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Separation and Recovery of Iodine from Aqueous Solution by Permeation and Chemical Desorption (PCD) Using a Silicone Rubber Membrane

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ABSTRACT

New technologies for iodine separation and recovery are required to decrease environmental pollution and improve iodine production. Separation and recovery of iodine (I_2) in aqueous solution was achieved using permeation and chemical desorption (PCD) with a silicone rubber membrane (SRM). The SRM separated an aqueous feed solution from an alkaline or reducing recovery solution such as a mixture of sodium hydrate and sodium sulfate. The I_2 crossed the membrane from the aqueous feed solution into the recovery solution, where it was converted into iodide (I^-). Iodide in the recovery solution did not return to the feed solution across the SRM. An acidic feed solution promoted a high recovery of iodine. The permeation process followed first-order kinetics, allowing the overall mass-transfer coefficient and parameters related to permeation of I_2 through the SRM to be determined. Permeability of I_2 increased with temperature, and the apparent activation energy (E_a) for penetration of I_2 through the SRM was determined. The value of E_a for I_2 was of the same order of magnitude as those for phenols and anilines. The large membrane/aqueous distribution coefficient for I_2 indicated that I_2 had a high affinity toward the SRM. These results indicate that the PCD method is effective and powerful for separation and recovery of iodine from aqueous solutions.

Keywords: Membrane Separation; Permeation and Chemical Desorption (PCD) Method; Diffusion Coefficient; Membrane/Aqueous Distribution Coefficient; Polydimethylsiloxane

1. Introduction

Iodine is used in many applications, such as X-ray contrast media [1] and disinfectants [2], because of its high reactivity. Potassium iodide, which is essential to growth, is often added to table salt and feed to prevent iodine deficiency. Iodine and iodine compounds are also used as photo-sensitizers, catalysts, stabilizers, polarizing films on liquid crystal displays, and reaction intermediates [3].

Iodine in brine is usually produced through a blowing-out process [3-5]. The iodine concentration in brine is 60 - 350 mg·L^{-1} (0.2 - 1.3 mM) [6], but the recovery of iodine from brine requires large-scale equipment. The development of new small-scale, energy-saving processes for iodine separation and recovery need to be developed to decrease environmental pollution and improve iodine production.

The membrane separation process is widely used in industry because it offers several advantages, such as low energy consumption, low space use, and simple process design, compared to other processes [7,8]. The permeation and chemical desorption (PCD) method has been proposed as an alternative method for iodine recovery [9,10]. A target substance can be separated and recovered from an aqueous solution by altering the characteristics of the substance from high affinity toward the membrane to poor affinity by a chemical reaction, such as neutralization or oxidation-reduction. A previous study [11] explored the separation and recovery of 4-substituted phenol and aniline derivatives from aqueous solutions using PCD with a silicone rubber membrane (SRM). Phenols or anilines in aqueous solution were recovered successfully into aqueous NaOH or HCl solutions, respectively. A comparison between PCD and pervaporation (PV) was performed, which revealed that the removal rate of phenols from aqueous solution by PCD was significantly greater than by PV, and showed that PCD is effective for separation and recovery of low-volatility compounds [12,13]. Ferreira *et al.* proposed a recovery system for

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phenols and anilines using an SRM as a membrane aromatic recovery system (MARS) and reported success of a pilot-scale plant [14,15].

Iodine is attracted to the hydrophobic silicone membrane because of its high solubility in organic solvents, offering the ability to separate it by PCD. Thus, a project was undertaken to recover iodine and its derivatives that had been released into the environment and to improve iodine production by PCD using an SRM [9,10]. In this study, we tried to apply the PCD method for separation of iodine from water and obtained the recovery conditions and parameters for permeation of I_2 to the SRM. We have showed that the PCD method was effective and powerful for separation and recovery of iodine from aqueous solutions and the possibility of the PCD method to simplify the production of iodine and reduce energy consumption and space needs in this paper.

