Solid/liquid Mass Transfer Correlated to Mixing Pattern in a Mechanically-stirred Vessel

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(Received on March 27, 2014; accepted on August 22, 2014)

Cold model experiment on ion-exchange reaction between pearlite particles and HCl aq. was carried out in order to understand the effect of particles dispersion and operating factors on solid/liquid mass transfer rate in a mechanically-stirred vessel. Inner diameter of vessel was varied in conjunction with both bath depth as 400 (base) and 300 mm. Rotation speed and volume ratio of particles to liquid were changed between 0–240 rpm, 0.02–0.24 (–), respectively.

When rotating speed increased, solid/liquid mass transfer rate increased moderately both in the regions I and III, whereas it increased in the region II. When impeller depth decreased, it was kept almost constant in the region I, increased in the region II and increased moderately in the region III. Solid/liquid mass transfer rate changed less than liquid/liquid one in the region II when rotation speed and impeller height were changed, whereas both of solid/liquid and liquid/liquid mass transfer rates were kept almost constant in the region I. The dimensionless equation on solid/liquid mass transfer rate of each region was given as a function of Sherwood number, Reynolds number, volume ratio of particles to liquid and bath depth normalized by vessel diameter. Dispersion ratio in the region II was ranged by solid/liquid mass transfer coefficient and rotation speed or impeller height of the transitions I–II and II–III. Solid/liquid mass transfer rate of mechanical stirring was larger than that of gas injection practice for the same supplied rate of energy into bath.

KEY WORDS: steelmaking; mechanical stirring; slag-metal reaction; mixing pattern; solid/liquid mass transfer; liquid/liquid mass transfer; ion exchange reaction.

1. Introduction

Hot metal desulfurization by mechanical stirring has been re-evaluated for high reaction efficiency and stable operation, and approach to accelerate desulfurization has been proposed in a variety of methods.5–8)

On the other hand, authors carried out the cold model experiment on solid/liquid mixing pattern,9) which was clarified into three types as well as liquid/liquid one.10) Figure 1 shows the categorized three types of solid/liquid mixing pattern. I: region where solid/liquid interface does not reach at upper part of impeller and has no dispersion, II: region where solid arrives at upper part of impeller and dispersion of particles starts into liquid phase, III: region where gas/liquid interface arrives at upper part of impeller and solid and gas dispersion occurs hard into liquid. Solid/liquid mixing pattern transits from I to II, and from II to III as impeller depth, H, decreased and rotation speed, N, increased. And some of authors11) revealed that liquid/liquid mass transfer rate showed characteristic trend depending on liquid/liquid mixing pattern. It was kept nearly constant at lower level in the region I, monotonically increased in the region II except near the region III and its increasing rate decreased in the region II near the region III. However, there is few cold model investigation on the effect of solid/liquid mixing pattern on solid/liquid mass transfer rate with particles lighter...
than liquid, with the exception that Nakai et al.\textsuperscript{12} studied a relation between solid/liquid mixing pattern by cold model experiment and desulfurization rate by hot model experiment.

Thus, in order to understand the effect of particles dispersion and operating factors on solid/liquid mass transfer rate, cold model experiment on ion-exchange reaction between pearlite particles and HCl aq. was carried out in a mechanically-stirred vessel. Additionally, solid/liquid mass transfer rate of mechanical stirring was compared with that of gas stirring under the same supplied rate of energy, and with liquid/liquid one.

2. Experimental

2.1. Solid/liquid Mass Transfer

Figure 2 shows a schematic view of experimental apparatus. Perlite particles and $2.35 \times 10^{-4}$ M of HCl aq. were put into an acrylic vessel of inner diameter, $D$ (mm) and height, $L$ (mm) so as to be static bath depth, $H_0$ (mm). Pearlite (density, $\rho_d=100$ kg/m$^3$, Mitsui Mining & Smelting Co., Ltd.) was arranged in the almost equal size, immersed into $2 \times 10^{-3}$ M of NaOH aq. for 6 hours and dried in the room for 12 hours. Four blades of impeller was used as shown schematically in Fig. 3. The impeller whose diameter was expressed as $d_i$ mm, thickness as $b_i$ mm and width as $w_i$ mm was set in the central axis of the vessel.

