Refractive Index, Density, and Viscosity in the $NaNO_3$ + H_2O + Poly(ethylene glycol) System at Various Temperatures

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Data are presented on the refractive index, density, and viscosity of unsaturated solutions of sodium nitrate + poly(ethylene glycol) + water at six temperatures between (15 and 40) °C. The range of concentration of the solutions was from (3 to 24) mass % of $NaNO_3$ and from (3 to 24) mass % of poly(ethylene glycol) (PEG) with an average molecular weight of 4000. The Othmer rule was used to obtain correlations between experimental data and the three parameters studied. Data are also presented on the excess molar volumes (V^E) of this system for different compositions and working temperatures.

Introduction

In recent years, numerous studies have been carried out on aqueous systems containing poly(ethylene glycol) (PEG). Poly(ethylene glycol) has numerous uses in biotechnology,[1,2] in chemical partitioning,[3−5] and most recently in extractive crystallization of inorganic salts.[6]

In a previous article[7] we reported on the liquid−liquid equilibrium of the PEG-4000 + sodium nitrate + water system at 25 °C. This system is of great interest for use in the extraction of iodine and other ions from nitrate−iodine mineral deposits in northern Chile. As a continuation of the study of this system, this report presents experimental data on refractive index, density, and viscosity for unsaturated solutions at temperatures between 15 °C and 40 °C.

Data are scarce on physical properties and transport in mixtures containing PEG and salts. Snyder et al.[8] measured the density and viscosity of this type of solution at 25 °C, in biphasic equilibrium. The molar masses of PEG utilized in their measurements were 1000, 3350, and 8000; the salts employed included magnesium sulfate, ammonium sulfate, sodium carbonate, and potassium phosphate.

Mei et al.[9] measured density and viscosity in two aqueous systems: PEG + K_3PO_4 and PEG + $(NH_4)_2SO_4$. Measurement of these properties was carried out with unsaturated solutions and solutions in liquid−liquid equilibrium. This study was carried out at 20 °C, and the molar masses of PEG utilized were (1, 2, 4, 6, and 20) × 10^3.

Zafarani-Moattar et al.[10] and Zafarani-Moattar and Mehrdad[11] presented experimental data on densities of different aqueous mixtures of PEG and salts at (25, 35, and 45) °C. In the first study, PEG molar masses of 1000 and 6000 were employed with the following salts; K_2HPO_4, KH_2PO_4, Na_2SO_4, Na_2CO_3, and $(NH_4)_2SO_4$. In the second study, the PEG molar masses were 2000 and 4000 while the salts utilized were $NaHSO_4$, NaH_2PO_4, and Na_2HPO_4

The values for refractive index, density, and viscosity for the system presently measured have not previously appeared in the literature.

Experimental Section

Materials. Synthesis grade samples of poly(ethylene glycol) with an average molecular weight of 4000 (3500−4500) and analytical reagent grade +99.5% sodium nitrate were obtained from Merck and used without further purification. Drying for a week at 50 °C showed the polymer to contain 0.46 mass % water. Before use, sodium nitrate was dried at 120 °C for 48 h. Distilled/deionized water was used in obtaining all measurements.

Apparatus and Procedures. The solutions were prepared by mass, using an analytical balance with a precision of ±0.0001 g (Denver Instrument Company, model AA-200). The experimental error in the concentration is less than 0.01%. Each solution was agitated using a Mini Vortexer (VWR Scientific Products Co.) and then allowed to rest for 24 h at each working temperature prior to measurement. The refractive index of each solution was measured in triplicate using a Mettler Toledo model RE-40 refractometer having a precision of ±0.0001. The densities of the solutions were measured in triplicate by means of a vibrating tube digital densimeter, model DE-50 (Mettler Toledo) having a precision of ±0.000 05 $g\cdot cm^{-3}$. Before initiation of each measurement run at a given temperature, calibrations of the refractometer and densimeter were made using air and distilled/deionized water as reference materials. An incorporated Peltier system was used to control temperatures with an uncertainty of ±0.01 °C in both the refractometer and the densimeter. The time to reach a stable temperature was 600 s.

