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Effect of Rheological Properties on Mass Transfer of Cr(VI) Through a Supported Liquid Membrane with Non-Newtonian Liquid

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ABSTRACT

Cr(VI) was transported using a non-Newtonian polyisobutylene liquid supported by microporous polytetrafluoethylene polymer. The effects of viscosity and viscoelasticity of toluene solution of polybutene and polyisobutylene on the membrane side-mass transfer coefficient of Cr(VI) have been investigated. The elasticity, considered in the form of

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Weisenberg number, was used to present unified correlations for mass transfer coefficients in Newtonian as well as non-Newtonian liquid.

Key Words: Hexavalent chromium; Viscoelasticity; Supported liquid membrane; Mass transfer coefficient.

INTRODUCTION

Hexavalent chromium, Cr(VI), is one metal that has received considerable attention. It has been used extensively in such industrial applications as electroplating, electrofinishing, steelmaking, leather tanning, and corrosion inhibition, and it has long been recognized as a toxic substance due to its strong oxidizing potential and the ease with which it can pass through biological membranes.[1] The removal of Cr(VI) from industrial wastewater is of great interest mostly due to the high toxicity of chromium(VI) compounds. During recent years the application of different solvent extraction technologies to chromium removal and concentration has been widely studied; conventional liquid–liquid extraction,[2] emulsion liquid membranes,[3] supported liquid membranes (SLMs),[4] and non-dispersive extraction[5] are some of the new alternatives reported in the literature.

The dependence of shear stress on shear rate of a fluid in a hydrodynamic system is different according to the type of the fluid, i.e., Newtonian or non-Newtonian fluid, and the mass transfer coefficient (k_L) of a solute in one phase is in inverse proportion to the viscosity of its phase due to the inverse proportion of viscosity to diffusivity. The rheological properties such as Deborah number (De), defined as the ratio of the characteristic material time to the characteristic process time, or Weissenberg number (Wi), defined as the ratio of the first normal stress difference to the shear stress, is taken into account to correlate the mass transfer coefficient in non-Newtonian fluid with that in Newtonian fluid. Mere use of the apparent viscosity of non-Newtonian fluids was not sufficient to obtain a unified correlation for k_L values in Newtonian and non-Newtonian fluids. Due to the complexities of mass transfer in non-Newtonian media, the correlations available in these studies were limited to just a few kinds of non-Newtonian fluids, such as Carbopol, carboxymethylcellulose (CMC), polyacrylate (PA), polyethylene-oxide (PEO), polyacrylamide (PAA), and polyisobutylene (PIB) solutions. Unified correlations have been proposed for volumetric mass transfer coefficient (k_La) in Newtonian as well as non-Newtonian solutions by introducing the dimensionless term such as $(1 + n_1\, De^{n_2})^{n_3}$, which is listed in Table 1. As shown in Table 1, figures in the dimensionless term are different from one another.

Suh et al.[11] have also proposed the dimensionless term such as $(1 + 0.18Wi^{0.93})^{-1}$ to fit the experimental k_La of O_2 absorption in a bubble

Table 1. Coefficients of dimensionless group of mass transfer.

Investigator	n_1	n_2	n_3	Polymer	Contactor
Yagi and Yoshida[6]	2	0.5	-0.67	CMC, PA	Agitated vessel
Ranade and Ulbrecht[7]	100	1	-0.67	CMC, PAA	Stirred tank
Nakanoh and Yoshida[8]	0.13	0.55	-1	CMC, PA	Bubble column
Park et al.[9]	100	1	-0.42	PB, PIB	Agitated vessel
Park et al.[10]	2461.3	1	-0.274	PB, PIB	Agitated vessel

column using CMC, PAA, and Xanthan as non-Newtonian liquids. Vlaev et al.[12] have used the apparent viscosity of the non-Newtonian liquid and proposed the dimensionless equation such as $Sh = 9.71 \times 10^{-7} Re^{2.47} (\mu/\mu_w)^{2.1}$ for the oxygen transfer in colloidal dispersions of corn starch in water using a stirred vessel.

Kawase et al.[13] presented a theoretical model for k_La in bubble columns using non-Newtonian fluids based on Higbie's penetration theory and Kolmogoroff's theory of isotropic turbulence. They used the flow behavior index, n, in the power law liquid of Ostwald de Waele to correlate k_La with the modified Schmidt and Reynolds number. There is little information about the effect of elastic properties on extraction of metal in the organic solvent-soluble non-Newtonian fluids through extraction of a solute in a liquid–liquid heterogeneous system.