$\text{H}^+$ in HCl aq. reacts with $\text{Na}^+$ on pearlite surface according to mechanical stirring. $\text{H}^+$ was measured by electrical conductivity meter (Automatic system research, mk-250EC) shown as EC in Fig. 2 and mass transfer rate was calculated from its temporal change. Experimental condition is shown in Table 1. The operating factors such as rotating speed, N (rpm), impeller depth, H (mm), volumetric ratio of solid to liquid, $V_S/V_L$ were changed on the basis of $D=H_0=300$ mm, $(d_i, b_i, w_i)=(116$ mm, 67 mm, 31 mm) and $d_p=2.3$ mm.

Gas injection practice was carried out for comparison with mechanical stirring. The nozzle with four holes at regular intervals was set in the central axis of the vessel. Gas was injected horizontally into liquid. Experimental condition is shown in Table 2. Gas flow rate, $Q$ (L/min) and nozzle depth, H (mm) were changed under $D=H_0=300$ mm, hole diameter, $d=3$ mm and $V_S/V_L=0.12$.

2.2. Conversion Principle of Conductivity to Concentration of Hydrochloric Acid

As hydrochloric acid is strong acid, it is ionized perfectly by Eq. (1) in water.

$$\text{HCl} \rightarrow \text{H}^+ + \text{Cl}^- \text{........................ (1)}$$

Mole electric conductivity, $\lambda$, of strong electrolyte defined by electric conductivity, $\kappa$ (S/cm), per electrolyte concentration, C (mol/L) was expressed as Eq. (2).

$$\lambda = 10^3 \kappa / C = \lambda_{\infty} + \lambda_{\text{(CT)}} = \lambda_{\infty} \text{........................ (2)}$$

where $\lambda_{\infty}$: infinitely-diluted mole electric conductivity (S cm$^2$/mol), $\lambda_{\text{(H)}} = 349.8$ (S cm$^2$/mol), $\lambda_{\text{(Cl)}} = 76.4$ (S cm$^2$/mol).\textsuperscript{13} Thus, Eq. (2) became

$$\lambda = 10^3 \kappa / C = \lambda_{\infty} = 349.8 + 76.4 = 426.2 \text{ ........ (3)}$$

| Variables | Values |
|-----------|--------|
| Vessel inner diameter, D (mm) | 300, 400 |
| Bath depth, $H_0$ (mm) | 300, 400 |
| Impeller diameter, $d_i$ (mm) | 116, 156 |
| Impeller thickness, $b_i$ (mm) | 67, 85 |
| Impeller width, $w_i$ (mm) | 31, 42 |
| Rotating speed, N (rpm) | 0–335 |
| Impeller depth, H (mm) | 0–300 |
| Solid-liquid ratio, $V_S/V_L$ (–) | 0.02, 0.06, 0.12, 0.24 |
| Dispersion phase | Perlite |
| Mean diameter of perlite, $d_p$ (mm) | 1.3, 2.3, 3.1 |

| Variables | Values |
|-----------|--------|
| Inner diameter, D(mm) | 300 |
| Bath depth, $H_0$ (mm) | 300 |
| Gas flow rate, Q (L/min) | 5, 20, 40 |
| Nozzle depth, H (mm) | 100, 150, 200, 250 |
| Solid-liquid ratio, $V_S/V_L$ (–) | 0.12 |
| Hole diameter, $d$ (mm) | 3 |
| Number of hole | 4 |
Equation (4) was verified by the experiment where a given amount of hydrochloric acid was dissolved in ion-exchanged water of 100 mL and electric conductivity was measured. Relation between electric conductivity and concentration of hydrochloric acid is shown in Fig. 4(a). “Theoretical” in Fig. 4(a) means Eq. (4). Theoretical electric conductivity values agreed well with measured ones. Figure 4(b) shows relation between measured and theoretical electric conductivity. It was found that concentration of hydrochloric acid is obtained from Eq. (4) by measuring electric conductivity from these two figures.

