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Evaluation of Water Activity in H_2O−PEG400−PEG6000 Solutions between 318.15 and 348.15 K

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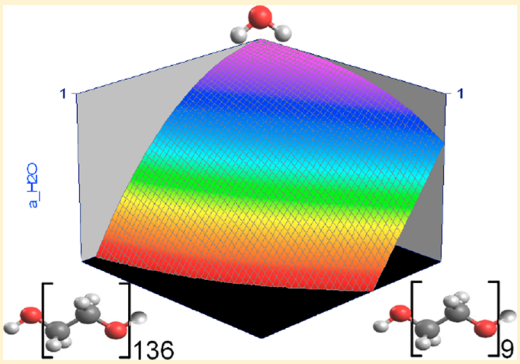
ABSTRACT: The vapor pressure of $H_2O(1)$-polyethylene glycol 400(2)-polyethylene glycol 6000(3) solutions was evaluated experimentally in a temperature range between 318.15 and 348.15 K and in a broad range of concentrations. The dependence of the solvent activity on composition was evaluated and correlated by means of a polynomial expression, in terms of concentrations, volumetric and mass fractions, and temperature, obtaining in all cases standard deviations less than 0.0086 and 0.0106 for $a_1(\phi_2,\phi_3,T)$ and $a_1(w_2,w_3,T)$, respectively.



1. INTRODUCTION

Polyethylene glycol (PEG, Figure 1) polymers have a great number of uses, mainly due to the wide variety of molecular weights with which they can be synthesized and their high solubility in aqueous phase. They are used as nonvolatile and inert components in pharmaceutical preparations (laxatives, drug excipients, ophthalmic drops), anticorrosive formulations, synthesis processes of oriented metallic nanoparticles (allowing regulation of nucleation and growth processes, defining morphology and nature of their crystal faces), development of electrocatalysts, etc. In organic chemistry, they are employed in enzymatic reactions of esterification and hydrolysis, as well as in homogeneous nucleophilic substitution reactions.[1] Currently, they are being used as an alternative to organic solvents to reduce the environmental pollution. Depending on the components of the solutions, concentrations and temperatures, polymers can present phase immiscibility, a feature increasingly used in biology and cell biology for the separation of organic molecules. This property has enabled the use of these systems for the separation of a large number of biomaterials and metal ions.[2] Thus, the availability of thermodynamic properties of aqueous solutions of PEGs over a wide range of temperatures and concentrations should be of great interest.[3] In this context and taking into account that the solvent activity can be evaluated from the experimental determination of the vapor pressure, this property has been measured in water−polymer binary systems, mainly in the temperature range 298.15−308.15 K.[3−11] However, data related to ternary systems formed by two polymers dissolved in water[7,12,13] are markedly scarce, information not having been found for temperatures higher than 308.15 K.

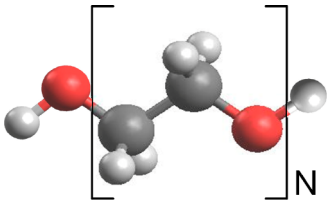


Figure 1. Chemical structure of polyethylene glycol.

In this context, and by virtue of their many industrial applications, the present work studies the dependence of water activity on the composition and temperature of aqueous solutions of polyethylene glycols with markedly different average molecular weights (400 and 6000 g mol^{-1}) at four different temperatures ranging from 318.15 to 348.15 K and in a broad range of concentrations. It should be noticed that the solvent activity of the respective binary solutions of these PEGs has different values for the same concentration.[6]

2. EXPERIMENTAL SECTION

The evaluation of water activity was performed by determining the vapor pressure of the ternary solutions, where both polyethylene glycols, $H(OCH_2CH_2O)_nH$, have a nonvolatile behavior in the temperature range $318.15 \leq T/K \leq 348.15$. PEG 400 ($\rho$ = 1.13 g cm^{-3}), and PEG 6000 (ρ = 1.2 g cm^{-3}, Merck) (used without further purification) and ultra pure water (Pure Lab, Elga Lab Water, 18 MΩ cm) were used for the preparation of the solutions (40 g mass) by gravimetric technique (analytical balance precision 0.0001 g). The comprehensive information on these materials is tabulated in Table 1.

