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Separation of Mercury from Aqueous Mercuric Chloride Solutions by Onion Skins

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Abstract

The separation of mercury from aqueous $HgCl_2$ solutions by onion skins (outermost coat) was studied both experimentally and theoretically. The distribution equilibria were measured by the batchwise method. The experimental results revealed that onion skin is a useful material for separating mercury from aqueous systems. The distribution data obtained at 25°C were analyzed by using the theory based on the law of mass action. The separation of dissolved mercury by onion skins was found to be a process accompanied by an ion-exchange reaction of the cationic complex $HgCl^+$ and an adsorption of the neutral complex $HgCl_2$. The equilibrium constants of the ion-exchange and adsorption processes at 25°C and the mercury-binding capacity of onion skins were determined. Further, it was found that the distribution equilibrium of mercury is comparatively insensitive to temperature.

INTRODUCTION

The separation of mercury from an aqueous environment by adsorption operations is an important treatment process for the control of water pollution. Synthetic polymers, such as ion-exchange resins and chelating resins, have been widely used as adsorbents for this purpose. Synthetic

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resins can effectively separate mercury from solution, but they are usually expensive.

Alternatively, it has been found that natural materials containing proteins, tannins, polyphenolic substances, and other polymers (such as wool, peanut skins, bark, and orange peel) have a binding ability toward mercury and thus reduce the mercury concentration in aqueous solutions of $HgCl_2$ or CH_3HgCl (*1–5*). The experimental results of these investigations suggest that some natural materials may be potentially useful as low-cost materials for the separation of mercury from contaminated water. However, little information is available on the equilibrium distribution of mercury between the solid and liuqid phases, except that sorption of mercuric salts by wool roughly follows a Freundlich isotherm at a given pH of the solution (*1, 2*).

In addition to the aforementioned natural materials, the effectiveness of onion skin for separating mercury may also be expected since natural chelating substances in onion skin, such as quercetin, contain functional groups capable of forming complexes with metal ions. However, the present system has not yet been investigated.

This work was undertaken to clarify the stoichiometric relation in the separation of dissolved mercury by onion skins, to evaluate the distribution equilibrium constant of mercury between solid and liquid phases, and to study the mercury-binding capacity of onion skins. The distribution ratios of mercury were measured by the batchwise method. The experimental results were analyzed by using the theory based on the law of mass action.

EQUILIBRIUM DISTRIBUTION OF MERCURY

The following chemical equilibria of Hg^{2+} and Cl^- ions and their various complexes are established in aqueous $HgCl_2$ solutions:

$$Hg^{2+} + iCl^- \rightleftharpoons HgCl_i^{2-i} \qquad (i = 1\text{–}4) \tag{1}$$

$$\beta_i = \frac{[HgCl_i^{2-i}]}{[Hg^{2+}][Cl^-]^i} \tag{2}$$

where β_1–β_4 are the overall stability constants of the chloro complexes of mercury, with values of $\beta_1 = 5.8 \times 10^6$ dm^3/mol, $\beta_2 = 1.4 \times 10^{13}$ dm^6/mol^2, $\beta_3 = 9.7 \times 10^{13}$ dm^9/mol^3, and $\beta_4 = 1.3 \times 10^{15}$ dm^{12}/mol^4 at 25°C and infinite dilution (*6*). The total concentration C_M of mercury in the liquid phase is obtained from Eq. (2) as

$$C_M = [Hg^{2+}] + [HgCl^+] + [HgCl_2] + [HgCl_3^-] + [HgCl_4^{2-}]$$
$$= [Hg^{2+}](1 + \beta_1[Cl^-] + \beta_2[Cl^-]^2 + \beta_3[Cl^-]^3 + \beta_4[Cl^-]^4) \tag{3}$$

In the range of the Cl^- ion concentrations studied in the present work, the third term on the right-hand side of Eq. (3) is greater by two orders of magnitude than the other terms, so Eq. (3) can be written as

$$C_M = \beta_2[Hg^{2+}][Cl^-]^2 \tag{4}$$

This indicates that the dissolved $HgCl_2$ in the solution exists primarily as the neutral complex $HgCl_2$, with much smaller concentrations of Hg^{2+}, $HgCl^+$, $HgCl_3^-$, and $HgCl_4^{2-}$.