3. Results and Discussion

3.1. Effect of Solid/liquid Mixing Pattern on Mass Transfer

3.1.1. Calculation of Solid/liquid Mass Transfer Coefficient

Ion-exchanged reaction of pearlite is expressed by Eq. (5).

\[
R - Na^+ + HCl \rightarrow R - H^+ + NaCl \quad \text{(5)}
\]

Ion-exchanged reaction rate is determined by diffusion in the fluid film, chemical reaction on the solid/liquid interface or diffusion in the solid phase. As the rate is normally controlled by the diffusion in the fluid film\(^{14}\) at the early stage, solid/liquid mass transfer rate is given by Eq. (6).

\[
-V dC / dt = k_{s-l} a_{s-l} (C - C_i) \quad \text{(6)}
\]

where, \(k_{s-l}\): solid/liquid mass transfer coefficient (m/s), \(a_{s-l}\): total surface area of pearlite particles (m\(^2\)), \(C\): HCl concentration (kgmol/m\(^3\)), \(C_i\): HCl concentration on pearlite (kgmol/m\(^3\)), \(V\): volume of HCl aq. (m\(^3\)). On the assumption that \(C_i\) equals to be 0,\(^{15,16}\) integration of Eq. (6) gives Eq. (7), where \(C_0\): initial HCl concentration (kgmol/m\(^3\)).

\[
\ln \left( \frac{C_0}{C} \right) = k_{s-l} a_{s-l} t \quad \text{(7)}
\]

Some examples of temporal change in \(C_0/C\) are shown in Fig. 5. As logarithmic \(C_0/C\) increased almost linearly with time, the slope enabled us to achieve \(k_{s-l}\). The \(a_{s-l}\) was obtained from the average diameter regarding as sphere. As perlite particles moved quickly on HCl aq. even at the minimum rotation speed, \(N=40\) rpm in the experimental condition, total surface area of perlite particles was used for Eq. (7).

3.1.2. Effect of Rotation Speed on Solid/liquid Mass Transfer Rate

Relation between \(k_{s-l}\) and rotating speed is shown in Fig. 6. \(V_s/V_l\) is fixed to 0.12 and \(H\) is varied between 50 and 200 mm. Each plot is differentiated by solid/liquid mixing pattern\(^9\) described in chapter 1. The \(k_{s-l}\) value was near 0 for \(N=0\) rpm, which meant that solid/liquid mass transfer rate depended on mechanical stirring. \(k_{s-l}\) increased with the increase in \(N\) due to the stirring energy in bath. \(k_{s-l}\) increased moderately with the increase in \(N\) in the region I, the increasing rate of \(k_{s-l}\) against \(N\) became larger in the region II, and it returned to the moderate increase in the region III. The reason of the behavior in the region II is why solid particles are partly dispersed in continuous liquid phase where solid/liquid stirring is seemed to be more severe than the floated solid particles/liquid. The additional discussion will be made in section 3.4. On the other hand, both in the region I where no solid particles disperse and in the region III where almost all of solid particles exist in liquid, the increase in \(k_{s-l}\) depends only on the stirring energy by impeller rotation.