2. Materials and Methods

2.1. Permeation Experiment

The apparatus used for PCD was the same as described previously [11]. The SRM (As One Co., Ltd., Osaka, Japan) was inserted between two glass cells (capacity: 250 mL) and was fixed by flanges. The thickness of the SRM varied from 0.05 to 0.3 mm, with an effective area of 1.5×10^{-3} m^2. The SRM was made of polydimethylsiloxane and fumed silica, which was confirmed using attenuated total reflectance Fourier-transform infrared spectroscopy (data not shown). An iodine (Kanto Kagaku Co., Ltd., Tokyo, Japan) solution was prepared at a concentration of 0.8 mmol-I_2 L^{-1} in distilled water. The pH of the iodine solution was adjusted using HCl and NaOH solutions. The iodine solution (250 mL) was added into the feed side cell. The composition of the solutions (250 mL) assayed on the recovery side is shown in **Table 1**. The two solutions were agitated using a magnetic stirrer. Samples (1 mL) were withdrawn from both cells at regular intervals, and the iodine and iodide concentrations on each side solution were determined as described below. The experimental temperature (15°C - 45°C) was controlled using a water bath.

2.2. Determination of Iodine or Iodide Concentration

High performance liquid chromatography (HPLC) using a UV detector measured the concentration of iodine. Volumes of 0.1 mL sodium sulfite solution (0.5 mol·L^{-1}) and 0.5 mL of acetic acid buffer solution (0.2 mol·L^{-1}) were added to 1 mL of the sample solution. This mixture was analyzed to determine iodide concentration under the conditions shown in **Table 2**. All reagents were analytical grade purchased from Kanto Kagaku Co., Ltd. (Tokyo, Japan).

2.3. Measurement of Membrane Distribution Coefficient of I_2

An SRM (20×20 mm) with a thickness of 0.05 mm was immersed for 24 h in a bottle containing 10 mL of fresh iodine solution at 0.80 or 1.6 mmol-I_2 L^{-1} at pH4.0, and the equilibrium iodine concentration (C_{E1}) was determined by using the HPLC as described above. The SRM was removed from the solution, drained, and immersed in 20 mmol·L^{-1} NaOH. After 24 h, the concentration of iodide released from the SRM in the NaOH solution (C_{E2}) was determined. The membrane distribution coefficient, m_c, was estimated based on the values of C_{E1} and C_{E2}. The equilibrium experiments were performed at 25°C.

Each experiment was conducted 3 times and average value was shown as a result; the SE of the average value remained below 15%.

3. Results and Discussion

3.1. pH Dependency of Iodine Permeation through SRM

The permeation of iodine through SRM was investigated using NaOH solution in the recovery at different pH values of the iodine solution in the feed cell. **Figure 1** shows the permeation ratio, R_p, at different feed cell pH values after 5 h. The SRM thickness was 0.05 mm, and temperature was 25°C. R_P was obtained using Equation (1):

Table 1. Overall mass transfer coefficients (K_{OL}) of iodine for several recovery solutions.

Reagents	Concentration (mmol·l^{-1})		$K_{OL} \times 10^5$ (m·s^{-1})
	Na$_2$CO$_3$	Na$_2$SO$_3$	
Na$_2$CO$_3$ + Na$_2$SO$_4$*	1	1	0.95
	10	10	1.2
	50	50	1.3
	100	100	1.2

*Na$_2$CO$_3$ and Na$_2$SO$_3$ are equimolar.

Table 2. HPLC measurement conditions.