Dynamic measurements of viscosity were made in triplicate using a Brookfield model DV-III cone/planar rheometer with a CP-40 spindle. A PolyScience model 9101 water circulation bath was used to maintain the temperature of the measurements to within ±0.02 °C. The average reproducibility of the viscosity measurements was ±0.2 mPa·s.

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Table 1. Values for the Refractive Index n_D, Density ρ, Viscosity η, and Excess Volume V^E in Aqueous Solutions of $NaNO_3$ and PEG-4000 for Different Mass Fractions of Sodium Nitrate (w_1) and PEG (w_2) between (15 and 40) °C

w_1	w_2	n_D	ρ/g·cm^{-3}	η/mPa·s	V^E/cm^3·mol^{-1}	w_1	w_2	n_D	ρ/g·cm^{-3}	η/mPa·s	V^E/cm^3·mol^{-1}
					t = 15 °C						
0.03	0.12	1.3540	1.041 01	4.66	0.025	0.12	0.09	1.3604	1.100 40	3.69	0.169
0.06	0.12	1.3575	1.062 19	4.83	0.069	0.12	0.15	1.3694	1.111 46	7.18	0.209
0.09	0.12	1.3613	1.083 81	5.02	0.123	0.12	0.18	1.3738	1.116 87	9.52	0.236
0.12	0.12	1.3649	1.105 82	5.21	0.190	0.12	0.21	1.3785	1.122 63	13.76	0.259
0.15	0.12	1.3687	1.128 53	5.48	0.265	0.12	0.24	1.3829	1.128 14	17.42	0.291
0.18	0.12	1.3724	1.151 69	5.83	0.354	0.06	0.06	1.3483	1.051 39	2.44	0.054
0.21	0.12	1.3762	1.175 59	6.28	0.453	0.06	0.18	1.3663	1.073 20	8.72	0.086
0.24	0.12	1.3797	1.200 18	6.76	0.565	0.18	0.06	1.3632	1.140 67	2.88	0.287
0.12	0.03	1.3517	1.089 54	1.76	0.134	0.18	0.18	1.3815	1.163 03	11.37	0.432
0.12	0.06	1.3560	1.094 98	2.56	0.150	0.24	0.24	1.3987	1.222 81	26.00	0.839
					t = 20 °C						
0.03	0.12	1.3533	1.039 41	4.07	0.022	0.12	0.03	1.3509	1.087 48	1.58	0.107
0.06	0.12	1.3567	1.060 32	4.26	0.058	0.12	0.06	1.3551	1.092 81	2.23	0.123
0.09	0.12	1.3604	1.081 71	4.43	0.103	0.12	0.09	1.3595	1.098 16	3.26	0.140
0.12	0.12	1.3639	1.103 50	4.60	0.160	0.12	0.15	1.3686	1.109 04	6.25	0.178
0.15	0.12	1.3677	1.126 00	4.86	0.223	0.12	0.18	1.3730	1.114 32	8.12	0.205
0.18	0.12	1.3714	1.148 97	5.12	0.300	0.12	0.21	1.3775	1.120 02	11.84	0.226
0.21	0.12	1.3752	1.172 69	5.47	0.385	0.12	0.24	1.3821	1.125 41	15.17	0.257
0.24	0.12	1.3788	1.197 16	5.95	0.481						
					t = 25 °C						
0.03	0.12	1.3524	1.037 44	3.51	0.020	0.12	0.09	1.3586	1.095 59	2.89	0.128
0.06	0.12	1.3559	1.058 11	3.64	0.053	0.12	0.15	1.3675	1.106 29	5.45	0.162
0.09	0.12	1.3595	1.079 26	3.82	0.094	0.12	0.18	1.3720	1.111 53	7.22	0.185
0.12	0.12	1.3630	1.100 85	4.03	0.146	0.12	0.21	1.3766	1.117 10	9.97	0.204
0.15	0.12	1.3668	1.123 17	4.22	0.204	0.12	0.24	1.3810	1.122 40	13.15	0.232
0.18	0.12	1.3703	1.145 94	4.49	0.274	0.06	0.06	1.3467	1.047 69	1.88	0.041