The present work was intended to obtain a unified correlation for membrane-side mass transfer coefficient (k_m) of Cr(VI) in the organic non-Newtonian fluids supported by microporous polymer and to observe the effect of elasticity of non-Newtonian liquid on k_m. The particular system chosen for this study was extraction of Cr(VI) in toluene solutions of polybutene (PB) and PIB with varying rheological properties.

EXPERIMENTAL

Chemicals

Chemicals used in this study were reagent grade and used without further purification. The supporting membrane was hydrophobic microporous polytetrafluoroethylene membrane with nominal thickness of 1.75×10^{-4} m, porosity of 0.7, and tortuosity of 1.353.

The concentrations of Cr(VI) in the feed side and NaOH in the stripping side were $50\,\mathrm{g/m^3}$ and $1\,\mathrm{kmol/m^3}$, respectively. The pH value of the feed solution was fixed at 3.

The polymer additives used in this study were PB with the mean molecular weight of 750 (Aldrich, USA) and PIB with the mean molecular weight of 1,000,000 (Aldrich, USA). The toluene solutions of PB and PIB of various concentrations were used as Newtonian and non-Newtonian liquids, respectively. For the transport experiments of Cr(VI), the concentration of PB in the toluene solution was 1, 5, 10, 20, 30 wt% and that of PIB was 0.1, 0.2, 0.5, and 1.0 wt%.

Membrane-Side Mass Transfer Coefficient

Aqueous Cr(VI) solutions as the feed solution were prepared by dissolving $K_2Cr_2O_7$ (Junsei Chemical Company, Ltd., Japan) in deionized water, and the pH was adjusted with sulfuric acid. The impregnation of the carrier into pores of the supporting membrane was achieved by immersing the membrane in the organic extractant solution for 24 hr by the same procedure as reported earlier.[14] An aqueous NaOH solution was used as the stripping solution. Cr(VI) concentrations in the aqueous solution were measured with a Hewlett Packard UV-visible spectrophotometer (model 8452A, USA) using a colorimetric diphenyl-carbazide method.[15]

The experimental procedure was the same as those reported earlier.[4] The flat-plate membrane separator module was made of Pyrex glass with inside diameter of 0.1 m and height of 0.03 m. After setting the impregnated support at the center of the module, a 0.175-dm^3 portion of aqueous Cr(VI) solution was put into the lower part of the module and the same amount of aqueous NaOH solution into the upper part. The lower part was agitated by a magnetic bar and the upper part by an impeller connected to a motor. The concentration of Cr(VI) in the feed side was measured periodically by sampling a small amount (1×10^{-3} dm^3) from the lower part. The same volume of water was replaced after sampling. The supporting membrane was used as a new one for each experiment. Experiments were repeated three times, and the overall mass transfer coefficient obtained from the time dependence of Cr(VI) concentration was averaged. All experiments were carried out at 25°C.

When Cr(VI) is transported from an aqueous feed side to the stripping side of an aqueous NaOH solution through an organic phase of non-Newtonian liquid in the microporous polymeric membrane support at steady-state, the flux of Cr(VI) at each step may be expressed as follows:

$$\begin{aligned} N_A^o &= K_o(C_{Af} - C_{Af}^*) = k_f(C_{Af} - C_{Afi}) = k_m(\bar{C}_{Af} - \bar{C}_{As}) \\ &= k_s(C_{As}^* - C_{As}) \end{aligned} \tag{1}$$

where the overbar means the membrane side.

Using the partition coefficient of Cr(VI), H_A, and Eq. (1), the relationship of overall and respective mass transfer resistances is rearranged to be

$$\frac{1}{K_o} = \frac{1}{k_f} + \frac{1}{H_A k_m} + \frac{1}{k_s} \tag{2}$$

If the resistances of the feed and stripping sides are negligible as compared with that of the membrane-side, the membrane side mass transfer coefficient is obtained from the following equation:

$$k_m = \frac{K_o}{H_A} \tag{3}$$

If the total volume of the organic extractant within the micropores of the membrane is negligible as compared with the volume of the aqueous solution in each part, the change of Cr(VI) concentration with time in the feed side is expressed as follows:

$$-\frac{dC_{Af}}{dt} = K_o(C_{Af} - C^*_{Af}) \tag{4}$$

If Cr(VI) transported to the alkaline side of the membrane is completely consumed by an instantaneous reaction in the alkaline stripping solution, C^*_{Af} is equal to zero due to $C_{AS} = 0$. Then, integration of Eq. (4) leads to

$$\ln \frac{C_{Af}}{C_{Af^o}} = -K_o a t \tag{5}$$

The membrane-side mass transfer coefficient is obtained from Eq. (3) using the value of K_o obtained from the observed time dependence of C_{Af} in Eq. (5).