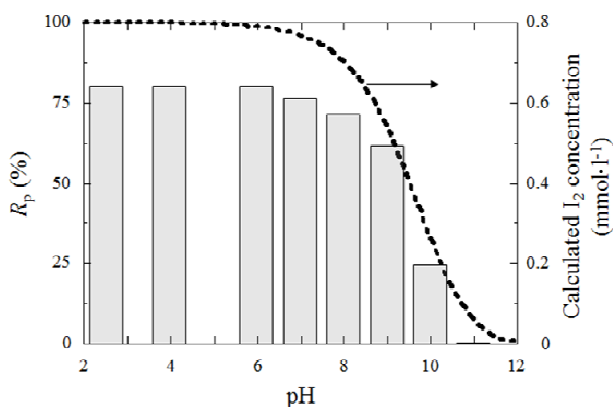| Model	LC-6A (Shimadzu Co, Kyoto, Japan)
Detector	UV spectrophotometric detector SPD-6A (Shimidzu Co.)
	220 nm
Column	Luna 5 m C18, 150 mm × 4.6 mm (Phenomenex, CA, USA)
Column temperature	40°C
Mobile phase	CH$_3$CN/H$_2$O-20 mmol·l^{-1} tetrabutylammonium phosphate = 25/75
	(Kanto Kagaku Co., LTD., Tokyo, Japan)
Flow rate	1.0 ml·min^{-1}
Injection volume	10 ml

Figure 1. The percentage of iodine permeated through theSRM after 5 h and theoretically calculated effective I_2 concentration at different pH values. (Recovery cell: 10 mmol·L^{-1} NaOH; membrane thickness: 0.05 mm; temperature: 25˚C).

$$R_p(\%) = \frac{C_0 - C}{C_0} \times 100 \qquad (1)$$

where C_0 and C are the initial iodine concentration and the concentration at time t, respectively, in the feed cell. The value of R_p remained constant up to pH 6, and 80% of the iodine migrated to the recovery side after 5 h. The value of R_p decreased with an increase in pH, and no permeation was observed at pH 11.

Iodine hydrolyzes in aqueous solution as described by Equation (2), and the hydrolysis constant, K_h, is expressed by Equation (3) [16,17]:

$$I_2 + H_2O \rightleftarrows HIO + H^+ + I^-, \qquad (2)$$

$$K_h = \frac{[HIO][H^+][I^-]}{[I_2]} = 4.6 \times 10^{-13} \left(mol \cdot l^{-1}\right)^2 (25˚C) \quad (3)$$

Equation (3) demonstrates that pH strongly influences the chemical equilibrium of I_2 and I^- in aqueous solution. The effective I_2 concentration calculated based on Equations (2) and (3) are shown as a dotted line in **Figure 1**. Here, the contribution of dismutation reaction of HIO in water ($2HIO \rightleftarrows 2HI + O_2$ and $3HIO \rightleftarrows HIO_3 + 2HI$) was neglected to simplify the analysis. **Figure 1** shows that the variation in R_p correlated with that of the calculated effective I_2 concentration. These results indicate that I_2 can be adsorbed in, dissolve in and penetrate through the SRM, but I^- cannot (**Figure 2**). Iodide ion with the negative charge is difficult to be adsorbed in SRM, because the SRM is a hydrophobic membrane. Thus, placing iodine in an acidic solution in the feed cell, with an alkaline solution in the recovery cell, is efficient for recovery of iodine. This system also can concentrate iodine on the recovery side, because I^- in the recovery cell cannot return to the feed side through the SRM.

3.2. Absorbent Solution in Recovery Cell

The type of absorbent solution in the recovery cell for I_2 was investigated. **Figure 3** shows typical changes in I_2 concentration in the feed solution. Iodine concentration in the feed cell decreased with time. Iodine and iodide concentration measurements on both sides showed that a mass balance was established in this system. Stripping of I_2 and absorption by the alkaline recovery solution occur simultaneously in this system. Thus, I_2 concentration on the recovery side can be considered to be zero ($I_2 \rightarrow 2I^-$), and the I_2 concentration on the feed side (C) can be represented by [18,19]:

$$\ln \frac{C}{C_0} = -\frac{A}{V} K_{OL} t \qquad (4)$$

where A and V are the effective membrane area and the liquid volume in the feed cell, respectively. K_{OL} is the overall mass transfer coefficient.