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Table 1. Chemical Samples

chemical name	source	purity	purification
polyethylene glycol 400	Merck	>99.9% (analytical grade)	without further purification
polyethylene glycol 6000	Merck	>99.9% (analytical grade)	without further purification
water	ELGA PureLAB (our lab)	conductivity = 18 MΩ cm	distillation/ ultrafiltration

The experimental device has already been used in previous works,[14,15] where operational details are described. Briefly, it consists of a round-bottom flask of 125 cm^3 capacity containing the solution to be evaluated, stirred by a magnetic bar, connected to a vacuum system and manometer and completely immersed in a thermostatic water bath. The temperature control is performed by a Lauda Thermostat UB20 (±0.01 K) with water recirculation by means of an external pump with a flow rate of 4 dm^3 min^{-1}.

To determine the values of the equilibrium pressure, the system was stabilized for 30 min at a given temperature, then the pressure was registered continuously and it was considered that the system reached equilibrium when pressure value was invariant. Moreover, the vapor pressure of the pure solvent at each temperature was used to ensure the correct operation of the equipment.

3. RESULTS AND DISCUSSION

The vapor pressure of aqueous solutions of PEG400 and PEG6000 mixtures was determined experimentally at 4 temperatures and 68 different compositions. The resulting values are shown in Table 2, where the value corresponding to pure water is indicated in the first row. The reference value obtained by Wagner and Pruss[16] is also included in brackets. The highest value of the difference between the experimental and reference values is 0.40% and corresponds to the lowest temperature.

From the experimental pressure, the activity of the solvent was calculated by the following equation,[17]

$$a_1^{\exp}(C_2, C_3, T) = \frac{P_1(C_2, C_3, T)}{P_1^0(T)} e^{(P_1 - P_1^0)(B_1^0(T) - v_1^0(T))/RT} \tag{1}$$

where P_1 and P_1^0 are the vapor pressures of the solvent in solution and pure water vapor, respectively (P_1^0 was calculated from Wagner and Pruss[16]); C_2 and C_3 are the concentrations of PEG400 and PEG6000, respectively (not used in this calculation); B_1^0 is the second virial coefficient of pure vapor water[18] and v_1^0 is the molar volume of liquid water.[19] These results are illustrated in Table 2.

3.1. Dependence of the Solvent Activity on Composition. To describe analytically the experimental dependence of the solvent activity on composition, a MacLaurin series in two

Table 2. Vapor Pressure (P) and Solvent Activity (a_1) as a Function of Mass Fraction of Solutes and Temperature for the $H_2O(1)-PEG400(2)-PEG6000(3)$ System

		318.15 K		328.15 K		338.15 K		348.15 K	
w_2	w_3	P/kPa	a_1	P/kPa	a_1	P/kPa	a_1	P/kPa	a_1
0	0	9.634 (9.595)	1	15.75 (15.76)	1	25.10 (25.04)	1	38.61 (38.60)	1
0	0.0548	9.561	0.9964	15.69	0.9955	25.01	0.9985	38.50	0.9975
0	0.3075	9.538	0.9940	15.64	0.9920	24.96	0.9966	38.42	0.9955
0	0.3183	9.527	0.9929	15.68	0.9945	24.89	0.9938	38.09	0.9868
0	0.3572	9.511	0.9912	15.65	0.9931	24.76	0.9887	37.94	0.9830
0	0.4008	9.435	0.9834	15.49	0.9824	24.61	0.9826	37.97	0.9837
0	0.4153	9.433	0.9830	15.46	0.9808	24.68	0.9856	38.18	0.9893
0	0.4539			15.42	0.9781	24.47	0.9771	37.69	0.9764
0	0.4480	9.377	0.9771	15.37	0.9751	24.41	0.9746		
0	0.5295	9.117	0.9502	15.04	0.9544	24.09	0.9618	37.31	0.9668
0	0.5383	9.075	0.9458	15.01	0.9522	23.79	0.9500	37.09	0.9610
0	0.5769	8.857	0.9230	14.76	0.9365	23.64	0.9440		
0.1027	0	9.545	0.9947	15.67	0.9939	24.91	0.9946	38.31	0.9927
0.1919	0			15.55	0.9867	24.60	0.9823	38.09	0.9870
0.2776	0	9.403	0.9800	15.43	0.9790	24.54	0.9800	37.95	0.9834
0.3508	0	9.257	0.9647	15.23	0.9660	24.39	0.9739	37.70	0.9768
0.3969	0	9.211	0.9599	15.05	0.9545	24.02	0.9592	36.91	0.9562
0.4926	0			14.64	0.9285	23.20	0.9263	36.39	0.9429
0.6044	0	8.410	0.8764	13.92	0.8834	22.23	0.8876	34.78	0.9011
0.6753	0	7.947	0.8282	13.24	0.8396	21.35	0.8527	33.40	0.8655
0.7471	0	7.174	0.7476	12.04	0.7635	19.67	0.7853	30.39	0.7874
0.7965	0			11.35	0.7200	18.46	0.7373	29.00	0.7513
0.8166	0	6.423	0.6694	10.93	0.6934	17.84	0.7124	28.04	0.7264
0.8406	0	6.191	0.6452	10.53	0.6683	17.08	0.6821	26.82	0.6949
0.8782	0	5.385	0.5612	9.426	0.5980	15.35	0.6129	24.00	0.6219
0.8933	0	5.105	0.5320	8.751	0.5552	14.31	0.5713	22.68	0.5877
0.9177	0	4.592	0.4785	7.671	0.4866	12.98	0.5182	20.11	0.5210
0.9381	0	3.836	0.3998	6.461	0.4099	10.59	0.4230	16.90	0.4378
0.9593	0	3.012	0.3139	5.026	0.3189	8.311	0.3319	13.11	0.3396
0.0504	0.4097	9.279	0.9671	15.36	0.9745	24.32	0.9710	37.60	0.9741
0.0506	0.5089	8.959	0.9337	14.82	0.9399	23.80	0.9502	36.63	0.9491