When onion skin is mixed with aqueous solutions of $HgCl_2$, the functional groups in the solid phase appear to act as a cation exchanger, and the cation Hg^{2+} and the cationic complex $HgCl^+$ alone in the liquid phase may exchange with the H^+ ions in the solid phase according to the following ion-exchange reactions:

$$Hg^{2+} + 2\overline{HR} \rightleftharpoons \overline{HgR_2} + 2H^+ \tag{5}$$

$$HgCl^+ + \overline{HR} \rightleftharpoons \overline{HgClR} + H^+ \tag{6}$$

where the overbar denotes a species in the solid phase, and HR represents the acid-type functional groups in the chelating substance (quercetin), capable of binding mercury. From the mass action law the equilibrium constants K_{Hg} and K_{HgCl} of the Ion-Exchange Reactions (5) and (6) are given by

$$K_{Hg} = \frac{[\overline{HgR_2}][H^+]^2}{[Hg^{2+}][\overline{HR}]^2} \tag{7}$$

$$K_{HgCl} = \frac{[\overline{HgClR}][H^+]}{[HgCl^+][\overline{HR}]} \tag{8}$$

The onion skin might also adsorb the neutral complex $HgCl_2$ in the liquid phase according to

$$HgCl_2 \rightleftharpoons \overline{HgCl_2} \tag{9}$$

The equilibrium constant of this adsorption is defined as

$$K_{HgCl_2} = [\overline{HgCl_2}]/[HgCl_2] \tag{10}$$

The total concentration $\overline{C_M}$ of mercury in the solid phase is given from Eqs. (2), (7), (8), and (10) as

$$\begin{aligned}\overline{C_M} &= [\overline{\mathrm{HgR_2}}] + [\overline{\mathrm{HgClR}}] + [\overline{\mathrm{HgCl_2}}] \\ &= \left\{\left(\frac{K_{\mathrm{Hg}}}{\beta_2}\right)\frac{[\overline{\mathrm{HR}}]^2}{[\mathrm{H^+}]^2[\mathrm{Cl^-}]^2} + \left(\frac{K_{\mathrm{HgCl}}\beta_1}{\beta_2}\right)\frac{[\overline{\mathrm{HR}}]}{[\mathrm{H^+}][\mathrm{Cl^-}]} \right. \\ &\quad \left. + K_{\mathrm{HgCl_2}}\right\}\beta_2[\mathrm{Hg^{2+}}][\mathrm{Cl^-}]^2 \end{aligned} \tag{11}$$

The distribution ratio D_M of mercury between the solid and liquid phases is obtained from Eqs. (4) and (11) as

$$\begin{aligned} D_M &= \overline{C_M}/C_M \\ &= \left(\frac{K_{\mathrm{Hg}}}{\beta_2}\right)\frac{[\overline{\mathrm{HR}}]^2}{[\mathrm{H^+}]^2[\mathrm{Cl^-}]^2} + \left(\frac{K_{\mathrm{HgCl}}\beta_1}{\beta_2}\right)\frac{[\overline{\mathrm{HR}}]}{[\mathrm{H^+}][\mathrm{Cl^-}]} + K_{\mathrm{HgCl_2}} \end{aligned} \tag{12}$$

The first term on the right-hand side corresponds to the ion-exchange equilibrium of the cation Hg^{2+} representing Eq. (5), the second term to the ion-exchange equilibrium of the cationic complex $HgCl^+$ representing Eq. (6), and the third term to the adsorption equilibrium of the neutral complex $HgCl_2$ representing Eq. (9). Depending on the composition of the solution used, one or two of the three terms in Eq. (12) may be negligible compared to the others.