As shown in **Figure 3**, $\ln(C/C_0)$ was proportional to t. A similar tendency was observed under other conditions,
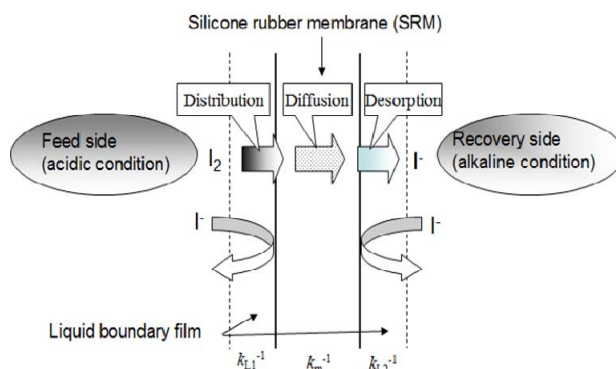

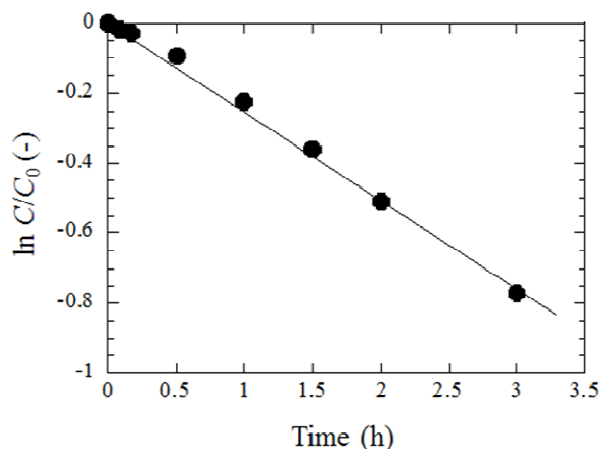**Figure 2. Model of permeation of I_2 through the SRM for the PDC method.**

Figure 3. Typical permeation of iodine through the SRM. (Feed cells: 0.8 mmol I_2 L^{-1} solution at pH 4.0; recovery cell: 10 mmol·L^{-1} Na$_2$CO$_3$ + Na$_2$SO$_3$; membrane thickness: 0.05 mm; temperature: 25˚C).

allowing determination of K_{OL} from the slope of the line (**Table 1**). A solution of Na_2SO_3 + Na_2CO_3 was used in the blowing-out process for recovery of iodine [3]. Values of K_{OL} for the Na_2SO_3 + Na_2CO_3 solution at concentrations greater than 10 mmol·L^{-1} became constant (1.2 - 1.3 m·s^{-1}); this constant value was similar to that for 10 mmol·L^{-1} NaOH. Thus, 10 mmol·L^{-1} Na_2SO_3 + Na_2CO_3 was used as the absorbent solution in the recovery cell, because its pH (11.1), was lower than that of 10 mmol·L^{-1} NaOH (pH 12). The PCD method does not need a phase change such as liquid to vapour in the blowing-out process and pressure operation basically. It can be said to be an excellent technique in energy saving.

3.3. Permeation Characteristics of I_2 into the SRM

The resistances-in-series model has been used to describe the transport of molecules through a membrane with liquid films on both sides (**Figure 2**). The term $1/K_{OL}$ represents overall mass transfer resistance and can be written as:

$$\frac{1}{K_{OL}} = \frac{1}{k_{L1}} + \frac{1}{m_c k_m} + \frac{1}{k_{L2}} \qquad (5)$$

where k_{L1} and k_{L2} are the liquid boundary film mass transfer coefficients of the feed and recovery sides, respectively, and k_m and m_c are the mass transfer coefficient in the membrane and membrane/aqueous distribution coefficient, respectively. When chemical absorption occurs as described, the mass transfer resistance of the recovery side (k_{L2}^{-1}) is negligible [18,19], and k_m relates to diffusivity, D, and film membrane thickness, d, as shown by Equation (6):