To find an experimental condition in which the feed-side mass transfer resistance is negligible, the time dependence of the concentration of Cr(VI) in feed side was measured at various agitation speeds, with a typical extractant of PIB of 1 wt% and PB of 30 wt%. The overall mass transfer coefficient, K_o, was obtained from the linear relationship between $\ln(C_{Af}/C_{Af^o})$ and time using Eq. (5) at the agitation speeds of 400, 500, and 700 rev/min, and its value was 2.963×10^{-7}, 2.974×10^{-7}, and 3.012×10^{-7} m/s, respectively, which may be taken as a constant value. Therefore, the membrane-side mass transfer coefficient, k_m, may be obtained from Eqs. (3) to (5), and the agitation speeds in the feed and stripping sides were fixed at 500 rev/min in all experiments mentioned below to study the elastic behavior of the non-Newtonian liquid.

To obtain k_m from Eq. (3), it is necessary to know the partition coefficient, H_A, which was measured by the same procedure as those reported earlier,[16] and its value was determined to be 0.0165.

Rheological Properties of Toluene Solution

The rheological properties of the toluene solutions of PB and PIB were measured by the parallel disk-type rheometer of the diameter of 0.05 m and the gap of 0.001 m (Ares, Rheometrics, USA).

Apparent Viscosity of Toluene Solution

The apparent viscosities of the toluene solutions of PB and PIB were measured using a Ubbelohde viscometer at 25°C.

RESULTS AND DISCUSSION

Rheological and Physicochemical Properties of the Liquid

We assume that a power-law model, which has been widely used for shear-dependent viscosity, can represent the non-Newtonian flow behavior of fluids:

$$\tau = K\gamma^n \tag{6}$$

$$\mu = K\gamma^{n-1} \tag{7}$$

$$N_1 = A\gamma^b \tag{8}$$

where n, K, b, and A are material parameters depending on temperature.

These parameters were obtained from the dependence of τ and N_1 on γ.

To observe the dependence of τ, and N_1 on γ, τ and N_1 of the toluene solution were measured as a function of γ by the rheometer.

Figure 1 shows typically the logarithmic plot of shear stress vs. the shear rate for toluene, the toluene solutions of PB of 30 wt%, and of PB of 30 wt% containing PIB of 1 wt%. The best-fit straight line was determined by the least-squares method with the plots in Fig. 1.

From the intercept and slope of the straight line, the values of K and n were evaluated. Also, Fig. 2 shows the logarithmic plot of primary normal stress difference vs. shear rate for the same solution as in Fig. 1.

As shown in Fig. 2, the plots for the toluene solution of 30 wt% PB and 1 wt% PIB are linear, whereas the values of the primary normal stress difference of toluene and toluene solution of 30 wt% PB are zero. From the intercept and slope of the straight line for the toluene solution of 30 wt% PB and 1 wt% PIB, the values of A and b were evaluated. The parameters K, n, A, and b for the toluene solution of various concentrations of PB and PIB are given in Table 2.

