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Liquid-Liquid Equilibrium of Aqueous Two-Phase Systems Containing PPG725 and Potassium Salts at 298.15 K

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The binodal data and tie-line data for the PPG_{725}-KOH/K_2CO_3/K_3PO_4 aqueous two-phase systems were determined at 298.15 K. Three empirical equations were used to correlate the binodal data and obtained good fit effect. The tie-line composition was correlated by the Othmer-Tobias and Bancraft equations. Furthermore, the effect of salt on the binodal curve for the disquisitive systems have been discussed and it was found that anions with a higher valence are better salting-out agents than anions with a lower valence, which is corresponded to the value of salting-out coefficient and effective excluded volume values. In the investigated systems, the order of salting-out abilities of salts is $K_3PO_4 > K_2CO_3 > KOH$.

Keywords: Aqueous two-phase system, Poly(propene glycol), Potassium hydroxide, Potassium carbonate, Potassium phosphate, LLE.

INTRODUCTION

As an economical and efficient method, liquid-liquid extraction utilizing aqueous two-phase systems (ATPSs) has attracted considerable attention for the separation of biomolecules[1,2], metal ions[3] and drug molecules[4]. Compared with the conventional extraction methods the aqueous two-phase system has several advantages, such as non-toxicity, the lower cost and the easy industrial experimentation. The formation of aqueous two-phase system can be obtained by mixing two different polymers in water or even a salt and a polymer in water above the certain critical concentration. Recently, the new type of aqueous two-phase system containing hydrophilic organic solvent and salt or ionic liquid and salt have been reported.

Poly(ethene glycol) (PEG) is often used as the polymer in the aqueous two-phase systems, the data of which were widely reported in the literature. Poly(propene glycol) (PPG) is a polymer that is structurally closely related to poly(ethene glycol). Poly(propene glycol) with low molecular masses are completely water-soluble and it can form two phases system with suitable salt. However, the liquid-liquid equilibrium (LLE) data of the aqueous poly(propene glycol)-salt systems are relatively scarce. The liquid-liquid equilibrium data of the PPG_{425}/PPG_{725}-NaCl ATPSs[5], PPG_{400}-Na_2SO_4/Na_2CO_3/$NaNO_3$ ATPSs[6], PPG_{425}-Na_2SO_4/$(NH_4)_2SO_4$ ATPSs[7], PPG_{400}-Na_2HPO_4/Na_3PO_4 ATPSs[8], PPG_{425}/PPG_{725}-$Na_3C_6H_5O_7$ ATPSs[9] and PPG_{400}-$K_3C_6H_5O_7$ ATPSs[10] have been reported.

In this paper, we reported liquid-liquid equilibrium data for the new aqueous poly(propene glycol)$_{725}$-salt (KOH, K_2CO_3 and K_3PO_4)-H_2O systems at T = 298.15 K that have not been previously published. The effect of salt on the binodal curves were discussed and it was found that increasing the charge of the anion from -1 (for KOH) to -3 (for K_3PO_4) will cause an expansion of two-phase area. The salting-out effect of salt increase with the increasing the charge of anion, which is proved through the effective excluded volume values and salting out coefficients.

EXPERIMENTAL

Three type of salt (KOH, K_2CO_3 and K_3PO_4) were analytical grade reagents (GR, min. 99 % by mass fraction), which were obtained from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). The polymer PPG_{725} was purchased from Aladdin Reagent Company (Shanghai, China) with the high-grade pure. All the reagents were used without further purification. Double distilled deionized water was used in the experiments.

The binodal curves were determined using cloud point method reported in the literature[11]. The determinations of the binodal curves were carried out in a 50 mL glass vessel. The glass vessel was equipped with an outer skin in which water was circulated using a DC-2008 water thermostat (Shanghai Hengping Instrument Factory, China). The temperature of

water was kept at the constant temperature (298.15 K) and the temperature fluctuation was controlled within 0.05 K. PPG_{725} solution of known mass fraction was taken from the stock and put into the vessel. Then the salt solution of known mass fraction was added until the appearance of turbidity. The composition of this mixture was noted in terms of the mass using an analytical balance (BS124S, Beijing Sartorius Instrument Co., China) with an uncertainty of $\pm 1 \times 10^{-7}$ kg. The water was added in drops until the mixture becomes clear and the above-mentioned procedures were repeatedly performed.