$$k_m = D/d \qquad (6)$$

Equation (6) can be re-written as:

$$\frac{1}{K_{OL}} = \frac{1}{k_{L1}} + \frac{d}{m_c D} = \frac{1}{k_{L1}} + \frac{d}{P} \qquad (7)$$

where $P = m_c D$. Equation (7) describes a linear relation between d and $1/K_{OL}$. The value of P can be obtained from the slope of the line and the D values can be estimated using the m_c values obtained in Section 2.3.

Figure 4 shows changes in the K_{OL} of I_2 with SRM thickness (d). The value for $1/K_{OL}$ increased linearly with d. The value of D was estimated by inserting experimentally determined m_c values into the expression for P. The value of log m_c was 3.7 and did not change with I_2 concentration. **Table 3** summarizes the values of octanol/water partition coefficient (P_{OW}), m_c, and D obtained for I_2 in this study, along with the values for phenols and anilines obtained previously [12]. Iodine (I_2) has larger values for m_c than for P_{OW}, indicating a high affinity of I_2 toward the SRM, which indicates the effectiveness of the SRM for the separation and recovery of iodine.

The D value of I_2 was smaller than those of phenols and anilines, despite the smaller molar volume of I_2. Watanabe and Miyauchi measured the solubility and diffusivity of I_2 in polydimethylsiloxane oils and organic solvents, and reported that the diffusivity of I_2 in polydimethylsiloxane did not change with the polymer chain length. They attributed this phenomenon to the
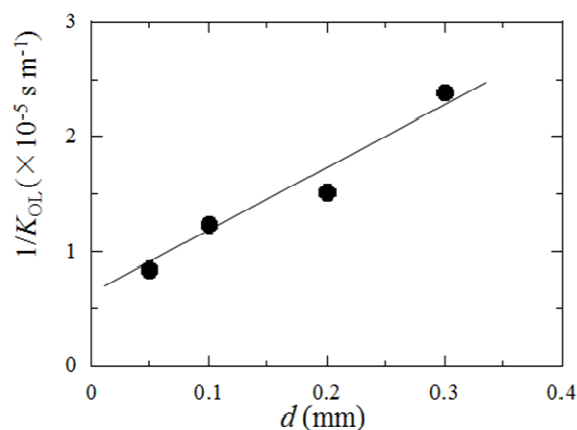

Figure 4. Effect of SRM thickness on the overall mass transfer coefficient (K_{OL}) of iodine. (Feed cells: 0.8 mmol I_2 L^{-1} at pH 4.0; recovery cell: 10 mmol·L^{-1} Na_2CO_3 + Na_2SO_3, temperature: 25°C).

Table 3. Parameters of compounds tested for affinity toward a silicone rubber membrane.

Chemicals	Molecular weight (g·mol^{-1})	Molar volume (ml·mol^{-1})	log P_{OW} (-)	log m_c (-)	D ×10^{10} (m^2·s^{-1})	E_a (kJ·mol^{-1})	Reference
I_2*	253.8	51.4	2.49[a]	3.7	0.11	41.9	
Phenol	94.1	88.6	1.5	–0.30	1.46	17[b]	
4-Cresol	108.1	104.5	1.9	0.36	2.43		[12]
4-Butylphenol	150.2	166.5	3.8	2.02	0.12		
Aniline	93.1	90.7	0.94[c]	0.56[d]	1.2[d]	18.1	
4-Chloroaniline	127.6	89.2	1.8[e]			15.6	[22]
Dimethylamine	45.1	64.4	2.3[f]			30.5	