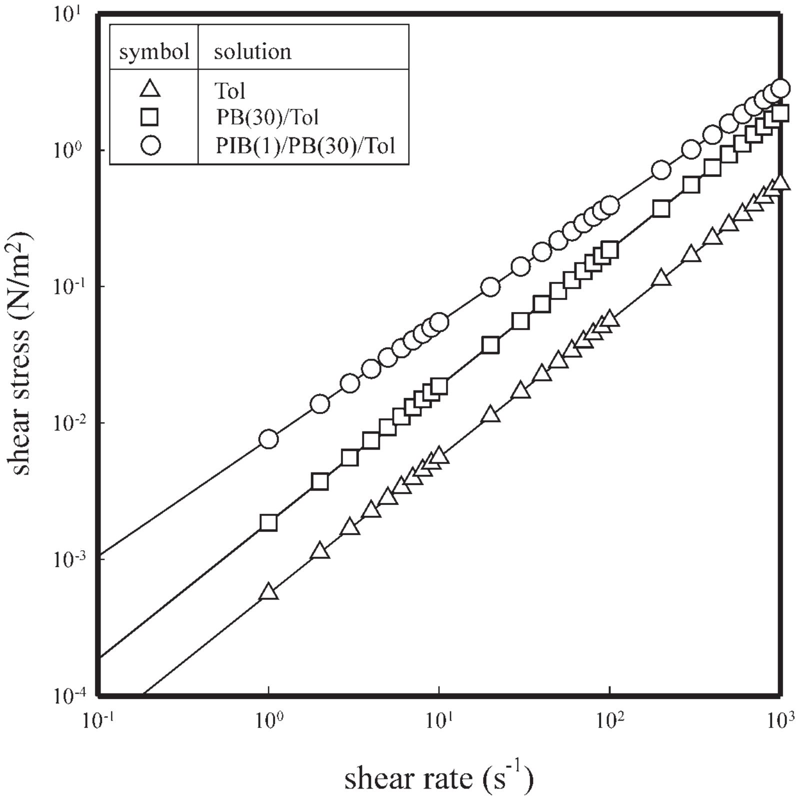

Figure 1. Shear stress of toluene solution as a function of shear rate.

As shown in Table 2, note that only the toluene solutions of PIB exhibit any measurable primary stress difference; therefore, they are considered viscoelastic.

Deborah number (*De*) or Weissenberg number (*Wi*) is used to relate the elastic properties of the non-Newtonian fluid with the process parameters such as agitation speed and velocity of fluid. Using Eqs. (6) and (8) and the definition of *De* ($=\lambda/t$), where λ is $N_1/(\mu\gamma^2)$ defined as the characteristic material time and *t* is the characteristic process time, and *Wi* ($=N_1/\tau$), *De* and *Wi* can be written, respectively, as follows:

$$De = \frac{A}{K}\gamma^{b-n-1}N \tag{9}$$

$$Wi = \frac{A}{K}\gamma^{b-n} \tag{10}$$

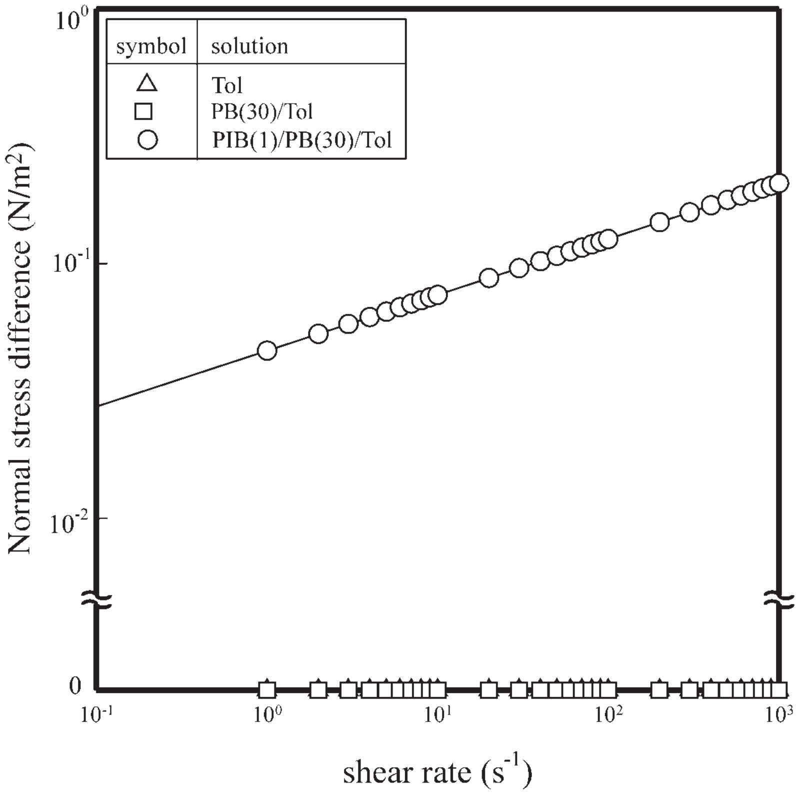

Figure 2. Normal stress difference of toluene solution as a function of shear rate.

If it is assumed that the non-Newtonian liquid existing in the pore of the polymer membrane is stagnant and independent of the agitation speed of the feed and stripping sides, *Wi* rather than *De* is used to correlate the elasticity of the non-Newtonian liquid to the behavior of the Newtonian liquid.

The shear rate in Eq. (10) is of importance in fixing the rheological or viscometric behavior of such a non-Newtonian liquid and it depends on the hydrodynamic characteristics of the fluid. Metzner and Otto[16] found that the shear rate could simply be related to the impeller speed by:

$$\gamma = \frac{4\pi N}{n} \tag{11}$$

If the shear rate is considered at the contact position between the feed side and the membrane side, the value of agitation speed is used as the agitation speed of the feed side.

Table 2. Rheological properties of PIB/PB/Tol solution.

