \quad (0.4 < R_f \leq 0.95) \tag{8}$$

where $B_R$ is regenerant bed volume and $R_f$ is fractional regeneration. The constants $A$ and $B$ for each concentration are given in Table 4. There is no appreciable difference in the slope of Eq. (8) among these concentration of regenerant.

Though the efficiency of the regenerant used, which is expressed as mol of regenerant applied per kg of
zeolite, was decreased by changing regenerant concentration from $5 \times 10^2$ to $5 \times 10^3$ mol NaCl/m³, the volume of regenerant required to reach the same regeneration extent becomes smaller. From this result a suitable concentration of regenerant can be selected by the combination of fractional regeneration and regenerant volumes by this result.

2) Selection of particle size When the concentration of regenerant is decided from the above principle, the second most influential parameter in deciding required regenerant volume is expected from the above discussion to be the particle size of clinoptilolite.

The effect of particle diameter on the average ammonium concentration in the effluent for each fractional regeneration is shown in Fig. 8 with $2 \times 10^3$ mol NaCl/m³ of regenerant. The particle diameter of clinoptilolite used in the literature for ammonium removal ranged from $1.4 \times 10^{-3}$ m to $0.3 \times 10^{-4}$ m, as listed in Table 5. The particle diameter range examined here covers these practical sizes.

The average eluted ammonium concentration was decreased with increasing particle size and fractional regeneration. This fact indicates a decrease of regenerant efficiency, which is one of the influential factors in determining regeneration cost.

4. Conclusion

A mathematical model to describe regeneration of spent clinoptilolite by concentrated sodium chloride solution as regenerant was developed and tested against experimental observations.

The conclusions of this work are summarized as follows:

1) The model predicted the trend observed for both the elution curve of ammonium in the effluent and fractional regeneration of the bed.

2) By the model calculation, the concentration of regenerant is one of the controlling factors in determining the volume of regenerant and fractional regeneration.

3) The effects of flow rate and bed length on regeneration results are not so influential a parameter in comparison with concentration of regenerant and particle size.

4) The particle size is defined as one of the influential design parameters in determining the cost of regeneration. Therefore, the selection of particle size should be considered from the viewpoint not only of adsorption but also of regeneration performance. It can be selected from the results of this model calculation.

Acknowledgment

K.-S. Ha is grateful to the Ministry of Education, Science and Culture, Japan for a scholarship for his study. The work was partially supported by the Japan Steel Industry Environmental Pollution Control Foundation.

Nomenclature

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
<th>Unit</th>
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<tr>
<td>$A, B$</td>
<td>constants for Eq. (8)</td>
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</tr>
<tr>
<td>$a_v$</td>
<td>external surface area of particles unit packed volume</td>
<td>$[m^2/m^3]$</td>
</tr>
<tr>
<td>$B_r$</td>
<td>regenerant bed volumes</td>
<td>[B.V.]</td>
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<tr>
<td>$C_{Na^+}$</td>
<td>sodium ion concentration</td>
<td>[mol/m³]</td>
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<tr>
<td>$C_{Na^+,-S}$</td>
<td>sodium ion concentration at particle surface</td>
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<td>$C_{NH_4^+}$</td>
<td>average ammonium ion concentration in effluent</td>
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<td>[mol/m³]</td>
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<tr>
<td>$D_p$</td>
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<tr>
<td>$D_s$</td>
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Table 4. Constants of A and B in Eq. (8)

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<th>B</th>
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<tr>
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Table 5. Particle diameters used in fixed bed for ammonium removal

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<tr>
<td>McLaren et al.</td>
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<td>Barrer et al.</td>
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<tr>
<td>Kinoshita, M.</td>
<td>1.4-0.59</td>
<td>Ref. 3</td>
</tr>
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</table>
\[ E_t = \text{dispersion coefficient in } L\text{-direction in the bed} \]  
\[ K_s = \text{selectivity coefficient of sodium ion} \]  
\[ k_f = \text{liquid-to-particle mass transfer coefficient} \]  
\[ L = \text{length of bed} \]  
\[ P_t = D_s \cdot u/E_s \]  
\[ P_x = L \cdot u/E_s \]  
\[ q_{Na+} = \text{particle phase sodium ion concentration at surface} \]  
\[ q_{Na+} = \text{particle phase sodium ion concentration} \]  
\[ r, R = \text{particle radius} \]  
\[ R_f = \text{fractional regeneration} \]  
\[ T = t/t_0 \]  
\[ t = \text{time} \]  
\[ t_0 = L/u \]  
\[ u = \text{superficial fluid velocity} \]  
\[ X = C_{Na+}/C_{T,0} \]  
\[ X_s = C_{Na+}/C_{T,0} \]  
\[ Y = q_{Na+}/q_0 \]  
\[ Y_x = q_{Na+}/q_0 \]  
\[ Z = z/L \]  
\[ a_1 = t_0 \cdot k_{av} \]  
\[ a_2 = D_s \cdot t_0/R \]  
\[ a_s = k_f \cdot R/\beta \cdot D_s \cdot \rho_s \]  
\[ \beta = q_0/C_{T,0} \]  
\[ \varepsilon = \text{void fraction} \]  
\[ \rho = r/R \]  
\[ \rho_s = \text{particle density} \]  

**Literature Cited**