In one of the limiting cases, in a region where the adsorption of the neutral complex $HgCl_2$ is controlling, the distribution ratio is independent of $[H^+]$ and $[Cl^-]$, and

$$D_M \simeq K_{\mathrm{HgCl_2}} \tag{13}$$

In a region where the ion-exchange reaction of $HgCl^+$ ion is solely responsible for the distribution of mercury, Eq. (12) reduces to

$$D_M \simeq \left(\frac{K_{\mathrm{HgCl}}\beta_1}{\beta_2}\right)\frac{[\overline{\mathrm{HR}}]}{[\mathrm{H^+}][\mathrm{Cl^-}]} \tag{14}$$

Thus the distribution ratio is inversely proportional to $[H^+]$ and $[Cl^-]$ for constant $[\overline{HR}]$. In a region where the ion-exchange reaction of Hg^{2+} ions contributes predominantly to the distribution, Eq. (12) becomes

$$D_M \simeq \left(\frac{K_{\mathrm{Hg}}}{\beta_2}\right)\frac{[\overline{\mathrm{HR}}]^2}{[\mathrm{H}^+]^2[\mathrm{Cl}^-]^2} \tag{15}$$

the distribution ratio varying as $[H^+]^{-2}$ and $[Cl^-]^{-2}$ for constant $[\overline{HR}]$.

EXPERIMENTAL

The onion skin (outermost coat of onion) used in this study was yellow danvers, native to America, and was obtained from a farm in Osaka Prefecture, Japan. The solid sample was ground to pass through a 500-μm screen and to be retained on a 250-μm screen.

The liquids used were aqueous $HgCl_2$ solutions ranging in initial concentration from 1.23×10^{-5} to 2.39×10^{-3} mol/dm³. In preparing the $HgCl_2$ solutions, the pH of the solution was varied by using NaOH or HCl. In some runs a small amount of NaCl or HCl was added to adjust the Cl^- ion concentrations of the solution. The ionic strength of the solution was very low (less than 9×10^{-3} mol/dm³).

All equilibrium experiments were carried out by the batchwise method and most of data were taken at 25°C. A mixture of 1.0 g ground onion skin and a known volume of aqueous $HgCl_2$ solution was shaken in a flask and allowed to attain equilibrium. After about 48 h, the mixture was filtered and the filtrate was saved for analysis.

The concentration of mercury in the solution was determined by chelatometry in the range of high concentrations and by spectrophotometry (7) in the range of low concentrations. The content of mercury in the solid sample was determined from the difference between the initial and final concentrations of mercury in the solution. The distribution ratio D_M of mercury was calculated from the number of moles per unit mass of dry solid $\overline{C_M}$ divided by that per unit volume of solution C_M. The pH of the solution after equilibrium was measured by using a pH meter. The equilibrium concentration of Cl^- ions in the solution was estimated as the sum of the amount of HCl or NaCl added to the solution and that of Cl^- ions liberated by the following reaction:

$$\mathrm{HgCl_2} + \overline{\mathrm{HR}} \rightleftharpoons \overline{\mathrm{HgClR}} + \mathrm{H}^+ + \mathrm{Cl}^- \tag{16}$$

which is the overall reaction composed of Reaction (1) with $i = 1$ and 2 and Reaction (6). Here, the amount of Cl^- ions liberated was evaluated to be equivalent to the amount of formation of $\overline{HgClR}$, which was determined in a manner that will be described later. This procedure for the estimation of equilibrium concentrations of Cl^- ions is justified because the extent of the Hg^{2+} ion-exchange reaction is negligible under the present experimental conditions, as described below, and the amounts of $HgCl_3^-$ and $HgCl_4^{2-}$ formed are very small, as described above. The concentration of Na^+ ions in the solution was analyzed with an atomic absorption spectrophotometer, and the difference in the concentrations before and after equilibration was found to be negligibly small.

The experimental conditions are given in Table 1.

