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Interfacial Tension of Poly(ethylene glycol) + Salt + Water Systems

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The interfacial tension of eight poly(ethylene glycol) (PEG) + salt aqueous two-phase systems, PEG + $(NH_4)_2SO_4$, PEG + K_2HPO_4, PEG + xK_2HPO_4 + KH_2PO_4 (x is the molar ratio of K_2HPO_4 to KH_2PO_4, $x = 1.42$, pH = 7.0), PEG4000 + xK_2HPO_4 + KH_2PO_4 (at different pH or x values), PEG + K_3PO_4, PEG + Na_2SO_4, PEG + $MgSO_4$, and PEG + Na_2CO_3 systems, is reported. The influence of polymer molecular weight and concentration, salt concentration and type, and the total solute compositions on the interfacial tension has been studied.

Introduction

Aqueous two-phase extraction is useful for separating materials of biological origin. In this technique, the interfacial tension (σ) between the phases is an important determining factor that influences the partitioning behavior of particles or cells because such materials usually distribute themselves between the interface and one of the two bulk phases, leaving the other phase depleted of particles (Bamberger et al., 1984). The degree to which interfacial adsorption will occur increases with increasing σ because of the decrease in free energy associated with loss of free liquid−liquid interfacial area between the native phases. To understand the mechanism behind the distribution of particles in aqueous two-phase systems, it is of interest to know the properties of the liquid−liquid interface and particularly the values of the interfacial tension.

Ryden and Albertsson (1971) were the first to report results on the interfacial tension of polyethylene glycol + dextran + water systems. They investigated the effect of total polymer concentration (tie-line length) on interfacial tension. Other studies of the interfacial tension of PEG + dextran systems include those of Bamberger et al. (1984) and Forciniti et al. (1990). Bamberger et al. studied the effect of the type and concentration of salt additives on the interfacial tension, while Forciniti et al. systematically determined the interfacial tension and explored the influence of polymer molecular weight, total polymer concentration, and temperature on the interfacial tension. As to polymer + salt + water systems, Jafarabad et al. (1992) measured the interfacial tension of PEG4000 + phosphate (pH = 7.0) and PEG4000 + Na_2SO_4 systems at 30 °C, using a stalogmometer with the determination error of 10%. Except for this, there are few published interfacial tension data, or descriptions of the factors that influence interfacial tension.

This study presents data on interfacial tension for different polymer + salt + water systems and explores the influence of the total solute concentration, polymer molecular weight and concentration, and salt type and concentration on the interfacial tension.

Materials and Methods

Materials. Poly(ethylene glycols) were purchased from Shanghai Chemical Reagent Factory (P.R. China), with six different number average molecular weights (M_n) of 1000 (950−1050), 1500 (1300−1700), 2000 (1900−2100), 4000 (3000−4500), 6000 (5500−7500), and 20 000 (18 000−20 000). All other chemicals were analytical grade with purities of more than 99.5 mass %. Stock aqueous solutions of PEG (30−50 mass %) and salt (30−50 mass %) were prepared using double-distilled water and were checked by measuring the dry mass (vacuum drying).

Methods. Phase systems of 80 g each were prepared by mass from the PEG and salt stock solutions (thoroughly mixed before weighing). Water was added to reach the final mass. After thorough mixing, the systems were placed in a water bath for 8 h to separate the two phases. The top and bottom phases were then separated with pipets (the bottom phase is taken through the top phase using a small positive pressure to avoid contamination). Each phase was kept in the water bath and was shaken violently before using. The compositions of each phase were determined from phase diagram data in the literature, using the average tie-line slope method as proposed by Zaslavsky (1995). The phase diagrams of PEG + $(NH_4)_2SO_4$ and PEG + K_3PO_4 systems are from our own research group (Mei, 1988), PEG + xK_2HPO_4 + KH_2PO_4 (x is the molar ratio of K_2HPO_4 to KH_2PO_4, $x = 1.42$, pH = 7.0) systems from Albertsson (1986), PEG + K_2HPO_4 and PEG4000 + xK_2HPO_4 + KH_2PO_4 (at different pH or x values) systems from Peng et al. (1994), and PEG + Na_2SO_4, PEG + Na_2CO_3, and PEG + $MgSO_4$ systems from Snyder et al. (1992), respectively. The density of each phase was also determined to be ± 0.2 kg·m^{-3} using a 5 cm^3 density bottle and a 0.1 mg balance.