PIB (wt%)	PB (wt%)	n	$K \times 10^4$ (N_s^n/m^2)	b	A (N_s^b/m^2)
0	0	1	5.62	0	0
	1	1	6.08	0	0
	5	1	6.75	0	0
	10	1	8.01	0	0
	20	1	11.79	0	0
	30	1	18.62	0	0
0.1	0	0.997	7.15	0.0006	0.0008
	1	0.994	7.93	0.0007	0.0009
	5	0.990	8.79	0.008	0.0017
	10	0.985	10.57	0.0008	0.0028
	20	0.981	15.35	0.0009	0.0034
	30	0.978	20.63	0.0010	0.0048
0.2	0	0.994	9.95	0.0056	0.0009
	1	0.991	10.93	0.0079	0.0009
	5	0.984	11.35	0.0115	0.0015
	10	0.982	12.18	0.0143	0.0038
	20	0.977	18.19	0.0168	0.0067
	30	0.970	27.93	0.0197	0.0095
0.5	0	0.991	15.38	0.0341	0.0009
	1	0.986	16.71	0.0429	0.0017
	5	0.983	18.61	0.0429	0.0037
	10	0.979	19.85	0.0512	0.0065
	20	0.964	26.26	0.0576	0.0124
	30	0.956	39.35	0.0608	0.0184
1.0	0	0.965	38.20	0.1534	0.0043
	1	0.951	39.14	0.1711	0.0105
	5	0.923	40.32	0.1820	0.0163
	10	0.905	48.67	0.2050	0.0259
	20	0.896	58.13	0.2111	0.0327
	30	0.857	76.08	0.2193	0.0455

To observe the elasticity of the non-Newtonian liquid, *Wi* is calculated using Eqs. (10) and (11) according to the change of concentration of PB and PIB, and given in Table 3 and plotted in Fig. 3.

As shown in Fig. 3, *Wi* increases with an increase in the concentration of PIB. The dependence of *Wi* on PIB concentration is reasonable in view of the elasticity of PIB.

The apparent viscosities of the toluene solution, which were measured according to the charge of concentration of PB and PIB, are given in Table 3.

Table 3. Mass transfer coefficient, viscosity, and Wi of PIB/PB/Tol solution.

PIB (wt%)	PB (wt%)	$K_o \times 10^7$ (m/s)	$k_m \times 10^5$ (m/s)	$\mu \times 10^4$ (Pa s)	*Wi*
0	0	3.84	2.327	5.62	0.0000
	1	3.77	2.285	6.08	0.0000
	5	3.63	2.200	6.75	0.0000
	10	3.50	2.121	8.01	0.0000
	20	3.35	2.030	11.79	0.0000
	30	2.89	1.752	18.62	0.0000
0.1	0	3.77	2.285	7.49	0.0108
	1	3.66	2.218	7.71	0.0111
	5	3.33	2.018	8.10	0.0192
	10	2.94	1.782	9.35	0.0268
	20	2.98	1.806	13.31	0.0228
	30	2.80	1.697	19.29	0.0242
0.2	0	3.75	2.273	9.21	0.0091
	1	3.35	2.030	9.24	0.0084
	5	3.31	2.006	9.26	0.0141
	10	3.21	1.945	11.01	0.0340
	20	3.07	1.861	15.68	0.0414
	30	3.02	1.830	23.87	0.0398
0.5	0	3.67	2.224	14.11	0.0068
	1	3.40	2.061	14.65	0.0125
	5	3.34	2.024	15.53	0.0254
	10	3.25	1.970	17.94	0.0429
	20	3.25	1.970	26.26	0.0685
	30	3.02	1.830	39.30	0.0699
1.0	0	3.69	2.236	28.00	0.0251
	1	3.14	1.903	28.52	0.0686
	5	3.19	1.933	29.67	0.1214
	10	3.10	1.879	35.83	0.1913
	20	3.16	1.915	53.51	0.2158
	30	2.97	1.800	83.41	0.2792

As shown in Table 3, the apparent viscosity increases with increasing concentration of PB and PIB.