0.21	0.12	1.3741	1.169 49	4.83	0.352	0.06	0.18	1.3643	1.068 64	6.71	0.069
0.24	0.12	1.3776	1.193 83	5.19	0.439	0.18	0.06	1.3612	1.135 19	2.23	0.218
0.12	0.03	1.3500	1.085 07	1.39	0.099	0.18	0.18	1.3794	1.156 95	8.40	0.340
0.12	0.06	1.3542	1.090 36	1.93	0.112	0.24	0.24	1.3963	1.215 86	18.78	0.672
					t = 30 °C						
0.03	0.12	1.3515	1.035 39	3.13	0.018	0.12	0.03	1.3491	1.082 64	1.28	0.090
0.06	0.12	1.3549	1.055 83	3.27	0.048	0.12	0.06	1.3533	1.087 86	1.76	0.102
0.09	0.12	1.3585	1.076 79	3.39	0.085	0.12	0.09	1.3576	1.093 01	2.57	0.116
0.12	0.12	1.3619	1.098 19	3.52	0.133	0.12	0.15	1.3666	1.103 54	4.84	0.148
0.15	0.12	1.3656	1.120 34	3.74	0.185	0.12	0.18	1.3708	1.108 70	6.41	0.169
0.18	0.12	1.3692	1.142 93	3.94	0.250	0.12	0.21	1.3756	1.114 19	8.65	0.186
0.21	0.12	1.3730	1.166 34	4.26	0.321	0.12	0.24	1.3799	1.119 40	11.22	0.212
0.24	0.12	1.3764	1.190 53	4.59	0.401						
					t = 35 °C						
0.03	0.12	1.3506	1.033 20	2.80	0.016	0.12	0.03	1.3482	1.080 11	1.15	0.084
0.06	0.12	1.3540	1.053 44	2.91	0.044	0.12	0.06	1.3523	1.085 26	1.61	0.094
0.09	0.12	1.3576	1.074 21	2.99	0.079	0.12	0.09	1.3567	1.090 34	2.28	0.108
0.12	0.12	1.3610	1.095 47	3.18	0.123	0.12	0.15	1.3653	1.100 72	4.32	0.137
0.15	0.12	1.3646	1.117 44	3.32	0.172	0.12	0.18	1.3698	1.105 79	5.60	0.157
0.18	0.12	1.3682	1.139 88	3.52	0.232	0.12	0.21	1.3743	1.111 22	7.65	0.172
0.21	0.12	1.3720	1.163 15	3.76	0.298	0.12	0.24	1.3788	1.116 32	9.60	0.197
0.24	0.12	1.3751	1.187 22	4.12	0.371						
					t = 40 °C						
0.03	0.12	1.3498	1.030 84	2.52	0.018	0.12	0.09	1.3556	1.087 55	2.08	0.111
0.06	0.12	1.3531	1.050 91	2.64	0.046	0.12	0.15	1.3642	1.097 79	3.84	0.139
0.09	0.12	1.3566	1.071 51	2.73	0.081	0.12	0.18	1.3685	1.102 82	4.95	0.158
0.12	0.12	1.3599	1.092 61	2.86	0.125	0.12	0.21	1.3733	1.108 15	6.79	0.174
0.15	0.12	1.3635	1.114 43	2.98	0.174	0.12	0.24	1.3777	1.113 21	8.63	0.198
0.18	0.12	1.3671	1.136 73	3.18	0.234	0.06	0.06	1.3443	1.041 02	1.38	0.036
0.21	0.12	1.3708	1.159 88	3.40	0.298	0.06	0.18	1.3612	1.060 89	4.66	0.060
0.24	0.12	1.3739	1.183 83	3.65	0.371	0.18	0.06	1.3578	1.126 38	1.63	0.187
0.12	0.03	1.3472	1.077 45	1.06	0.087	0.18	0.18	1.3761	1.147 37	5.88	0.288
0.12	0.06	1.3513	1.082 80	1.46	0.093	0.24	0.24	1.3925	1.205 23	13.15	0.568

All viscosity measurements were carried out by adjusting the velocity of the cone such that the torque was always at 50% (±0.2%).

Results and Discussion

Table 1 includes experimental values for refractive index, density, and viscosity for the system studied as related to temperature and mass fractions of $NaNO_3$ and PEG-4000.