RESULTS AND DISCUSSION

Experimental results obtained at 25°C are shown in Fig. 1, where the distribution ratio D_M of mercury for each solution, with almost constant $[Cl^-]$, is plotted on logarithmic coordinate against $[H^+]$. It can be seen in Fig. 1 that for each solution of $[Cl^-] = 1.02 \times 10^{-5}$ to 1.20×10^{-5}, 8.06×10^{-5} to 8.76×10^{-5}, and 3.04×10^{-4} to 3.54×10^{-4} mol/dm^3 (except at the three lower values of $[H^+]$), the distribution ratio D_M decreases rapidly with an increase in the $[H^+]$ value and is inversely proportional to $[H^+]$. For the solution where $[Cl^-] = 5 \times 10^{-3}$ mol/dm^3, however, the distribution ratio D_M decreases gradually with increasing $[H^+]$ value and finally reaches a finite value. Further, it is also seen that the distribution ratio D_M decreases greatly as the $[Cl^-]$ value increases. The cross plot of D_M vs $[Cl^-]$ at a constant value of $[H^+]$ shows that the distribution ratio D_M is in inverse proportion to $[Cl^-]$, except for the case where $[Cl^-] = 5 \times 10^{-3}$ mol/dm^3.

These observed effects of $[H^+]$ and $[Cl^-]$ values on the distribution ratio D_M are the same as those predicted theoretically from Eqs. (13) and (14), provided $[\overline{HR}]$ remains unchanged. On the other hand, the behavior expected from Eq. (15) with respect to $[H^+]$ and $[Cl^-]$ was not observed, indicating that Ion-Exchange Reaction (5) of the cation Hg^{2+} made a negligible contribution to the distribution ratio of mercury. Thus it may be concluded that the distribution of mercury between the solid and liquid phases is caused by Ion-Exchange Reaction (6) and Adsorption Reaction (9): for the solution where $[Cl^-] = 5 \times 10^{-3}$ mol/dm^3, the adsorption of the neutral complex $HgCl_2$ is predominant, while for the solutions where $[Cl^-]$ is below 3.54×10^{-4} mol/dm^3, the ion-exchange reaction of the cationic complex $HgCl^+$ is controlling.

TABLE 1
Experimental Conditions and Compositions of Solutions Used

Initial concentration of mercury, $C_{M0} \times 10^5$ mol/dm^3	Initial concentration of NaCl, $[\mathrm{NaCl}]_0 \times 10^5$ mol/dm^3	Initial concentration of HCl, $[\mathrm{HCl}]_0 \times 10^5$ mol/dm^3	Temperature (°C)	Equilibrium concentration of mercury, $C_M \times 10^5$ mol/dm^3	Equilibrium concentration of H^+ ion, $[H^+] \times 10^5$ mol/dm^3	Equilibrium concentration of Cl^- ion, $[Cl^-] \times 10^5$ mol/dm^3
1.23–1.36	0	0	25	0.064–0.220	6.08–37.3	1.02–1.20
9.30–10.4	0	0	25	0.536–2.25	2.93–9.29	8.06–8.76
42.2–50.6	0	0	25	11.0–20.2	0.437–5.35	30.4–35.4
10.5	500	0	25	8.00	10.6	500
31.0	0	500	25	24.6	132	500
47.4–50.2	500	0	25	27.5–39.1	1.10–63.1	500
239	500	0	25	155–171	14.3–14.7	500
49.2	0	0	15	20.1	6.25	29.1
50.6	0	0	30	23.4	5.03	27.1