Empirical Correlation

To obtain the value of membrane-side mass transfer coefficient, the time dependence of the concentration of Cr(VI) in the feed side was measured at

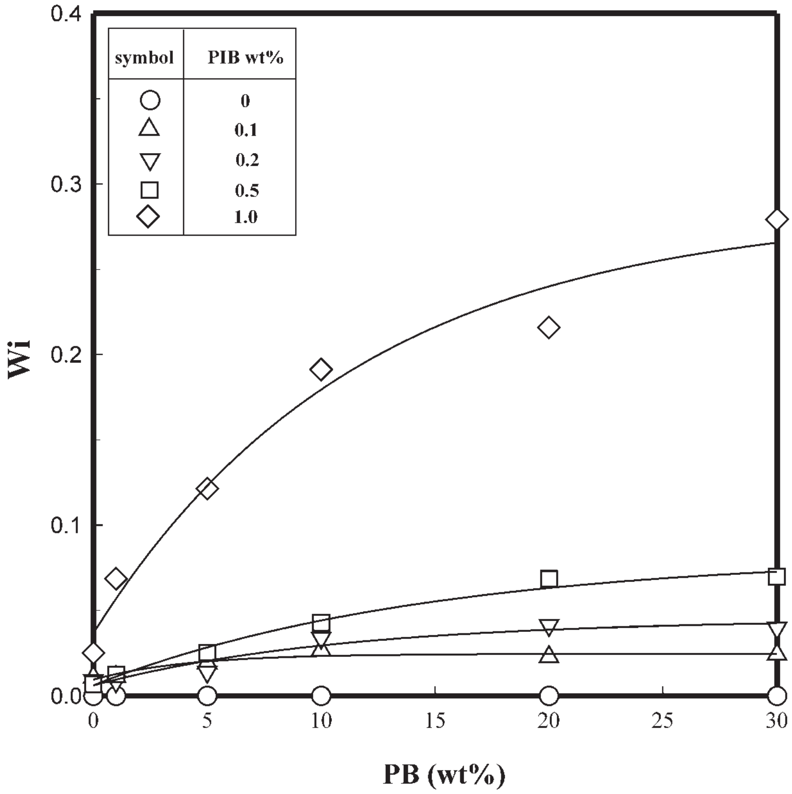

Figure 3. Effect of Weissenberg number on the concentrations of PB and PIB.

various concentrations of PB and PIB. The overall mass transfer coefficients obtained from the measured values are given in Table 3. As shown in Table 3, K_o and k_m decrease with increasing concentrations of PB and PIB.

To correlate the membrane-side mass transfer coefficient with the viscosity of the toluene solution, k_m was plotted against μ for the toluene solution of PIB of 0–1 wt%. Figure 4 shows logarithmic plots of k_m against μ, in which the full circles and the open circles represent the plots of toluene solution of PB (PB/Tol) and toluene solution of PIB and PB (PIB/PB/Tol), respectively.

As shown in Fig. 4, the plots of PB/Tol are linear, but the plots of PIB/PB/Tol deviate from the plots of the PB/Tol solution. The slope and intercept from the straight line of plots of full circles are obtained by the least-square method with correlation coefficient of 0.965, mean deviation of 1.25%, and

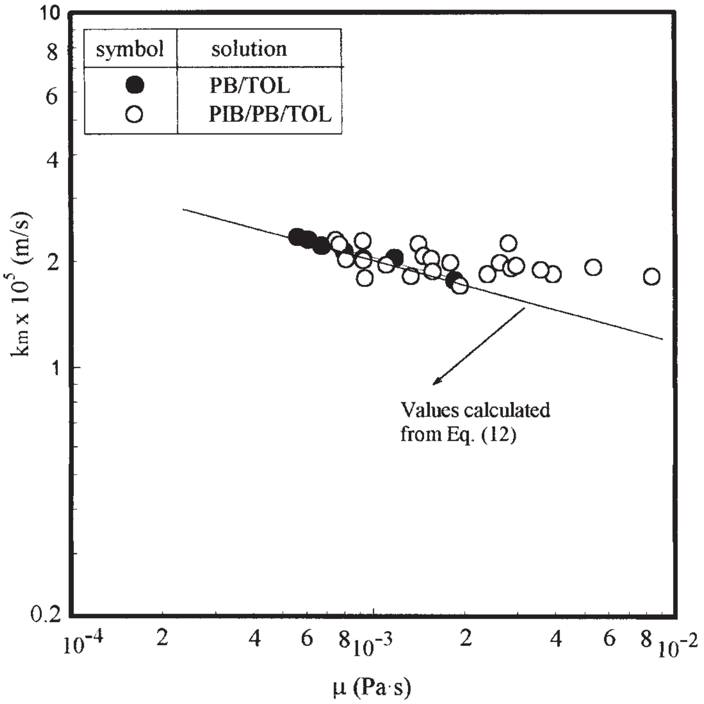

Figure 4. Plot of k_m of Cr(VI) in liquid membrane vs. viscosity of PIB/PB/Tol solution (MD = 13.66%, SD = 3.31%).

standard deviation of 0.03%, and their values are -0.222 and 4.40×10^{-6}, respectively. Also, the plots of open circles (PIB/PB/Tol) deviate from the straight line of PB/Tol with mean deviation of 13.66% and standard deviation of 3.31%.