The appropriate amounts of PPG_{725}, salt and water were added into the vessel to determine the tie-line data. After the mixed solution kept stirring for 0.5 h, it was placed in the thermostat water bath whose temperature was controlled at the constant temperature. The two phases were completely separated after 48 h, then, the mass fraction of PPG_{725} and salt in both phases was determined. The mass fraction of salt in two phases was determined using flame photometry (TAS-968, Beijing Purkinje General Instrument Co., Ltd., China). The uncertainty in the measurement of the mass fraction of the salts was 0.0003.

The mass fraction of PPG725 in the top and bottom phases was determined by a refractometer[12] (WZS-I 811639, Shanghai, China) with a precision of ± 0.0001. The following equation (eqn. 1) is used to represent the relationships between the index of refraction of the mixed solution (n_D), the mass fractions of PPG_{725} (w_1) and the mass fractions of salt (w_2).

$$n_D = n_0 + a_1 w_1 + a_2 w_2 \quad (1)$$

where n_0, a_1 and a_2 are constants, the values of which for the investigated systems are given in Table-1. The precision of the mass fraction of PPG_{725} using this method was better than 0.0002.

TABLE-1
VALUES OF PARAMETERS OF Eqn. 1 FOR AQUEOUS SOLUTION OF PPG_{725} + KOH/ K_2CO_3/K_3PO_4 at 298.15 K

System	n_0	a_1	a_2
PPG_{725} + KOH	1.3325	0.1517	0.1925
PPG_{725} + K_2CO_3	1.3325	0.1678	0.1410
PPG_{725} + K_3PO_4	1.3325	0.1345	0.1914

RESULTS AND DISCUSSION

Binodal data and correlation: The binodal data of the systems containing the PPG_{725} and KOH/K_2CO_3/K_3PO_4 were determined at 298.15 K and were shown in the Table-2. The binodal curves for the investigated systems were given in the Fig. 1. The binodal curves were correlated using following equations:

$$w_1 = \exp\left(a + bw_2^{0.5} + cw_2 + dw_2^2\right) \quad (2)$$

$$w_1 = a + bw_2 + cw_2^2 + dw_2^3 \quad (3)$$

$$w_1 = a\exp\left(bw_2^{0.5} - cw_2^3\right) \quad (4)$$

where w_1 and w_2 are the mass fraction of PPG_{725} and salts and a, b, c and d are fitting parameters. For the investigative systems the fitting parameters a, b, c and d obtained from the correlation of experimental binodal data along with the square of correlation coefficients (R^2) and the standard deviations (sd) of eqs. 2, 3 and 4 are given in Tables 3-5, respectively. It was found that eqn. 2 was satisfactorily used to correlate the binodal curves for these three aqueous two-phase systems through analyzing the values of correlation coefficients and standard deviations.

Effective excluded volume: On the basis of the statistical geometry methods, the effective excluded volume (EEV) is calculated with the binodal model that was developed by

TABLE-2
BINODAL DATA FOR THE PPG_{725} + KOH/K_2CO_3/K_3PO_4 (2) + H_2O (3) ATPSs AT 298.15 K AND PRESSURE p = 0.1 MPa[a]