This table shows that for any composition of the mixture the three physical properties measured all decrease with an increase in temperature. The dependence of these three properties on mass concentration at a given temperature can be observed when the concentration of either $NaNO_3$ or PEG-4000 remains fixed. In both cases, the refractive index, as well as density and viscosity, increases with an increase in the concentration of $NaNO_3$ or PEG-4000. This behavior is observed at each of the six temperatures utilized in this study.

Observations on viscosity presented in Figure 1 (w_1 = 0.12) and Figure 2 (w_2 = 0.12) clearly illustrate the

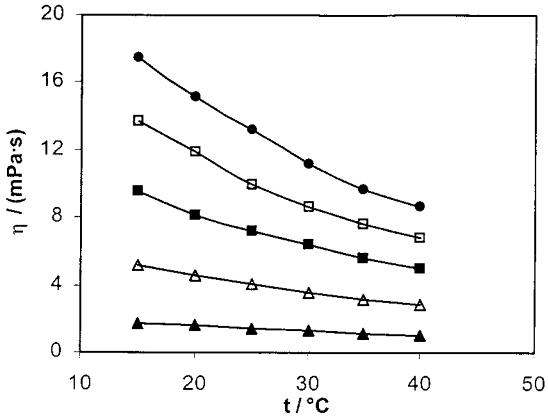

Figure 1. Viscosity of aqueous solutions of PEG-4000 containing 12% mass $NaNO_3$ between (15 and 40) °C: ▲, 3% mass PEG; △, 12% mass PEG; ■, 18% mass PEG; □, 21% mass PEG; ●, 24% mass PEG.

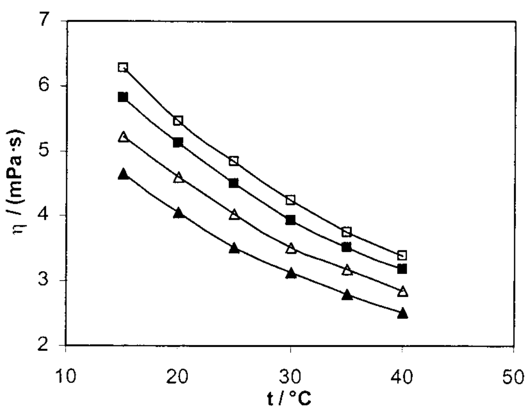

Figure 2. Viscosity of aqueous solutions of $NaNO_3$ containing 12% mass PEG-4000 between (15 and 40) °C: ▲, 3% mass $NaNO_3$; △, 12% mass $NaNO_3$; ■, 18% mass $NaNO_3$; □, 21% mass $NaNO_3$.

tendencies mentioned above. From these figures it may also be observed that the concentration of PEG had a greater influence than that of sodium nitrate on the magnitude of the viscosity of the mixture. The occurrence of this behavior was as expected, given that aqueous solutions of PEG routinely demonstrate viscosity values greater than those of aqueous solutions of electrolytes at the same concentration and temperature.

Graphs prepared similarly to Figures 1 and 2 for refractive index and density (not presented here) showed that these properties both demonstrated (a) a linear decrease with an increase in temperature in contrast to the decrease in viscosity, which was exponential (Figures 1 and 2), and (b) a slightly greater influence of mass % PEG-4000 compared with the concentration of sodium nitrate on the refractive index value of the ternary mixture. The opposite happens with density.

Experimental values for the refractive index, density, and viscosity have been correlated using Othmer's rule.[12] The general expression for the three measured properties in this study as a function of temperature and composition is

$$\ln\left[\frac{Y}{Y_w}\right] = A + B \ln[Y_w] \quad (1)$$

where Y is the physical property of the solution (refractive index, density in g·cm^{-3}, or viscosity in mPa·s) and Y_w is that of water, both at the same temperature.