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Table 2. continued

		318.15 K		328.15 K		338.15 K		348.15 K	
w_2	w_3	P/kPa	a_1	P/kPa	a_1	P/kPa	a_1	P/kPa	a_1
0.0765	0.2205	9.463	0.9862	15.48	0.9820	24.65	0.9842	38.11	0.9874
0.0768	0.2397	9.434	0.9832	15.56	0.9874	24.69	0.9860	38.07	0.9864
0.0991	0.4880	8.702	0.9069	14.61	0.9268	23.45	0.9362	36.49	0.9455
0.1018	0.3066	9.279	0.9671	15.44	0.9796	24.72	0.9872	37.59	0.9739
0.1076	0.1600	9.449	0.9847	15.58	0.9886	24.81	0.9907	38.25	0.9909
0.1077	0.3797	9.170	0.9556	15.04	0.9544	24.07	0.9612	37.31	0.9666
0.1197	0.2005	9.435	0.9833	15.50	0.9832	24.34	0.9719	37.35	0.9676
0.1221	0.4092	8.941	0.9318	14.78	0.9373	23.59	0.9421	36.68	0.9503
0.1493	0.1006	9.467	0.9866	15.48	0.9821	24.61	0.9826	38.17	0.9889
0.1511	0.3334	9.039	0.9421	14.91	0.9460	24.00	0.9583	37.19	0.9635
0.1518	0.4541	8.493	0.8851	14.27	0.9051	23.13	0.9237	36.27	0.9396
0.1580	0.2625	9.135	0.9521	15.10	0.9580	24.03	0.9597	37.37	0.9682
0.1822	0.1817	9.317	0.9709	15.44	0.9793	24.47	0.9769	37.87	0.9813
0.2060	0.2928	9.366	0.9497	15.17	0.9465	23.84	0.9521	37.00	0.9552
0.2145	0.3704	8.645	0.9009	14.41	0.9144	23.11	0.9228	36.05	0.9341
0.2165	0.0984	9.313	0.9706	15.33	0.9727	24.47	0.9771	37.79	0.9791
0.2373	0.2315	8.990	0.9369	15.06	0.9552	23.86	0.9527	36.93	0.9567
0.2503	0.3643	8.403	0.8758	13.85	0.8786	22.48	0.8978	35.02	0.9074
0.2737	0.1421	9.171	0.9558	15.07	0.9562	23.99	0.9581	37.13	0.9619
0.2757	0.2738	8.705	0.9071	14.49	0.9195	23.34	0.9320	36.44	0.9442
0.2776	0.0461	9.341	0.9735	15.35	0.9735	24.38	0.9735	37.66	0.9757
0.2848	0.0942	9.193	0.9580	15.18	0.9632	24.51	0.9786	37.93	0.9828
0.3173	0.3172	8.269	0.8617	13.95	0.8848	22.51	0.8987	35.21	0.9122
0.3515	0.1510	9.062	0.9314	14.74	0.9275	23.44	0.9360	36.10	0.9352
0.3515	0.0768	9.179	0.9566	15.04	0.9540			37.00	0.9585
0.3569	0.2046	8.671	0.9037	14.40	0.9138	23.10	0.9223	35.98	0.9323
0.3926	0.1018	8.975	0.9354	14.65	0.9295	23.57	0.9412	36.32	0.9410
0.3979	0.1489	8.726	0.9094	14.49	0.9193	22.99	0.9179		
0.4158	0.2583	8.059	0.8399	13.40	0.8501	21.56	0.8611	33.96	0.8799
0.4339	0.0540	9.069	0.9451	14.93	0.9473	23.61	0.9429	36.61	0.9486
0.4340	0.0413	8.862	0.9236	14.61	0.9269	23.51	0.9387	36.51	0.9460
0.4814	0.1110	8.431	0.8787	13.98	0.8870	22.48	0.8976	35.16	0.9109
0.5152	0.0492	8.638	0.9002	14.33	0.9089	22.75	0.9082	35.38	0.9166
0.5258	0.1561	7.861	0.8193	13.18	0.8365	21.51	0.8587	33.62	0.8712
0.5844	0.1003	8.051	0.8391	13.41	0.8506	21.58	0.8617	33.24	0.8611
0.5852	0.0357	8.265	0.8614	13.66	0.8664	22.08	0.8817	34.46	0.8927
0.6627	0.0503	7.833	0.8163	13.05	0.8277	21.05	0.8406	32.36	0.8385
0.7591	0.0475	6.613	0.6892	11.16	0.7077	17.95	0.7168	28.64	0.7419