Interfacial Tension Measurements. A self-constructed spinning drop tensiometer was used in this work to determine the interfacial tension. We have reported its construction and calibration elsewhere (Wu et al., 1996) and will not describe the details here. The apparatus is capable of determining very low interfacial tensions (<0.01 mN·m^{-1}) with a precision of 3%. For interfacial tensions larger than 0.05 mN·m^{-1}, the apparatus has been tested by determining the interfacial tension of butan-1-ol + water, 2-methylpropan-1-ol + water, butyl acetate + water, butan-1-ol + air, and some PEG + dextran + water systems, with an error of about 3%. The temperature of the apparatus was thermostated at the determination temperature to ±2 °C using an outer air bath. The spinning drop tensiometer consists of a one-side sealed, Pyrex glass tube which spins about its axis of symmetry in a horizontal position. The rotating tube was filled with the bottom phase using a 10 cm^3 syringe; a tube lid with a small central hole was put on to avoid the liquid flowing out. Then a small drop of top phase (usually 0.5−5 μL)

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Table 1. System Composition (100W_A and 100W_B), Phase Concentration Differences (100ΔW_A and 100ΔW_B), Density Difference ($\Delta\rho$), Tie-Line Length (TLL), and Interfacial Tension (σ)[a]

100W_A	100W_B	100ΔW_A	100ΔW_B	$\Delta\rho$/kg·m^{-3}	TLL	σ/mN·m^{-1}
PEG1000 (A) + $(NH_4)_2SO_4$ (B), at 25 °C						
13.00	14.18	24.20	12.80	20.5	27.40	0.0180
15.00	14.00	28.80	15.75	30.2	32.80	0.0702
16.75	15.50	35.25	19.75	43.8	40.40	0.265
18.50	16.00	41.20	22.75	56.7	47.06	0.501
PEG2000 (A) + $(NH_4)_2SO_4$ (B), at 25 °C						
9.00	12.45	21.00	9.80	13.2	23.17	0.0211
9.00	13.40	27.10	12.60	16.7	29.89	0.0870
9.00	14.90	31.73	15.30	22.6	35.23	0.214
9.00	15.70	34.40	16.65	25.4	38.22	0.300
PEG4000 (A) + $(NH_4)_2SO_4$ (B), at 25 °C						
7.00	10.95	19.35	7.95	6.5	20.92	0.0200
7.00	12.00	23.83	10.09	10.6	25.88	0.103
12.00	11.30	28.06	12.72	15.0	30.81	0.208
12.90	12.46	32.20	14.70	20.9	35.40	0.342
PGE20000 (A) + $(NH_4)_2SO_4$ (B), at 25 °C						
8.00	8.62	17.24	6.57	1.5	18.45	0.0105