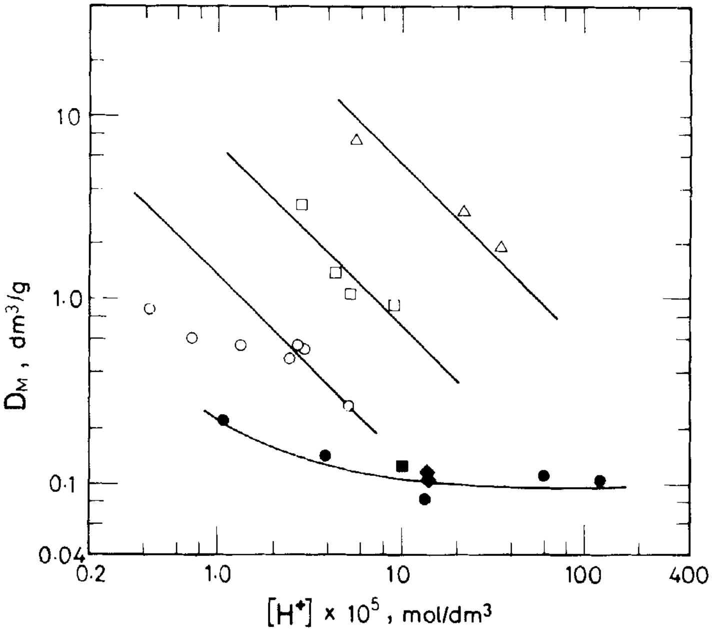

FIG. 1. Distribution ratio of mercury between onion skin and aqueous $HgCl_2$ solutions at 25°C: (△) $C_{M0} = (1.23\text{–}1.36) \times 10^{-5}$ mol/dm³, $[Cl^-] = (1.02\text{–}1.20) \times 10^{-5}$ mol/dm³; (□) $C_{M0} = (9.30\text{–}10.4) \times 10^{-5}$ mol/dm³, $[Cl^-] = (8.06\text{–}8.76) \times 10^{-5}$ mol/dm³; (○) $C_{M0} = (42.2\text{–}50.6) \times 10^{-5}$ mol/dm³, $[Cl^-] = (30.4\text{–}35.4) \times 10^{-5}$ mol/dm³; (■) $C_{M0} = 10.5 \times 10^{-5}$ mol/dm³, $[Cl^-] = 500 \times 10^{-5}$ mol/dm³; (●) $C_{M0} = (31.0\text{–}50.2) \times 10^{-5}$ mol/dm³, $[Cl^-] = 500 \times 10^{-5}$ mol/dm³; (♦) $C_{M0} = 239 \times 10^{-5}$ mol/dm³, $[Cl^-] = 500 \times 10^{-5}$ mol/dm³.

Three data points corresponding to the lower $[H^+]$ region for the solution where $[Cl^-] = 3.04 \times 10^{-4}$ to 3.54×10^{-4} mol/dm³, shown in Fig. 1, are more weakly dependent on the $[H^+]$ value. This may be attributed to the fact that the condition of constant $[\overline{HR}]$ is not met, owing to the higher content of mercury in the solid phase compared to the other data points.

From the experimental results described above, it is evident that the first term on the right-hand side of Eq. (12) is negligible compared to the other terms. Thus, the equilibrium concentration $[\overline{HR}]$ of functional groups in the solid phase can be expressed as

$$[\overline{HR}] = Q_{HR} - [\overline{HgClR}] \tag{17}$$

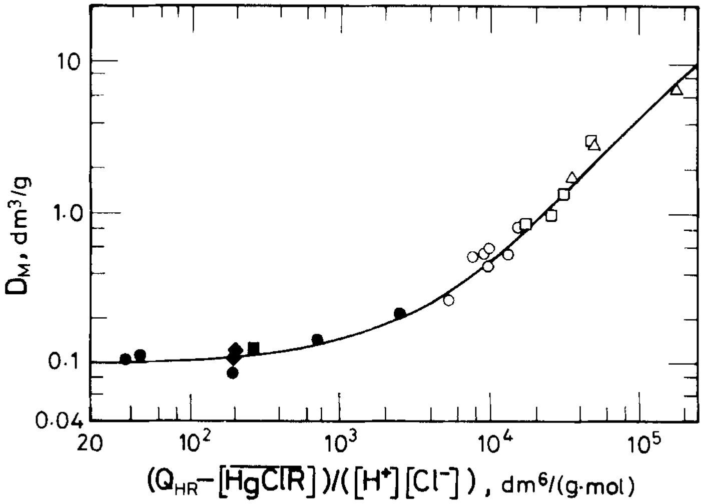

FIG. 2. Correlation of observed distribution ratio at 25°C by Eq. (19): the keys are as in Fig. 1.