variables was used, which can be summarized in the following expression,

$$a_1(C_2, C_3) = \alpha_0 + \beta_{12}C_2 + \beta_{13}C_3 + \gamma_{12}{C_2}^2 + \gamma_{23}C_2C_3 + \gamma_{13}{C_3}^2 + \delta_{12}{C_2}^3 + \delta_{23}{C_2}^2C_3 + \delta_{32}C_2{C_3}^2 + \delta_{13}{C_3}^3 + \rho_{12}{C_2}^4 + \rho_{13}{C_3}^4 + \ldots \quad (2)$$

where α_{ij}, β_{ij}, γ_{ij}, δ_{ij}, and ρ_{ij} represent the coefficients of different orders involving compounds ij, and C_i is the concentration expressed in volume or weight fraction ($C_i = \phi_i$ or w_i). The zero order coefficient (α_0) must be unitary,

$$\lim_{x_1 \to 1} a_1(\phi_2, \phi_3) = \lim_{x_1 \to 1} a_1(w_2, w_3) = \alpha_0 = 1 \quad (3)$$

The first order coefficients must satisfy the boundary behavior determined by the Flory−Huggins equation for a ternary system,[20]

$$\lim_{x_1 \to 1} \frac{\partial a_1(\phi_2, \phi_3)}{\partial \phi_j} = \beta_{1j} = -\frac{v_1^0}{v_j^0} \quad (4)$$

$$\lim_{x_1 \to 1} \frac{\partial a_1(w_2, w_3)}{\partial w_j} = \beta_{1j} = -\frac{M_1}{M_j} \quad (5)$$

where j = 2 or 3, and v_i^0 and M_i the molar volume and molecular weight of each compound, respectively. Consequently, the correlation equations on terms of volume fractions can be rewritten as

$$a_1(\phi_2, \phi_3) = 1 - \frac{v_1^0}{v_2^0}\phi_2 - \frac{v_1^0}{v_3^0}\phi_3 + \gamma_2^{\phi}{\phi_2}^2 + \gamma_{23}^{\phi}\phi_2\phi_3 + \gamma_3^{\phi}{\phi_3}^2 + \delta_2^{\phi}{\phi_2}^3 + \delta_{23}^{\phi}{\phi_2}^2\phi_3 + \delta_{32}^{\phi}\phi_2{\phi_3}^2 + \delta_3^{\phi}{\phi_3}^3 \quad (6)$$