10.00	8.55	22.57	8.25	4.7	24.03	0.0503
10.00	9.13	25.58	9.41	5.7	27.26	0.0923
11.00	9.90	32.00	11.84	9.5	34.12	0.203
PEG1500 (A) + $xK_2HPO_4 + KH_2PO_4$ ($x = 1.42$, pH = 7.0) (B), at 20 °C						
10.74	13.66	18.92	11.03	65.2	21.90	0.0732
9.99	15.21	23.96	14.02	83.3	27.76	0.181
9.28	16.67	27.48	16.34	98.0	31.97	0.266
8.56	18.13	30.52	18.55	110.3	35.72	0.425
PEG4000 (A) + $xK_2HPO_4 + KH_2PO_4$ ($x = 1.42$, pH = 7.0) (B), at 20 °C						
9.42	11.37	11.73	5.87	43.8	13.12	0.0200
8.67	13.36	22.60	11.85	62.8	25.52	0.204
7.93	15.30	27.14	14.86	78.6	30.94	0.342
6.40	19.34	32.64	19.22	110.4	37.88	0.641
4.87	23.37	38.14	23.58	138.6	44.84	1.19
PEG6000 (A) + $xK_2HPO_4 + KH_2PO_4$ ($x = 1.42$, pH = 7.0) (B), at 20 °C						
8.26	10.65	17.55	7.71	34.5	19.17	0.0530
7.79	12.17	22.73	10.50	50.0	25.04	0.161
7.41	13.27	25.18	12.02	57.3	27.90	0.213
7.04	14.37	27.63	13.53	66.2	30.76	0.268
PEG20000 (A) + $xK_2HPO_4 + KH_2PO_4$ ($x = 1.42$, pH = 7.0) (B), at 20 °C						
10.04	7.92	5.89	2.00	16.7	6.22	0.0091
9.91	8.36	11.30	4.05	22.0	12.00	0.0288
9.69	8.94	15.28	5.63	28.6	16.28	0.0686
9.30	9.97	20.32	7.35	34.1	21.61	0.0870
PEG1000 (A) + K_2HPO_4 (B), at 25 °C						
11.68	12.25	22.34	10.43	41.5	24.65	0.0250
13.33	12.65	29.74	14.17	75.9	32.94	0.146
13.64	13.50	32.38	17.20	94.0	36.66	0.242
13.86	15.34	34.11	21.31	123.8	40.22	0.524
PEG2000 (A) + K_2HPO_4 (B), at 25 °C						
10.43	10.46	18.86	8.32	42.0	20.61	0.0436
11.13	10.66	22.01	9.65	50.6	24.03	0.0742
12.12	11.12	27.49	12.57	62.8	30.23	0.140
13.07	11.64	31.43	15.03	74.0	34.84	0.280
PEG4000 (A) + K_2HPO_4 (B), at 25 °C						
9.90	8.99	10.96	4.57	25.0	11.87	0.0270
9.97	9.08	13.61	5.59	28.5	14.71	0.0420
10.45	9.81	21.86	9.21	40.9	23.72	0.116
10.89	10.28	26.00	11.67	50.4	28.50	0.169
12.48	11.43	31.10	15.59	65.9	34.79	0.316
PEG4000 (A) + $xK_2HPO_4 + KH_2PO_4$ ($x = 0.353$, pH = 6.0) (B), at 25 °C						
9.38	12.45	11.75	6.53	36.5	13.44	0.0372
9.53	12.70	15.22	8.48	41.2	17.42	0.0546
9.89	13.15	18.92	10.60	49.3	21.69	0.0895
9.86	13.88	22.04	12.86	60.3	25.52	0.133
PEG4000 (A) + $xK_2HPO_4 + KH_2PO_4$ ($x = 1.26$, pH = 7.0) (B), at 25 °C						
10.05	10.71	15.29	7.46	38.6	17.01	0.0511