where Q_{HR} is the initial concentration of functional groups in the solid phase, i.e., the mercury-binding capacity. Consequently, the measured values of the distribution ratios D_M might be expected to be correlated by

$$D_M = \left(\frac{K_{HgCl}\beta_1}{\beta_2}\right)\frac{(Q_{HR} - [\overline{HgClR}])}{[H^+][Cl^-]} + K_{HgCl_2} \tag{18}$$

The three constants in this expression, $K_{HgCl}\beta_1/\beta_2$, K_{HgCl_2}, and Q_{HR}, were determined by fitting Eq. (18) to all the experimental data obtained at 25°C. In these procedures the $[\overline{HgClR}]$ values for the case where $[Cl^-]$ is below 3.54×10^{-4} mol/dm^3 were assumed to be equal to the total mercury concentration $\overline{C_M}$ in the solid phase, since $[\overline{HgR_2}]$ and $[\overline{HgCl_2}]$ have been found to be unsubstantial. On the other hand, $[\overline{HgClR}]$ values for the case where $[Cl^-] = 5 \times 10^{-3}$ mol/dm^3 were assumed to be negligible compared with the Q_{HR} value, because the ion-exchange reaction of $HgCl^+$ is insignificant. The values of the three constants in Eq. (18) which gave minimum average deviation between the experimental and calculated results were $K_{HgCl}\beta_1/\beta_2 = 4.15 \times 10^{-5}$ mol/dm^3, corresponding to $K_{HgCl} = 100$, $K_{HgCl_2} = 0.101$ dm^3/g, and $Q_{HR} = 1.46 \times 10^{-4}$ mol/g.

The experimental data given in Fig. 1 are also shown in Fig. 2, where the distribution ratio D_M is plotted against the concentration ratio

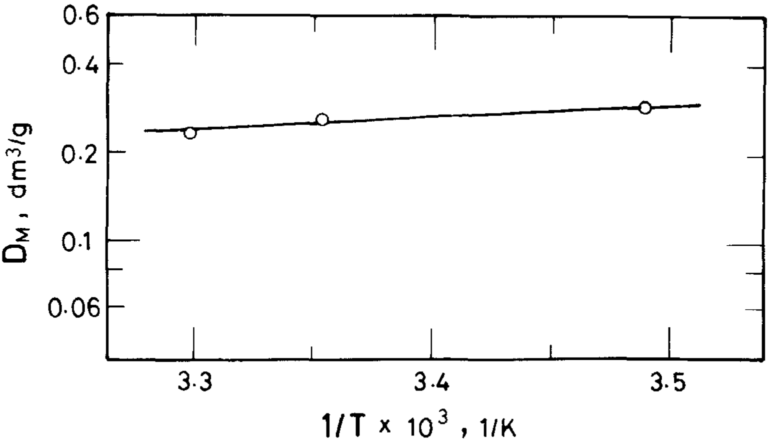

FIG. 3. Effect of temperature on the distribution ratio: $C_{M0} = (48.9\text{–}50.6) \times 10^{-5}$ mol/dm^3, $[Cl^-]_0 = 0$.

$(Q_{HR} - [\overline{HgClR}])/([H^+][Cl^-])$ on logarithmic coordinates. The solid line represents the established correlation of the distribution ratio at 25°C:

$$D_M = 4.15 \times 10^{-5} \frac{(1.46 \times 10^{-4} - [\overline{HgClR}])}{[H^+][Cl^-]} + 0.101 \qquad (19)$$

It can be seen that the measured distribution ratios are highly correlated with Eq. (19), regardless of the composition of the solution. At higher values of $(Q_{HR} - [\overline{HgClR}])/([H^+][Cl^-])$, the slope of the curve is equal to unity, while at lower values the curve approaches the horizontal asymptote, as expected. This agreement justifies the validity of the assumed distribution equilibrium for the present system.

The effect of temperature on the distribution ratio D_M is shown in Fig. 3, where the D_M values obtained by varying the temperature from 15 to 30°C at an almost constant value of the initial $HgCl_2$ concentration are plotted on semilogarithmic coordinates against the reciprocal of the thermodynamic temperature T. It can be seen that the distribution ratio is a weak function of temperature.