The empirical correlation between k_m and μ of toluene solution of PB is given as follows:

$$k_m = 4.40 \times 10^{-6} \mu^{-0.222} \tag{12}$$

where μ is in Pa s and k_m in m/sec.

The solid line in Fig. 4 presents the calculated line of k_m from Eq. (12).

To lessen the deviation of the plots for PIB/PB/Tol from those for PB/Tol as shown in Fig. 4, a new term of k_m combined with the elasticity such as $(1 + a_1 Wi^{a_2})$ plotted against μ was used to apply the behavior of non-Newtonian viscoelastic liquid to the empirical form such as Eq. (12). A simple multiple regression exercise gave the values of a_1 and a_2, and their values were

1.153 and 0.817, respectively, with correlation coefficient of 0.98, mean standard of 7.0%, and standard deviation of 0.98%; the correlation is plotted in Fig. 5.

The empirical correlation of k_m with μ for toluene solution of PB and PIB is given as follows:

$$k_m = 4.40 \times 10^{-6}\mu^{-0.222}(1 + 1.16Wi^{0.817}) \tag{13}$$

Figure 6 shows that all of the k_m data with Newtonian toluene solution of PB and non-Newtonian toluene solution of PIB at various concentrations of PB and PIB agree within mean deviation of 7.0% and standard deviation of 0.98% with the prediction by Eq. (13). As shown in Fig. 6, the deviation of plots of PIB/Tol can be eliminated by incorporating *Wi* into the correlation, although the deviation is small because the elasticity of PIB is small in the low concentration range of PIB such as 0–1.0 wt%.

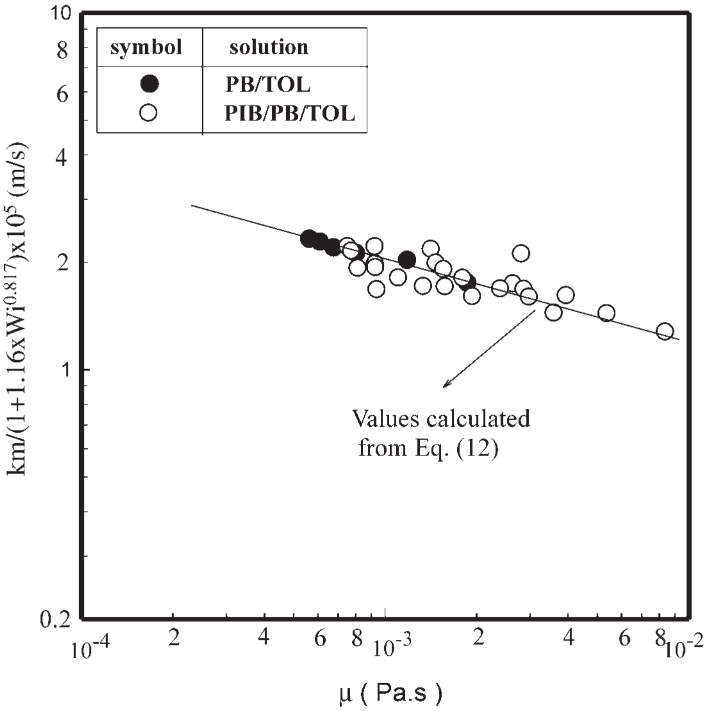

Figure 5. Correlation of k_m of Cr(VI) in liquid membrane vs. viscosity of PIB/PB/Tol solution by Weissenberg number (MD = 7%, SD = 0.98%).