Moreover, on terms of weights fractions, the expression turns to be,

$$a_1(w_2, w_3) = 1 - \frac{M_1}{M_2}w_2 - \frac{M_1}{M_3}w_3 + \gamma_{12}^{w}{w_2}^2 + \gamma_{23}^{w}w_2w_3 + \gamma_{13}^{w}{w_3}^2 + \delta_{12}^{w}{w_2}^3 + \delta_{23}^{w}{w_2}^2w_3 + \delta_{32}^{w}w_2{w_3}^2 + \delta_{13}^{w}{w_3}^3 + \rho_{12}^{w}{w_2}^4 + \rho_{13}^{w}{w_3}^4 \quad (7)$$

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Table 3. Correlation Parameters of the Solvent Activity as a Function of the Volumetric (Eq 6) and Mass (Eq 7) Fractions of the Solutes at Different Temperatures

	volumetric fraction								
param.	γ_{12}	γ_{23}	γ_{13}	δ_{12}	δ_{23}	δ_{32}	δ_{13}	ρ_{12}	ρ_{13}
K_{iio}	−4.0480	−5.9982	−1.6781	3.1629	8.1224	−9.4174	−5.7329		
K_{ii1} 10^2	1.0888	1.2233	0.4116	−1.0533	−2.2402	3.2893	1.5929		
	mass fraction								
param.	γ_{12}	γ_{23}	γ_{13}	δ_{12}	δ_{23}	δ_{32}	δ_{13}	ρ_{12}	ρ_{13}
K_{iio}	−1.0489	−2.4945	0.2255	0.5193	−2.1808	−0.4257	−1.7067	−0.7172	−2.1439
$K_{ii1}10^3$	1.3059	9.7110	−0.8671	4.3078	−1.5316	−4.7003	5.1072	−4.0693	5.4907

3.2. Correlation of Experimental Results. A linear temperature dependence for each adjustable parameter K_{ij} (= γ_{ij}, δ_{ij}, ρ_{ij}) was assumed,

$$K_{ij}(T) = K_{ij0} + K_{ij1}T \tag{8}$$

The parameters K_{iio} and K_{ii1} were obtained in the temperature range $298.15 \leq T/\mathrm{K} \leq 348.15$ and include the experimental values obtained by other authors at 298.15, 303.15, and 308.15 K.[5−7] The values of the parameters obtained from the correlations are shown in Table 3. The corresponding values of the standard deviation are illustrated in Table 4. On the other hand, the dependence of water activity on the composition of both polymers correlated by eqs 6 and 7 are illustrated in Figures 2−5.

Table 4. Standard Deviations of Each Correlation As a Function of Temperature

T/K	SD($a_1(\phi_2,\phi_3)$)	SD($a_1(w_2,w_3)$)
all temperatures	0.0086	0.0106
318.15	0.0081	0.0100
328.15	0.0091	0.0104
338.15	0.0088	0.0107
348.15	0.0083	0.0110

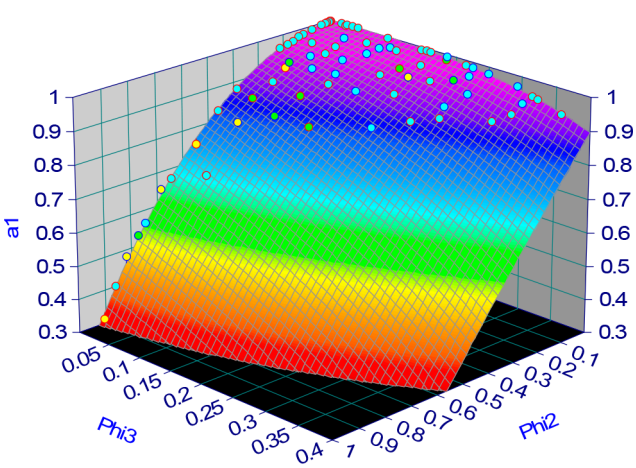

Figure 2. Dependence of water activity as a function of the volumetric fraction of PEG400(2) and PEG6000(3) at 318.15 K. Symbols: experimental data; surface: correlation eq 6.

From the observation of Figures 2−5, it can be clearly appreciated the high quality of the correlation of the experimental results in the range of temperature and composition analyzed. It also shows how the mixture of polymers of the same nature but with a marked difference in molecular weights affects the value of the solvent activity, which is directly related to the linear terms of eq 7.