3.1.3. Effect of Impeller Height on Solid/liquid Mass Transfer Rate

Relation between \(k_{s-l}\) and depth, \(H\), from gas/solid interface to upper part of impeller is shown in Fig. 7. \(V_s/V_l\) is fixed to be 0.12 and \(N\) is varied between 59 and 244 rpm. \(k_{s-l}\) increased with the decrease in \(H\) because of the increasing ratio of dispersed particles in the region II. However, \(k_{s-l}\) was kept to be almost constant in the region I shown in \(N=59\) rpm, because the change in \(H\) gave no change in stirring energy nor dispersion.
3.1.4. Effect of Ratio of Solid/liquid on Solid/liquid Mass Transfer Rate

Effect of \( V_S/V_L \) on \( k_{S-L} \) for \( N=111 \) rpm is shown in Fig. 8. Both in the regions of II and III, \( k_{S-L} \) had no change in \( V_S/V_L \). However, \( k_{S-L} \) in the region I increased with the decrease in \( V_S/V_L \) for the equal \( H \) because dispersed solid particles contacted with liquid more sufficiently.

3.1.5. Effect of Impeller Diameter on Solid/liquid Mass Transfer Rate

Relation between \( k_{S-L} \) and \( H \) in the region II is shown in Fig. 9. Here, both impeller diameter and rotation speed changed so as to supplied rate, \( \varepsilon \), of energy into bath and \( V_S/V_L \) was fixed to be \( 1.86 \times 10^{-2} \) w/kg, 0.12 (–), respectively. \( \varepsilon \) was calculated from the method in section 3.2. Solid/liquid mass transfer rate has no change for the same \( \varepsilon \).

3.1.6. Effect of Vessel Diameter on Solid/liquid Mass Transfer Rate

Relation between \( k_{S-L} \) and \( H \) in the region II is shown in Fig. 10. Here, both impeller diameter and rotation speed changed and \( V_S/V_L \), \( d_i \) was fixed to be 0.12 (–), 116 mm, respectively. \( \varepsilon \) equaled to \( 1.86 \times 10^{-2} \) w/kg for \( (D,N)=(300 \text{ mm}, 144 \text{ rpm}) \) as well as \( (400 \text{ mm}, 178 \text{ rpm}) \). Both of larger vessel diameter and rotation speed increased \( k_{S-L} \), for the same \( \varepsilon \) value.

3.1.7. Effect of Pearlite Diameter on Solid/liquid Mass Transfer Rate

Relation between \( k_{S-L} \) and \( H \) is shown in Fig. 11. Pearlite diameter, \( d_p \) was changed from 1.3 mm to 3.1 mm, although \( V_S/V_L \), \( N \) and \( D \) are fixed to be 0.12 (–), 144 rpm, 300 mm, respectively. \( k_{S-L} \) did not depend on pearlite diameter.

3.2. Comparison between Solid/liquid and Liquid/liquid Mass Transfer Rates

Relations between \( k_{S-L} \) and \( H \), and liquid/liquid capacity coefficient, \( k_{L-L} \) (m$^3$/s) and \( H^{111} \) are shown in Fig. 12. Although both of solid/liquid and liquid/liquid mass transfer rates increased with the decrease in \( H \) in the region II, the increasing ratio of the liquid/liquid is more remarkable than that of the solid/liquid. It is due to the fact that the decrease in \( H \) causes the increase in liquid/liquid interfacial area, \( a_{L-L} \), as the dispersed phase increases, whereas it is not expected for the solid/liquid. Both of liquid/liquid and solid liquid mass transfer rates were almost constant in the region I.
3.3. Dimensionless Equation of Solid/liquid Mass Transfer Rate

Assuming that a single particle moves into liquid at a terminal velocity, Froessling formula\(^\text{17}\) on solid/liquid mass transfer rate was introduced as Eq. (8). Semi-empirical equations\(^\text{18}\) for particles/liquid mass transfer rate in a mechanically-stirred vessel were proposed as Eq. (9), based on Eq. (8).

\[
\text{Sh} = 2 + 0.6 \text{Re}_p^{1/2} \text{Sc}^{1/3} \\
\text{Sh} = A + \text{BRe}_p^{n} \text{Sc}^m
\]

where Sh: Sherwood number (=k\(_{S-L}\)/D\(_{HCl}\)), Re\(_p\): particle Reynolds number (=\(\rho N_\text{dp}d_i/\mu\)), Sc: Schmidt number (=\(\mu/\rho D_{HCl}\)), D\(_{HCl}\): diffusivity coefficient of HCl (m\(^2\)/s), \(\rho\): density of HCl aq. (kg/m\(^3\)), \(\mu\): viscosity coefficient of HCl (Pa·s). A, B, n and m in Eq. (9) are factors determined by the experiment.