w_1	w_2	w_1	w_2	w_1	w_2	w_1	w_2
PPG_{725} + KOH + H_2O							
3.4463	64.5216	4.3010	46.4160	7.3964	22.3872	13.0251	2.6016
3.6003	59.0592	4.4550	44.0256	8.5668	16.3584	13.9876	1.5936
3.6696	56.6784	5.1557	37.1520	9.4754	11.7216	14.8423	0.9792
3.8544	52.5408	5.6870	33.3792	10.6304	7.6320	15.5888	0.8064
4.0777	48.8544	6.2260	29.5776	11.6237	5.2224	16.4136	1.1712
4.7322	40.8864	6.8189	25.8528	12.4553	3.3792	17.1754	1.0272
PPG_{725} + K_2CO_3 + H_2O							
0.1330	80.6520	0.9100	58.0200	3.7240	27.9840	8.8410	3.2520
0.2730	73.8240	1.0500	55.0320	4.7880	20.4480	9.7160	1.9920
0.3360	70.8480	1.6870	46.4400	5.6140	14.6520	10.4930	1.2240
0.5040	65.6760	2.1700	41.7240	6.6640	9.5400	10.8080	1.0080
0.7070	61.0680	2.6600	36.9720	7.5670	6.5280	11.3760	0.9640
1.3020	51.1080	3.1990	32.3160	8.3230	4.2240	12.6140	0.7840
PPG_{725} + K_3PO_4 + H_2O							
0.1200	73.1880	1.3260	44.8320	3.5940	24.9000	7.4760	4.1040
0.2400	68.1600	1.5420	42.3120	3.9660	22.3200	8.2320	2.4840
0.3180	63.3840	1.7940	39.4680	4.2840	20.1000	8.8560	1.6560
0.4560	59.3520	2.2200	35.4840	5.0340	15.1080	9.9520	0.8024
0.6060	56.2920	2.7000	31.4760	5.3100	13.3560	10.6480	0.3920
0.8760	50.7240	2.9280	29.6400	6.3000	8.3160	12.0440	0.0860
1.0680	47.8080	3.2820	26.8320	6.8520	6.4440		

[a]Standard uncertainties u are u(w) = 0.0001, u(T) = 0.05 K, and u(p) = 10 kPa

TABLE-3
VALUES OF PARAMETERS OF Eqn. 2 FOR THE PPG_{725} (1) + $KOH/K_2CO_3/K_3PO_4$ (2) + H_2O (3) ATPSs AT 298.15 K

System	a	b	c	d	R^2	100 sd^{α}
PPG_{725} + KOH + H_2O	8.8851	-94.0263	261.3343	-755.4863	0.99905	0.06171
PPG_{725} + K_2CO_3 + H_2O	0.0384	-7.4191	15.5099	-333.7651	0.99988	0.02749
PPG_{725} + K_3PO_4 + H_2O	-0.0208	-8.9189	24.2612	-436.5690	0.99966	0.03937

[a] $sd = \left(\Sigma_{i=1}^{n}\left(w_1^{cal} - w_1^{exp}\right)^2 / n\right)^{0.5}$, where w_1^{exp} is the experimental mass fraction of PPG_{725} in Table-2, w_1^{cal} is the corresponding data calculated using eqn. 2. n represents the the number of binodal data

TABLE-4
VALUES OF PARAMETERS OF Eqn. 3 FOR THE PPG_{725} + $KOH/K_2CO_3/K_3PO_4$ (2) + H_2O (3) ATPSs AT 298.15 K

System	a	b	c	d	R^2	100 sd^{α}
PPG_{725} + KOH + H_2O	1.2076	-22.5309	145.9316	-324.1294	0.99269	1.71653
PPG_{725} + K_2CO_3 + H_2O	0.7644	-19.2417	173.9396	-553.9552	0.99280	2.13390
PPG_{725} + K_3PO_4 + H_2O	0.6861	-18.0269	169.9096	-567.7830	0.98950	2.17735

[a] $sd = \left(\Sigma_{i=1}^{n}\left(w_1^{cal} - w_1^{exp}\right)^2 / n\right)^{0.5}$, where is w_1^{exp} the experimental mass fraction of PPG_{725} in Table-2, w_1^{cal} is the corresponding data calculated using eqn. 3. n represents the the number of binodal data