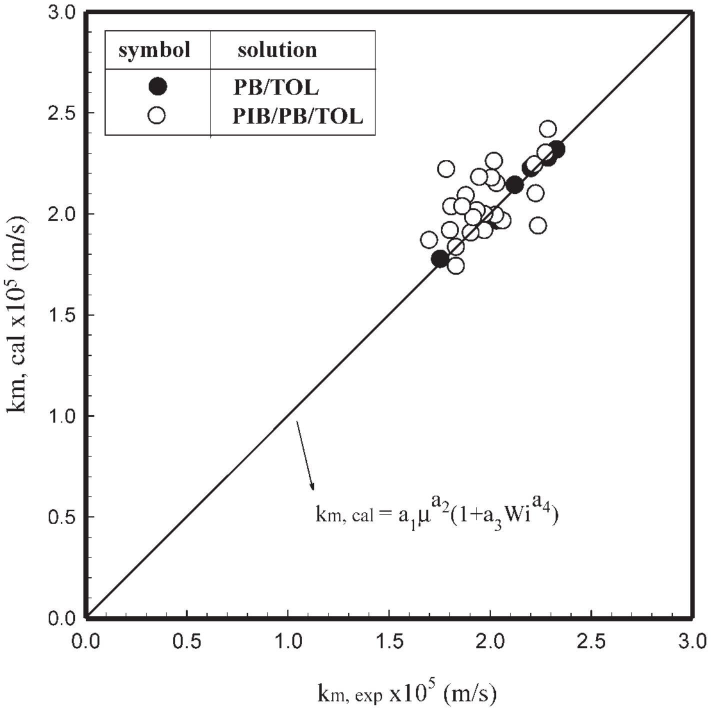

Figure 6. Comparison of $k_{\mathrm{m,cal}}$ with $k_{\mathrm{m,exp}}$ (MD = 6.45%; SD = 0.69%; $a_1 = 4.40 \times 10^{-6}$; $a_2 = -0.222$; $a_3 = 1.16$; $a_4 = 0.817$).

CONCLUSIONS

Transport rates of Cr(VI) from the aqueous solution of the feed side to the aqueous NaOH solution of the stripping side through the toluene solution of PB and PIB of the membrane side supported by the microporous polymer were measured at the agitation speed of the feed and stripping side of 500 rev/min and the initial concentration of Cr(VI) in the feed side of $50\,\mathrm{g/m^3}$. The toluene solutions of PB and PIB were used as pseudoplastic and viscoelastic liquid, respectively, and the concentration range of PIB was 0.1–1.0 wt%, and that of PB, 0–30 wt%. The rheological properties of the toluene solution, such as n, K, b, and A, were used to correlate the primary normal stress difference presenting the elastic properties with the membrane-side mass transfer coefficient. The elastic properties, considered in the form of Weissenberg number, were found to decrease the value of membrane-side mass transfer coefficient. An empirical correlation of the membrane-side mass transfer coefficient incorporating Weissenberg number was obtained as follows:

$$k_{\mathrm{m}} = 4.40 \times 10^{-6}\mu^{-0.222}(1 + 1.16Wi^{0.817}).$$

NOMENCLATURE

a	Contact area per unit volume ($\mathrm{m}^2/\mathrm{m}^3$).
A	Rheological properties defined as in Eq. (8) ($N_s^{\mathrm{b}}/\mathrm{m}^2$).
b	Rheological properties defined as in Eq. (8).
C_{Ai}	Concentration of Cr(VI) in i phase ($\mathrm{kmol}/\mathrm{m}^3$).
De	Deborah number defined as λ/t.
C_{af}^*	Concentration of A in feed side at equilibrium of that in stripping side ($\mathrm{kmol}/\mathrm{m}^3$).
C_{as}^*	Concentration of A in stripping side at equilibrium of that in feed side ($\mathrm{kmol}/\mathrm{m}^3$).
d	Impeller diameter (m).
D_{C}	Diffusivity of solute ($\mathrm{m}^2/\mathrm{sec}$).
H_{A}	Partition coefficient of A defined as the ratio of concentration in the organic phase to that in the aqueous phase at phase equilibrium state.
K	Rheological properties defined as in Eq. (6) ($N_s^{\mathrm{n}}/\mathrm{m}^2$).
K_{o}	Overall mass transfer coefficient of A in feed side (m/sec).
k_i	Mass transfer coefficient of i side (m/s).
$k_{\mathrm{L}}a$	Volumetric mass transfer coefficient (s^{-1}).
n	Rheological properties defined as in Eq. (6).
N	Agitation speed (sec^{-1}).
$N_{\mathrm{A}}^{\mathrm{o}}$	Flux of A ($\mathrm{kmol}/\mathrm{m}^2\mathrm{sec}$).
N_1	Primary normal stress difference defined as in Eq. (8) (N/m^2).
Re	Reynolds number defined as $d^2N\rho/\mu$ in an agitated vessel.
Sh	Sherwood number defined as $k_{\mathrm{L}}ad^2/D_{\mathrm{L}}$ in an agitated vessel.
t	Time (sec).
Wi	Weissenberg number defined as N_1/τ.

Greek Letters

γ	Shear rate (sec^{-1}).
λ	Characteristic liquid time (sec).
μ	Viscosity of the membrane-side liquid (Ns/m^2).

ρ	Density ($\mathrm{kg/m^3}$).
τ	Shear stress defined as in Eq. (6) ($\mathrm{N/m^2}$).

Subscripts

f	Feed side.
m	Membrane side.
s	Stripping side.
o	Initial value.

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