The k\(_{S-L}\) values were affected on \(V_s/V_L\) in addition to N in the region I as seen in Fig. 8, and on H in the regions II and III like Figs. 6–12. Thus, the dimensionless Eq. (9) for region I and II was modified as Eqs. (10) and (11), respectively.

The region I;

\[
\text{Sh} = 2 + \text{BRe}_p^{n} \text{Sc}^{1/3} \left( V_s/V_L \right)^p \hspace{1cm} (10)
\]

The region II and III;

\[
\text{Sh} = 2 + \text{BRe}_p^{n} \text{Sc}^{1/3} \left( H/D \right)^q \hspace{1cm} (11)
\]

where p and q are factors obtained from regression analysis.

The dimensionless equation of each region is obtained as shown in Fig. 13. Data in the region II dispersed more widely than those in the regions I or III due to partly dispersed two reaction zones. Factors (B, n, p) in the region I were (0.071, 0.65, –0.24) obtained from the data of N \(\geq 40\) rpm (Re\(_p\) \(\geq 4.0\)) in this experimental condition, whereas (B, n, q) in the regions II and III are (0.063, 0.87, –0.38), (0.28, 0.46, –0.09), respectively. Power of Re\(_p\) in the region II was larger than any other regions and that of (H/D) in the region II was smaller than that in the region III. It result from the change in dispersion behavior of particles which generates only in the region II. From the point of dispersion ratio of particles, the change in k\(_{S-L}\) is described in the following section 3.4.

The dimensionless equation of all data in the regions I to III is obtained from Fig. 14. As power of \((V_s/V_L)\) was negligible small, (B, n, q) became (0.079, 0.74, –0.24).

3.4. Dispersion Ratio in the Region II

All particles exists on liquid in the region I and most particles disperse into liquid in the region III. On the other hand, in the region II dispersion of particles increases with the increase in rotation speed and the decrease in distance between free surface and impeller position. Defining k\(_{S-L}\) between floating particles and liquid as k\(_I\), and that between dispersed particles and liquid as k\(_{III}\), k\(_{II}\) of the region II, k\(_{II}\) is expressed by the dispersed particles ratio, \(\alpha\), as follows.

\[
k_{II} = (1 - \alpha)k_I + \alpha k_{III} \hspace{1cm} (12)
\]

Rearranging Eq. (12),
Here, $\alpha$ is expressed using the $k_{S-L}$ value of the region I near the transition I–II as $k_I$ and the $k_{S-L}$ value of the region III near the transition II–III as $k_{III}$.

When $H$ is changed under constant $N$, $H$ of the transition I–II and II–III are defined as $H_{II,I}$, $H_{II,III}$, respectively. Relation between $\alpha$ and $(H_{II}-H_{II,I})/(H_{II,III}-H_{II,I})$ is shown in Fig. 15. $H_{II}$ means $H$ in the region II. Dispersion ratio in the region II was ranged by normalized $H$. Change rate of $\alpha$ was shelving in the center of the region II and sharp near the regions I and III.

When $N$ is changed under constant $H$, $N$ of the transition I–II and II–III are defined as $N_{II,I}$, $N_{II,III}$, respectively. Relation between $\alpha$ and $(N_{II}-N_{II,I})/(N_{II,III}-N_{II,I})$ is shown in Fig. 16. $N_{II}$ means $N$ in the region II. As well as Fig. 15, $k_{S-L}$ values of the region I near the transition I–II and of the region III near the transition II–III were used as constant $k_I$, $k_{III}$, respectively. However, as $k_I$ and $k_{III}$ in the region II change normally with $N$ because of change in supplied rate of energy into bath, $k_I$ is underestimated, whereas $k_{III}$ overestimated in Fig. 16. Thus, it was lead to larger $\alpha$ value than the actual one in Eq. (13). From Fig. 16, dispersion ratio in the region II was roughly ranged by normalized $N$ in spite of an overestimate of $\alpha$ curve.