TABLE-5
VALUES OF PARAMETERS OF Eqn. 4 FOR THE PPG_{725} (1) + $KOH/K_2CO_3/K_3PO_4$ (2) + H_2O (3) ATPSs at 298.15 K

System	a	b	c	R^2	100 sd^{α}
PPG_{725} + KOH + H_2O	4.2325	-10.3444	489.6882	0.99621	1.26659
PPG_{725} + K_2CO_3 + H_2O	0.9973	-5.7988	2729.9170	0.99968	0.45922
PPG_{725} + K_3PO_4 + H_2O	0.8998	-6.0125	3352.7591	0.99976	0.33855

[a] $sd = \left(\Sigma_{i=1}^{n}\left(w_1^{cal} - w_1^{exp}\right)^2 / n\right)^{0.5}$, where is w_1^{exp} the experimental mass fraction of PPG_{725} in Table-2, w_1^{cal} is the corresponding data calculated using eqn. 4. n represents the the number of binodal data

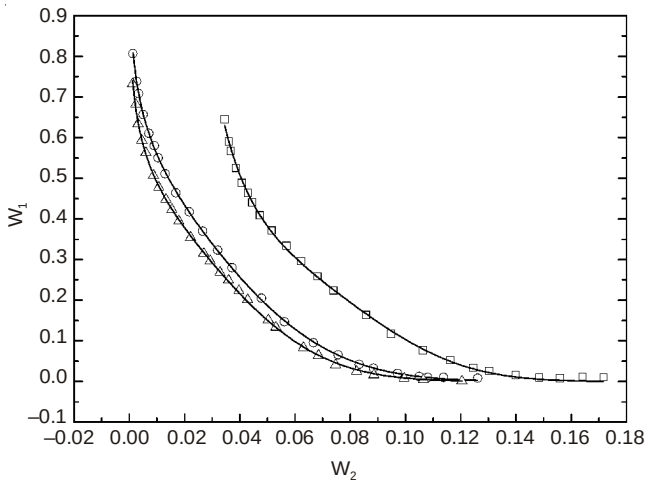

Fig. 1. Binodal curves of the PPG_{725} (1) + $KOH/K_2CO_3/K_3PO_4$ (2) + H_2O (3) ATPSs at 298.15 K. □, KOH; o, K_2CO_3; Δ, K_3PO_4; solid line, reproduced by eqn. 2

Guan *et al.*[13]. In the paper, we applied this model to the PPG_{725}-$KOH/K_2CO_3/K_3PO_4$ systems and the corresponding equation is written as

$$\ln\left(V^*_{213}\frac{w_2}{M_2}\right) + V^*_{213}\frac{w_1}{M_1} = 0 \qquad (5)$$

where w_1 and w_2 are the mass fraction of PPG_{725} and salts, M_1 and M_2 are molecular mass of PPG_{725} and salts, V^*_{213} is the scaled effective excluded volume of salt, respectively. The effective excluded volume values along with the square of correlation coefficients (R^2) and standard deviations (sd) for the investigated systems are given in Table-6. According to the values of effective excluded volume in the table, it was found that the effective excluded volume values rises with the increase in the charge of the anion of the salts at the same temperature because that the salt containing higher valence anion has stronger salting-out ability and it can exclude more effective volume.

TABLE-6
VALUES OF PARAMETERS OF Eqn. 5 FOR THE PPG_{725} (1) + $KOH/K_2CO_3/K_3PO_4$ (2) + H_2O (3) ATPSs AT 298.15 K

T	V^*_{213} (g mol^{-1})	R^2	100 sd^{α}
PPG_{725} + KOH + H_2O	605.38	0.99021	0.06125
PPG_{725} + K_2CO_3 + H_2O	1930.79	0.99380	0.03301
PPG_{725} + K_3PO_4 + H_2O	1750.40	0.99361	0.04747

[a] $sd = \left(\Sigma_{i=1}^{n}\left(w_1^{cal} - w_1^{exp}\right)^2 / n\right)^{0.5}$, where w_1^{exp} is the experimental mass fraction of PPG_{725} in Tables-2, w_1^{cal} is the corresponding data calculated using eqn. 5. n represents the the number of binodal data