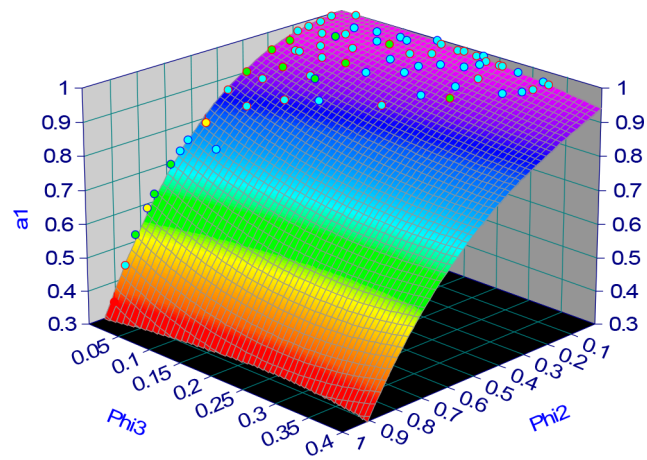

Figure 3. Dependence of water activity as a function of the volumetric fraction of PEG400(2) and PEG6000(3) at 348.15 K. Symbols: experimental data; surface: correlation eq 6.

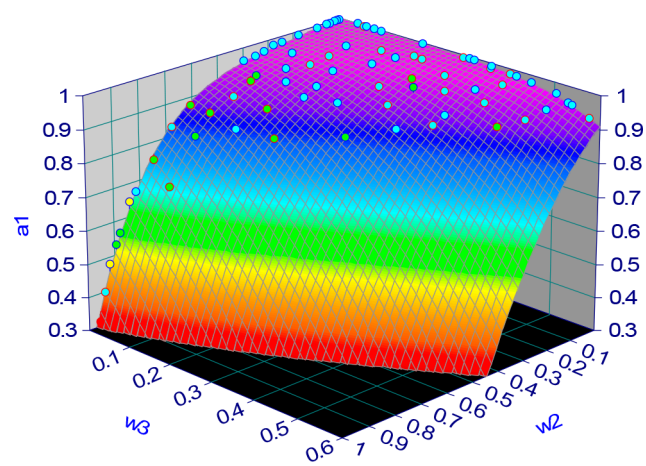

Figure 4. Dependence of water activity as a function of the mass fraction of PEG400(2) and PEG6000(3) at 318.15 K. Symbols: experimental data; surface: correlation eq 7.

4. CONCLUSIONS

The vapor pressure of aqueous solutions of PEG400 and PEG6000 mixtures was determined experimentally at four temperatures and 68 different compositions. The solvent activity was evaluated in the range of $318.15 \leq T/\mathrm{K} \leq 348.15$. From these results and the addition of other data at lower temperatures obtained from the literature, it was possible to obtain for the first time an accurate description of the dependence of water

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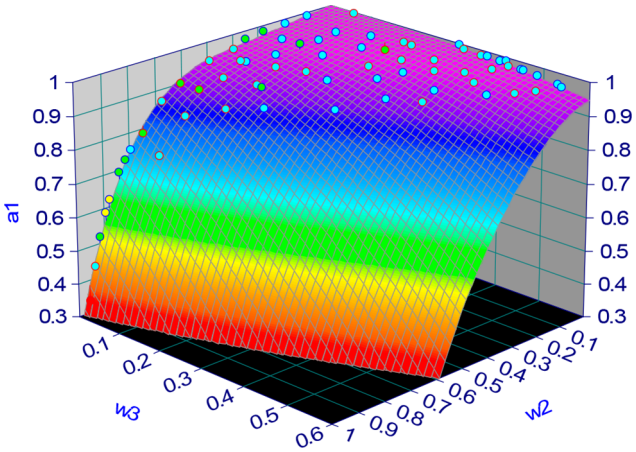

Figure 5. Dependence of water activity as a function of the mass fraction of PEG400(2) and PEG6000(3) at 348.15 K. Symbols: experimental data; surface: correlation eq 7.

activity on composition and temperature for this ternary system. Standard deviations less than 0.0086 and 0.0106 for $a_1(\phi_2,\phi_3,T)$ and $a_1(w_2,w_3,T)$, respectively, were obtained in the temperature range 298.15 < T/K < 348.15 and concentrations range 0 < w_{PEG400} < 0.959; 0 < $w_{PEG6000}$ < 0.538. A thermodynamically consistent equation is thus established for the modeling of the solvent activity over a wide range of temperature and concentrations.

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Notes

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