3.5. Comparison of Solid/liquid Mass Transfer Rate between Mechanical Stirring and Gas Injection

3.5.1. Solid/liquid Mass Transfer Rate of Gas Injection

Relation between $k_{S-L}$ and the distance between gas/solid surface and hole position of the immersed nozzle, $H$, for the various gas flow rate, $Q$, is shown in Fig. 17. $k_{S-L}$ increased with the increase in $H$ and $Q$, because supplied rate of energy into bath increased.

3.5.2. Comparison of Solid/liquid Mass Transfer Rate between Mechanical and Gas Stirrings

Here, solid/liquid mass transfer rate was compared between mechanical and gas stirring in the case of the same supplied rate of energy into bath. Supply rate of energy by gas stirring expressed as $e_g$ (W/kg) is given by Eq. (14).

$$e_g = 0.0143QT/W_w \log (1+H/10.3) \quad \ldots \ldots \ldots \ldots (14)$$

where $Q$: gas flow rate (NL/min), $T$: bath temperature (K), $W_w$: mass of water (kg), $H$: depth from free surface to the position of gas injection (m).

On the other hand, supplied rate of energy by mechanical stirring expressed as $e_i$ (W/kg) is given by Eq. (15).

$$e_i = N_P \rho_w \rho_w N^5 d_i^5 / W_w \quad \ldots \ldots \ldots \ldots (15)$$

where $N_P$: power number (–), $\rho_w$: liquid density (kg/m$^3$), $N$: rotation speed (rps), $d_i$: impeller diameter (m). $N_P$ is calculated from Nagata’s formula.$^{20}$

Both of $e_g$ for $Q=5, 10$ and $20$ L/min were calculated from Eq. (14) for $D=H_{II}=0.3$ m, $H=0.1$ m and $T=298$ K. Rotation speed for mechanical stirring was obtained by substituting $e_i$ values corresponding to the above calculated $e_g$ ones and $N_P$ given by Horiiuchi et al.$^{11}$ into Eq. (15). As shown in Table 3, the rotation speeds, $N=111, 146$ and $189$ rpm correspond to gas flow rates, $Q=5, 10$ and $20$ L/min, respectively.

Comparison of $k_{S-L}$ between mechanical and gas stirring
increase in rotation speed, the decrease in impeller height and volumetric ratio of solid to liquid in the region I, although it was not influenced by particles diameter. Solid/liquid mass transfer rate did not depend on impeller diameter, but it increased with the increase in vessel diameter for the same supplied rate of energy into bath.

(3) Solid/liquid mass transfer rate changed less than liquid/liquid one in the region II when rotation speed and impeller height were changed, whereas both of solid/liquid and liquid/liquid mass transfer rates were kept almost constant in the region I.

(4) The dimensionless equation of each region was obtained as follows:
- Region I: $Sh = 2 + 0.071Re^{0.65}Sc^{1/3}(V_s/V_L)^{-0.24}$
- Region II: $Sh = 2 + 0.063Re^{0.87}Sc^{1/3}(H/D)^{-0.38}$
- Region III: $Sh = 2 + 0.28Re^{0.46}Sc^{1/3}(H/D)^{-0.09}$

(5) Dispersion ratio in the region II was ranged by solid/liquid mass transfer coefficient and rotation speed or impeller height of the transitions I–II and II–III.

(6) Solid/liquid mass transfer rate of mechanical stirring was larger than that of gas injection practice for the same supplied rate of energy into bath.

Acknowledgements

This work was performed under the Cooperative Research Program of “Network Joint Center for Materials and Devices”.

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