Effect of the salt type on the binodal curves: It was found that increasing the charge of the anion from -1 (for KOH) to -3 (for K_3PO_4) will cause an expansion of two-phase area from the binodal curves in the Fig. 1. In other word, at the same concentration of PPG_{725} the concentration of salt will decrease with the increase in the charge of the anion. It can be concluded that the order of the salting-out strength of three salts will be $K_3PO_4 > K_2CO_3 > KOH$ in investigated systems, which lives up to the conclusion obtained by Lu *et al.*[11] On the basis of the effective excluded volume values mentioned above, the anions with a higher valence are better salting-out agents than anions with a lower valence. Therefore, at the same concentration of the PPG_{725} and salt, the two-phase system containing salt with higher valence anion is easier to form than that with lower valence.

Liquid-liquid equilibrium data and correlation: The liquid-liquid equilibrium data of the aqueous two-phase systems composed of PPG_{725} and three salts (KOH, K_2CO_3 and K_3PO_4) were given in Table-7. Also the tie lines for the studied systems were given in Figs. 2 to 4. The Othmer-Tobias and Bancraft equations[14] (eqn. 6 and 7) were used to fit the tie line data.

$$\frac{1-w_1^t}{w_1^t} = k_1\left(\frac{1-w_2^b}{w_2^b}\right)^n \quad (6)$$

$$\frac{w_3^b}{w_2^b} = k_2\left(\frac{w_3^t}{w_1^t}\right)^r \quad (7)$$

where w is the mass fraction, the subscript "1", "2" and "3" represent the PPG_{725}, salt and water, respectively; and the superscripts "t" and "b" stand for the top phase and bottom phase; and k_1, n, k_2 and r are the fit parameters. A linear dependency of the plots $\log\left[\left(1-w_1^t\right)/w_1^t\right]$ against $\log\left[\left(1-w_2^b\right)/w_2^b\right]$ and $\log\left(w_3^b/w_2^b\right)$ against $\log\left(w_3^t/w_1^t\right)$ indicated an acceptable consistency of the results. The Othmer-Tobias and Bancraft equations have been widely used to fit the tie-lines composions[15,16]. The values of the parameters k_1, n, k_2 and r of equations with the corresponding correlation coefficient values (R^2) and standard deviations (sd) are given in Table-8, the values in which show that the fit effect is satisfactory.