As stated above, the separation of mercury from aqueous $HgCl_2$ solutions by natural and synthetic polymers was experimentally studied by Friedman and Masri (*3*) and Masri et al. (*4*). Table 2 lists their representative experimental results, together with our data. It is difficult to discuss the comparative merits of these natural polymers as mercury

TABLE 2
Summary of Data on Removal of Mercury from $HgCl_2$ Solutions by Various Polymers

Investigator	Material	Temperature (°C)	Initial concentration of mercury (mg/dm^3(ppm))	Initial concentration of HCl (mol/dm^3)	Amount of mercury removed from solution (mg/g)	Distribution ratio D_M (dm^3/g)
Friedman and Masri (*3*)	Wool	25	1,000	0	40 (0.050)[a]	0.200
	Wool	25	40,000	0	925	0.043
	Wool	21	600	0.01	33 (0.100)[a]	0.122
	Ion-exchange resin	21	600	0.01	28	0.0875
	Chelating resin	21	600	0.01	13	0.0227
Masri et al. (*4*)	Activated sludge	—	40,000	0	460 (0.025)[a]	0.0209
	Bark, black oak	—	40,000	0	400	0.0167
	Bark, redwood	—	40,000	0	250	0.00833
	Senna leaves	—	40,000	0	250	0.00833
	Bark, douglas fir	—	40,000	0	100	0.00278
	Orange peel	—	20,000	0	275	0.0306
	Dry pine needles	—	20,000	0	175	0.0135
	Dry redwood leaves	—	20,000	0	175	0.0135
Present work	Onion skin	25	98	0	7.1 (0.100)[a]	0.262
	Onion skin	25	2.5	0	0.94 (0.400)[a]	7.30
	Onion skin	25	600	0.01	30[b] (0.100)[a]	0.101[b]

[a] Parenthetical values are the mix ratio of solution to material (dm^3/g).
[b] Predicted value.

collectors in the concentration region of interest for wastewater treatment because the previous work was performed at high mercury concentrations (ranging from 10^3 to 10^4 ppm). However, the present distribution ratio at an initial concentration of 600 ppm $HgCl_2$ and 0.01 mol/dm^3 HCl, which was predicted from Eq. (19), may be compared favorably with that of the synthetic resins.

CONCLUSION

The distribution ratios of mercury between onion skin and aqueous $HgCl_2$ solutions were measured by the batchwise method. The experimental results at 25°C were analyzed by using the theory based on the law of mass action. It was concluded that the equilibrium distribution of mercury between the solid and liquid phases is due to Ion-Exchange Reaction (6) of the cationic complex $HgCl^+$, and adsorption of the neutral complex $HgCl_2$ represented by Eq. (9). The equilibrium constants of the ion-exchange and adsorption processes at 25°C and the mercury-binding capacity of onion skins were evaluated as $K_{HgCl} = 100$, $K_{HgCl_2} = 0.101$ dm^3/g, and $Q_{HR} = 1.46 \times 10^{-4}$ mol/g. Further, it was found that the distribution ratio of mercury is comparatively insensitive to temperature.

SYMBOLS

C_M	total concentration of mercury in the liquid phase (mol/dm^3)
$\overline{C_M}$	total concentration of mercury in the solid phase (mol/g)
D_M	distribution ratio of mercury (dm^3/g)
K_{Hg}	equilibrium constant defined by Eq. (7) (g/dm^3)
K_{HgCl}	equilibrium constant defined by Eq. (8) (dimensionless)
K_{HgCl_2}	equilibrium constant defined by Eq. (10) (dm^3/g)
Q_{HR}	initial concentration of functional groups in the solid phase (mol/g)
T	thermodynamic temperature of the liquid phase (°K)
$[\ ]$	concentration in the liquid phase (mol/dm^3)
$\overline{[\ ]}$	concentration in the solid phase (mol/g)

Greek

β_i	stability constant defined by Eq. (2) (dm^3/mol)^i (i = 1–4)

Subscript

0	initial concentration

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