10.65	11.13	19.96	9.70	46.5	22.19	0.101
11.37	11.47	23.35	11.35	54.0	25.96	0.144
11.56	12.03	26.09	13.01	60.2	29.15	0.209
12.43	12.88	29.85	15.34	74.4	33.56	0.301
PEG4000 (A) + $xK_2HPO_4 + KH_2PO_4$ ($x = 3.55$, pH = 8.0) (B), at 25 °C						
9.05	9.84	11.28	5.25	27.4	12.44	0.0291
9.31	9.92	14.92	6.88	32.3	16.43	0.0434
9.40	10.66	19.42	9.12	40.8	21.45	0.104
9.87	10.86	21.82	10.37	48.9	24.16	0.159
10.81	11.22	25.57	12.34	52.7	28.39	0.201
PEG1000 (A) + K_3PO_4 (B), at 25 °C						
15.07	12.25	18.05	10.85	108.7	21.06	0.386
15.08	13.10	24.75	14.15	116.4	28.51	0.527
15.19	13.98	29.65	17.15	125.3	34.25	0.715
14.98	15.04	32.80	19.40	133.2	38.11	0.861
PEG4000 (A) + K_3PO_4 (B), at 25 °C						
9.92	10.09	14.15	7.10	54.4	15.83	0.251
9.84	11.40	21.15	10.25	64.0	23.50	0.394
10.32	13.04	29.10	13.75	83.9	30.69	0.585
9.97	14.00	32.82	15.15	92.6	36.15	0.747
PEG1000 (A) + Na_2CO_3 (B), at 25 °C						
15.37	6.97	22.44	8.11	40.4	23.86	0.0687
16.27	7.48	28.41	9.93	55.5	30.10	0.229
17.93	7.98	34.28	12.39	72.0	36.45	0.459
20.22	8.45	38.42	14.46	88.4	41.05	0.751
22.11	9.53	43.05	17.09	109.6	46.32	1.20
PEG4000 (A) + Na_2CO_3 (B), at 25 °C						
14.04	6.44	26.30	7.88	36.4	27.46	0.189
16.885	7.03	33.77	10.80	52.1	35.45	0.477
18.90	7.945	37.80	13.07	70.7	40.00	0.897
21.37	8.605	42.74	15.35	88.1	45.41	1.33
23.285	9.70	46.47	17.98	110.0	49.92	1.99
PEG1000 (A) + Na_2SO_4 (B), at 25 °C						
15.90	9.10	23.6	10.6	62.0	25.87	0.0809
18.35	9.25	31.1	14.2	85.2	34.19	0.220
18.30	10.30	35.0	15.8	99.3	38.40	0.410
18.90	11.10	38.2	17.3	113.4	41.93	0.633
PEG4000 (A) + Na_2SO_4 (B), at 25 °C						
12.45	8.50	20.1	8.00	43.0	21.63	0.248
14.075	8.725	24.1	9.50	51.4	25.90	0.368
15.70	8.95	28.5	11.2	60.4	30.62	0.479
17.65	9.90	34.7	13.7	78.9	37.31	0.800
PEG1000 (A) + $MgSO_4$(B), at 25 °C						
18.45	8.25	24.0	9.30	77.8	25.74	0.0589
18.65	8.85	29.3	11.3	96.4	31.4	0.155
19.35	9.40	33.2	12.9	114.2	35.62	0.280
19.90	9.80	35.4	13.7	123.9	37.96	0.372
PEG4000 (A) + $MgSO_4$ (B), at 25 °C						
15.10	7.65	20.6	9.10	72.0	22.52	0.308
16.85	8.25	25.1	11.0	88.6	27.4	0.532
18.60	8.35	27.5	12.1	99.8	30.04	0.661
19.40	8.80	29.9	13.1	108.8	32.64	0.785