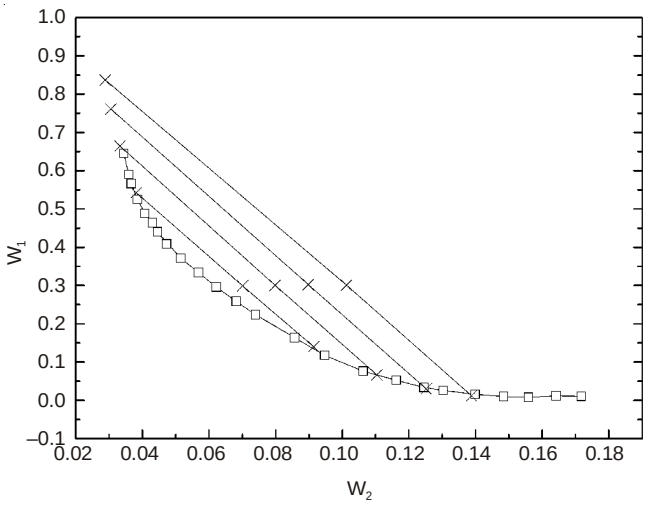

Fig. 2. Tie lines of the PPG_{725} (1) + KOH (2) + H_2O (3) ATPSs at 298.15 K

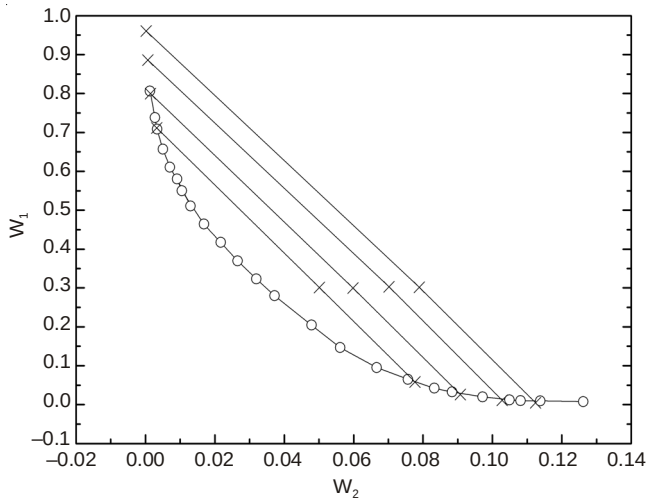

Fig. 3. Tie lines of the PPG_{725} (1) + K_2CO_3 (2) + H_2O (3) ATPSs at 298.15 K

TABLE-7
TIE-LINE DATA FOR THE PPG_{725} (1) + KOH/K_2CO_3/K_3PO_4 (2) + H_2O (3) ATPSs AT 298.15 K AND PRESSURE p = 0.1 MPa[a]

Total system		PPG_{725}-rich phase		Salt-rich phase		Slope (k)	Average of slope
100 w_1	100 w_2	100 w_1^t	100 w_2^t	100 w_1^b	100 w_2^b		
PPG_{725} + KOH + H_2O							
29.91	7.01	54.24	3.82	13.99	9.14	-7.57096	-7.63971
30.01	7.99	66.47	3.33	6.63	11.04	-7.76577	
30.20	8.98	76.08	3.06	3.00	12.52	-7.72797	
30.09	10.13	83.71	2.89	1.20	13.88	-7.49414	
PPG_{725} + K_2CO_3 + H_2O							
30.09	5.01	71.16	0.33	5.85	7.77	-8.77795	-8.61432
29.97	5.98	79.94	0.15	2.58	9.08	-8.65054	
30.29	7.02	88.61	0.07	1.15	10.28	-8.53920	
30.18	7.89	96.06	0.02	0.51	11.24	-8.48957	
PPG_{725} + K_3PO_4 + H_2O							
31.02	3.99	64.00	0.31	8.11	6.40	-9.15928	-9.22662
30.19	4.99	74.99	0.12	3.39	7.88	-9.22445	
29.93	6.01	84.21	0.05	1.38	9.03	-9.20449	
29.81	6.98	93.91	0.01	1.12	9.94	-9.31826	

[a]Standard uncertainties u are u(w) = 0.0001, u(T) = 0.05 K, and u(p) = 10 kPa

TABLE-8
VALUES OF PARAMETERS OF Eqn. 6 AND 7 FOR THE PPG_{725} (1) + $KOH/K_2CO_3/K_3PO_4$ (2) + H_2O (3) ATPSs AT 298.15 K (SYSTEM 1, PPG_{725} + KOH + H_2O; SYSTEM 2, PPG_{725} + K_2CO_3 + H_2O; SYSTEM 3, PPG_{725} + K_3PO_4 + H_2O)

System	$\times 10^{-3} k_1$	n	k_2	r	R_1^2	R_2^2	100 sd_1^{α}	100 sd_2^{α}
System 1	0.75143	3.08193	8.80299	0.20162	0.97996	0.99618	2.7106	1.8271
System 2	0.63281	5.32421	7.97301	0.37919	0.99695	0.99891	1.1431	0.9437
System 3	0.74533	4.43286	6.94261	0.39382	0.99913	0.99923	0.0819	0.2917

[a] $sd = \left[\sum_{i=1}^{N}\left(\left(w_{i,j,cal}^{top} - w_{i,j,exp}^{top}\right)^2 + \left(w_{i,j,cal}^{bot} - w_{i,j,exp}^{bot}\right)^2\right)/2N\right]^{0.5}$, where N is the number of tie lines and j = 1 and j = 2, sd_1 and sd_2 represent the mass percent standard deviations for PPG_{725} and salt, respectively