*This work; [a]International Chemical Safety Cards (ICSCs) No. 0167; [b][22]; [c]International Chemical Safety Cards (ICSCs) No. 0011; [d][12]; [e]International Chemical Safety Cards (ICSCs) No. 0026; [f]International Chemical Safety Cards (ICSCs) No. 0877.
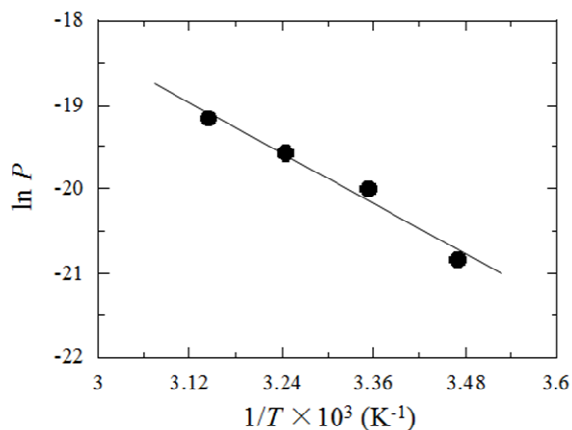
Figure 5. Effect of temperature on permeability (*P*) of I$_2$ through the SRM. (Feed cells: 0.8 mmol I$_2$ L^{-1} solution at pH 4.0; recovery cell: 10 mmol·L^{-1} Na$_2$CO$_3$ + Na$_2$SO$_3$; membrane thickness: 0.2 mm).

20% - 30% larger size of the siloxane unit [-OSi(Me)$_2$-] compared to I$_2$, which did not change with increasing polymer chain length [20]. Therefore, the small *D* value of I$_2$ regardless small molar weight will depend on the siloxane unit size.

Figure 5 shows variation in *P* values with temperature (SRM thickness: 0.2 mm). The value of *P* increased with temperature, due to temperature-enhanced mobility of the polymer chains that allow the penetrant to diffuse more easily. According to molecular models for rubbery polymers above their glass transition temperatures, the temperature dependence of *P* of the penetrant through a polymer follows the van't Hoff-Arrhenius relation [21]:

$$P = P_0 \exp\left(-\frac{E_a}{RT}\right) \qquad (8)$$

where P_0 is a pre-exponential factor, *R* is the molar gas constant, *T* is the absolute temperature, and E_a is the apparent activation energy of penetration required for sorption of the penetrant into polymers. Higher E_a values indicate that additional energy is needed for the penetrant to permeate through the polymer.

The value of E_a for I$_2$ to the SRM was obtained from Equation (8) (**Table 3**). Compared with the E_a values of phenols [22] and aniline [23], the E_a of I$_2$ was the same order of magnitude, suggesting that thermal activity also is effective for the separation and recovery of I$_2$ using PCD.

Although this technique is applicable to various compounds except iodine [9-13], the recovery of the substance, which is ionized in the recovery side, is theoretically difficult. It is necessary to also challenge separation of such a substance and to extend the scope of application.

4. Conclusion

In this study, permeation and chemical desorption (PCD)

using a silicone rubber membrane (SRM) was effective and powerful for the separation and recovery of iodine. The I$_2$ had high affinity toward the SRM. The I$_2$ in aqueous solution was recovered into an alkaline or Na$_2$SO$_3$ + Na$_2$CO$_3$ recovery solution by placing iodine solution and alkali or Na$_2$SO$_3$ + Na$_2$CO$_3$ solution on opposite sides of an SRM. Higher recovery of I$_2$ was obtained by controlling the pH of the feed solution. This PCD method can simplify the production of iodine and reduce energy consumption and space needs. Iodine can be obtained as a solid after neutralization from the recovery solution. An initial pilot study for recovery of iodine and iodides has been initiated using a silicone hollow-fiber membrane manufactured from the same silicone rubber used in this work. In a continuous experiment using the membrane, no change was found in the recovery rate of iodine over one month (data not shown), indicating adequate lifespan of the silicone membrane. We propose this method as an alternative to the blowing-out process for separation of iodine.

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