[a] x is the molar ratio of K_2HPO_4 to KH_2PO_4. The phase diagrams of PEG + $(NH_4)_2SO_4$ and PEG + K_3PO_4 systems are from our own research group (Mei, 1988), PEG + $xK_2HPO_4 + KH_2PO_4$ ($x = 1.42$, pH = 7.0) systems at 20 °C from Albertsson (1986), PEG + Na_2CO_3, PEG + Na_2SO_4, and PEG + $MgSO_4$ systems from Snyder et al. (1992), and PEG + K_2HPO_4 and PEG + $xK_2HPO_4 + KH_2PO_4$ (at different pH or x values) systems from Peng et al. (1994).

Table 2. Correlation Results of Interfacial Tension versus TLL, ΔW_A, and ΔW_B for PEG (A) + salt (B) Systems with Eqs 4−6

system	a_1	b_1	a_2	b_2	a_3	b_3
PEG1000 + $(NH_4)_2SO_4$	−10.59	6.19	−10.42	6.31	−8.16	5.82
PEG2000 + $(NH_4)_2SO_4$	−8.99	5.37	−8.88	5.45	−6.64	5.03
PEG4000 + $(NH_4)_2SO_4$	−8.68	5.35	−8.75	5.55	−5.66	4.50
PEG20000 + $(NH_4)_2SO_4$	−8.06	4.85	−7.89	4.82	−5.98	5.01
PEG1500 + xK_2HPO_4 + KH_2PO_4 (x = 1.42, pH = 7.0)	−5.86	3.53	−5.73	3.60	−4.59	3.33
PEG4000 + xK_2HPO_4 + KH_2PO_4 (x = 1.42, pH = 7.0)	−5.37	3.29	−5.36	3.43	−3.90	2.91
PEG6000 + xK_2HPO_4 + KH_2PO_4 (x = 1.42, pH = 7.0)	−5.73	3.49	−5.78	3.64	−3.85	2.93
PEG20000 + xK_2HPO_4 + KH_2PO_4 (x = 1.42, pH = 7.0)	−3.55	1.90	−3.51	1.91	−2.59	1.80
PEG1000 + K_2HPO_4	−9.92	5.96	−10.66	6.63	−5.78	4.15
PEG2000 + K_2HPO_4	−5.85	3.41	−5.85	3.51	−4.13	3.02
PEG4000 + K_2HPO_4	−3.98	2.23	−3.96	2.28	−2.86	1.98
PEG4000 + xK_2HPO_4 + KH_2PO_4 (x = 0.353, pH = 6.0)	−3.70	1.99	−3.62	2.02	−3.00	1.90
PEG4000 + xK_2HPO_4 + KH_2PO_4 (x = 1.26, pH = 7.0)	−4.81	2.81	−4.73	2.85	−3.66	2.66
PEG4000 + xK_2HPO_4 + KH_2PO_4 (x = 3.55, pH = 8.0)	−4.34	2.52	−4.25	2.54	−3.32	2.43
PEG1000 + K_3PO_4	−2.21	1.35	−2.10	1.33	−1.86	1.39
PEG4000 + K_3PO_4	−2.19	1.32	−2.06	1.26	−1.80	1.39
PEG1000 + Na_2CO_3	−6.99	4.25	−6.99	4.33	−3.93	3.27
PEG4000 + Na_2CO_3	−6.43	3.96	−6.61	4.14	−3.30	2.88
PEG1000 + Na_2SO_4	−7.09	4.23	−6.93	4.24	−5.39	4.16
PEG4000 + Na_2SO_4	−3.41	2.10	−3.34	2.10	−2.53	2.13
PEG1000 + $MgSO_4$	−7.93	4.76	−7.79	4.76	−5.82	4.75
PEG4000 + $MgSO_4$	−3.95	2.55	−3.84	2.54	−2.98	2.58

was injected, and the rotating rate of the axis was raised slowly. The above process needs rapid and skillful operation to avoid having the top phase stray to the tube wall. The drop size (diameter and length) at different rotational velocities, ω, was recorded to calculate the interfacial tension. The interfacial tension of each system was measured over a range of 2000 to 7000 rotations per minute (rpm) to guarantee that gyrostatic equilibrium is reached. The estimation method, as developed by Princen et al. (1967), was used to calculate interfacial tension. The estimated accuracy of this method is better than 0.05% and is negligibly small when compared with the uncertainty of the measured drop size. Thus, the total accuracy of the interfacial tension measurement is in agreement with the calibration error of 3%.

Results and Discussion

For each PEG (A) + salt (B) system, Table 1 lists the total compositions ($100W_A$ and $100W_B$), concentration difference of PEG ($100\Delta W_A$), concentration difference of salt ($100\Delta W_B$), density difference between the phases ($\Delta\rho$), the tie-line length (TLL) and interfacial tension (σ) studied at the given temperature. ΔW_A, ΔW_B, $\Delta\rho$, and TLL are defined as follows:

$$\Delta W_i = W_i^T - W_i^B \qquad i = A \text{ or } B \tag{1}$$

$$\Delta\rho = \rho^T - \rho^B \tag{2}$$

$$\mathrm{TLL} = 100(\Delta W_A{}^2 + \Delta W_B{}^2)^{1/2} \tag{3}$$

where the superscripts T and B stand for the top phase and the bottom phase, respectively. W_A and W_B are the mass fractions of PEG and salt, respectively. In Table 1, xK_2HPO_4 + KH_2PO_4 is the mixed salt, the molar ratio, x, of K_2HPO_4 to KH_2PO_4, and therefore the pH value, may be different. Table 2 shows the correlation results according to the following three equations as adopted from the literature (Bamberger et al., 1984; Forciniti et al., 1990):

$$\log\sigma = a_1 + b_1 \log(\mathrm{TLL}) \tag{4}$$

$$\log\sigma = a_2 + b_2 \log(100\Delta W_A) \tag{5}$$

$$\log\sigma = a_3 + b_3 \log(100\Delta W_B) \tag{6}$$

where a_i and b_i are correlation parameters. It is found that the above three equations provide good fits to the experimental data with most of the correlation coefficients being better than 0.98 (not shown).