TABLE-9
VALUES OF PARAMETERS OF Eqn. 8 FOR THE PPG_{725} (1) + $KOH/K_2CO_3/K_3PO_4$ (2) + H_2O (3) ATPSs AT 298.15 K

System	k_4	β	R^2	100 sd_1^{α}	100 sd_2^{α}
PPG_{725} + KOH + H_2O	1.64309	-0.21198	0.99983	0.16644	0.06781
PPG_{725} + K_2CO_3 + H_2O	2.87644	-0.01721	0.99340	0.24192	0.09973
PPG_{725} + K_3PO_4 + H_2O	3.72802	0.05181	0.99941	0.03745	0.07012

[a] $sd = \left[\sum_{i=1}^{N}\left(\left(w_{i,j,cal}^{top} - w_{i,j,exp}^{top}\right)^2 + \left(w_{i,j,cal}^{bot} - w_{i,j,exp}^{bot}\right)^2\right)/2N\right]^{0.5}$, where N is the number of tie lines and j = 1 and j = 2, sd_1 and sd_2 represent the mass percent standard deviations for PPG_{725} and salt, respectively

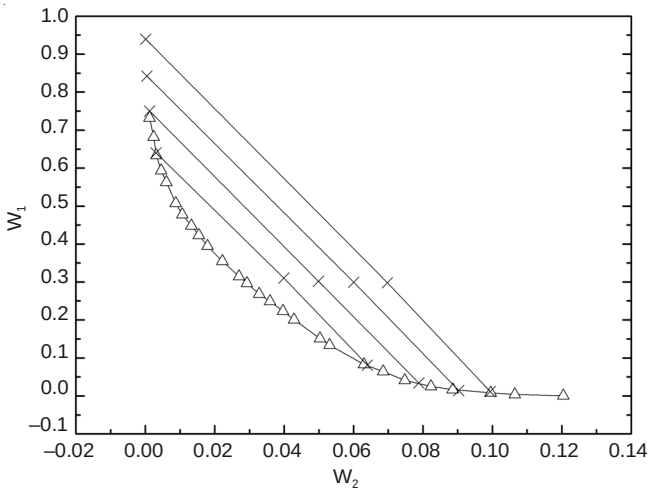

Fig. 4. Tie lines of the PPG725 (1) + K_3PO_4 (2) + H_2O (3) ATPSs at 298.15 K

Salting out coefficient: The simply Setschenow-type equation derived from the binodal theory[17] was used to fit the tie-line data for the investigated systems.

$$\ln\left(\frac{w_2^t}{w_2^b}\right) = \beta + k_4(w_1^b - w_1^t) \quad (8)$$

where the k_4 is the salting-out coefficient and β is the constant related to the activity coefficient. Superscripts "t" and "b" stand for the PPG_{725}-rich phase and salt-rich phase, respectively. For the investigated systems the fitting parameters along with the corresponding standard deviations are shown in Table-9. It was found that the values of salting-out coefficients increase by increasing the charge of the anion, namely, the salting-out effect of the salts containing higher valence anion is stronger than that with lower valence anion. This trend is in agreement with the binodal curves in Fig. 1 and effective excluded volume values in the Table-6.

Conclusion

The phase diagrams for the systems containing poly(propene glycol) (PPG) and three salts (KOH, K_2CO_3 and K_3PO_4) have been determined experimentally. Three experiential equations and Othmer-Tobias and Bancroft and simply Setschenow-type equations were used to correlate the binodal curve and the tie-line data, respectively. The effect of salt type on the binodal data was discussed through the effective excluded volume values and salting out coefficient. It was found that the salting-out effect of salt increase with increasing the valence of anion, namely, the order of the salting-out effect is $K_3PO_4 > K_2CO_3 > KOH$ for the studied salts.

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