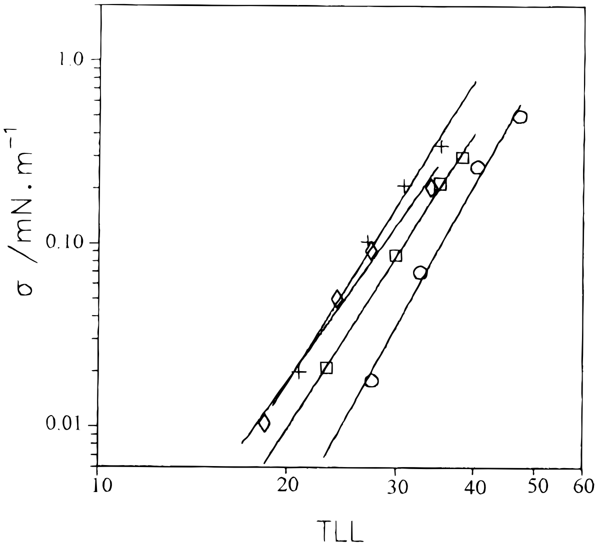

Figure 1. Log−log plots of interfacial tension versus tie line length (TLL) for PEG + $(NH_4)_2SO_4$ systems at 25 °C: (○) PEG1000; (□) PEG2000; (+) PEG4000; (◊) PEG20000.

Figures 1 and 2 are log−log plots of interfacial tension versus TLL and ΔW_B, respectively, for PEG (A) + $(NH_4)_2SO_4$ (B) systems. In general, increasing the molecular weight of polymer increases the interfacial tension. This can best be seen by referring to the log−log plots of σ versus ΔW_B. For example, the log−log plots of σ versus TLL in Figure 1 show that the interfacial tension of the PEG20000 + $(NH_4)_2SO_4$ system is below that of the PEG4000 + $(NH_4)_2SO_4$ system at the same TLL value, but the log−log plots of σ versus ΔW_B in Figure 2 reflect the correct relationship of increasing interfacial tension with increasing molecular weight of polymer at the same ΔW_B value. A similar situation also occurs in PEG + K_3PO_4 systems. It reveals that the salt concentration and the unsymmetrical partitioning of salt between the two bulk phases are the primary determining factors which affect the dependence of interfacial tension on polymer molecular weight, while the total composition (represented by the

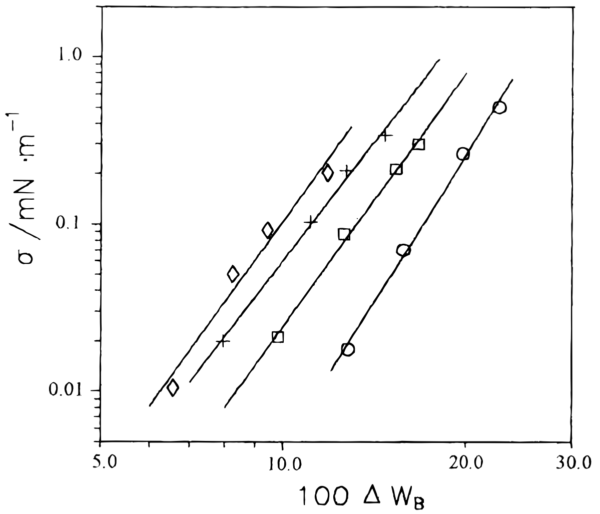

Figure 2. Log−log plots of interfacial tension versus salt difference (100ΔW_B) for PEG + $(NH_4)_2SO_4$ systems at 25 °C: (○) PEG1000; (□) PEG2000; (+) PEG4000; (◊) PEG20000.

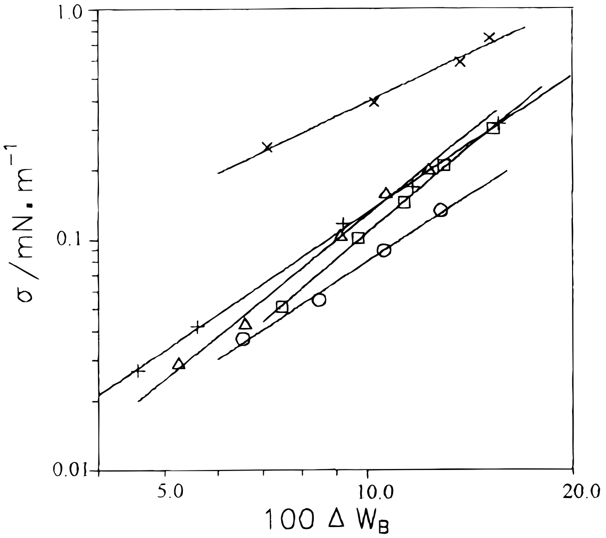

Figure 3. Log−log plots of interfacial tension versus salt difference (100 ΔW_B) for PEG4000 + phosphate systems at 25 °C: (○) $xK_2HPO_4 + KH_2PO_4$, $x = 0.353$, pH = 6.0; (□) $xK_2HPO_4 + KH_2PO_4$, $x = 1.26$, pH = 7.0; (△) $xK_2HPO_4 + KH_2PO_4$, $x = 3.55$, pH = 8.0; (+) K_2HPO_4; (×) K_3PO_4.

plots of σ versus TLL) and the polymer concentration are the secondary ones. It can also be inferred from the correlation results in Table 2 that the slopes, b_i, have a general trend of decreasing with increasing polymer molecular weight. Exceptions to this trend may be attributed to experimental errors of the interfacial tension and the tie-line data of phase diagrams.

It is found that the salt type has also a very strong influence on the interfacial tension. Figure 3 shows the log−log plots of σ versus ΔW_B at 25 °C for PEG4000 + $xK_2HPO_4 + KH_2PO_4$ (at different pH or x values), PEG4000 + K_2HPO_4, and PEG4000 + K_3PO_4 systems. A tendency of increasing interfacial tension with increasing pH value is found. It implies that the higher valence of phosphate has a larger influence on the interfacial tension, and the order of the increasing interfacial tension is $PO_4^{3-} > HPO_4^{2-} > H_2PO_4^{-}$. As to the interfacial tension of PEG + sulfate systems, Figure 4 gives the log−log plots of σ versus ΔW_B. The order of increasing interfacial tension is $Mg^{2+} > Na^+ > NH_4^+$.

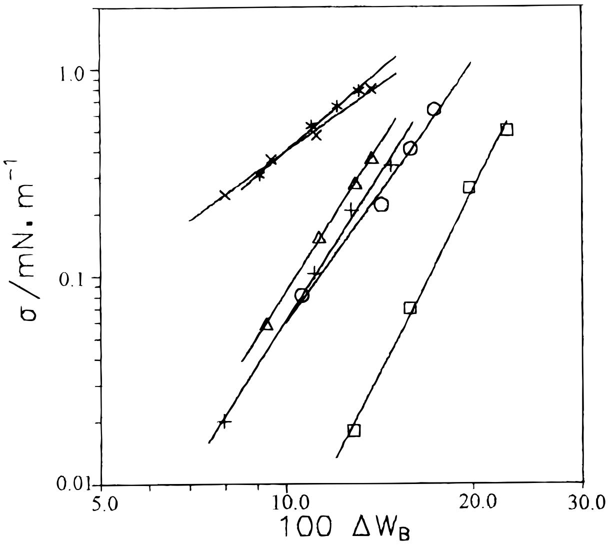

Figure 4. Log−log plots of interfacial tension versus salt difference (100ΔW_B) for PEG1000 + sulfate and PEG4000 + sulfate systems at 25 °C: (□) PEG1000 + $(NH_4)_2SO_4$; (○) PEG1000 + Na_2SO_4; (△) PEG1000 + $MgSO_4$; (+) PEG4000 + $(NH_4)_2SO_4$; (×) PEG4000 + Na_2SO_4; (*) PEG4000 + $MgSO_4$.

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