Table 2. Values for the Parameters in Eqs 2 and 3 for Refractive Index, Density, and Viscosity

	parameters for refractive index	parameters for density	parameters for viscosity
A_1	−0.6984	0.6794	2.1690
A_2	−0.5235	0.1674	10.4164
B_1	2.7312	3.3472	−0.2013
B_2	2.1914	2.1044	1.2662
median error %	0.03	0.04	4
max. error %	0.11	0.17	15

Table 3. Apparent Molar Volumes at Infinite Dilution of $NaNO_3$ ($V^{\infty}_{\phi 1}$) and PEG-4000 ($V^{\infty}_{\phi 2}$)

t/°C	$V^{\infty}_{\phi 1}$/cm^3·mol^{-1}	$V^{\infty}_{\phi 2}$/cm^3·mol^{-1}
15	25.21	3325
20	26.79	3335
25	27.70	3356
30	28.54	3374
35	29.22	3390
40	29.50	3405

In eq 1 the dependency of each physical property on temperature is given in Y_w and the dependence on the composition of the solution is given by the parameters A and B, which represent a linear function of the mass fraction of sodium nitrate (w_1) and PEG (w_2) and are independent of the temperature according to

$$A = A_1 w_1 + A_2 w_2 \quad (2)$$

$$B = B_1 w_1 + B_2 w_2 \quad (3)$$

Values for the constants A_1, A_2, B_1, and B_2 for the three properties are given in Table 2. For the refractive index, eq 1 gave an average deviation of 0.03% and a maximum deviation of 0.09% when compared with the experimental values. For density, the average deviation observed from experimental values was 0.05% with a maximum deviation of 0.2%, while viscosity showed an average deviation of 5% and a maximum deviation of 19%.

Fitting of experimental values in eq 1 was carried out using n_D and η for water at different temperatures. For the density of water the Chen[13] equation was used with a standard deviation of 4.4×10^{-8} g·cm^{-3}.

$$n_{D_w} = 1.18 + (1.7535 \times 10^{-3})(T/K) - (7.193 \times 10^{-5})(T/K)^{3/2} \quad (4)$$

$$\eta_w/\text{mPa·s} = (1.05256 \times 10^{-3})e^{2011/(T/K)} \quad (5)$$

The validity interval for these equations is between 288.15 K and 313.15 K. The values for the refractive index of water used in the fit are those given by Horvath,[14] while for viscosity the data of Isono[15] were used. Equations 4 and 5 showed average deviations relative to experimental values obtained on water of 0.0014% and 0.43%, respectively.

The values of excess molar volumes (V^E) can be calculated from the experimental data according to the following equation:

$$V^E = \frac{\sum_{i=1}^{3} x_i M_i}{\rho_M} - \sum_{i=1}^{3} x_i V_i \quad (6)$$

where ρ_M is the mixture density and x_i, M_i, and V_i are the mole fraction, the molecular weight, and the molar volume

Table 4. Densities of Aqueous Solutions of PEG-4000 at Different Temperatures

m/mol·kg^{-1}	ρ/g·cm^{-3} t = 15 °C	ρ/g·cm^{-3} t = 20 °C	ρ/g·cm^{-3} t = 25 °C	ρ/g·cm^{-3} t = 30 °C	ρ/g·cm^{-3} t = 35 °C	ρ/g·cm^{-3} t = 40 °C
0.01042	1.005 95	1.004 96	1.003 63	1.002 09	1.000 36	0.998 44
0.01596	1.009 44	1.008 37	1.006 97	1.005 36	1.003 57	1.001 59
0.01882	1.011 20	1.010 09	1.008 64	1.007 00	1.005 18	1.003 17
0.02174	1.012 96	1.011 81	1.010 33	1.008 65	1.006 81	1.004 76
0.02475	1.014 75	1.013 55	1.012 03	1.010 31	1.008 43	1.006 37
0.02778	1.016 52	1.015 27	1.013 72	1.011 97	1.010 07	1.007 96
0.03410	1.020 14	1.018 76	1.017 16	1.015 34	1.013 37	1.011 21

of the ith component of the mixture, respectively. The equation $V_3 = M_3/\rho_3$ describes the molar volume of pure water, while those of sodium nitrate and PEG are described by $V_1 = V^{\infty}{}_{\phi 1}$ and $V_2 = V^{\infty}{}_{\phi 2}$, where $V^{\infty}{}_{\phi 1}$ and $V^{\infty}{}_{\phi 2}$ are the apparent molar volumes at infinite dilution of sodium nitrate and PEG, which must be determined on the basis of data on apparent molal volume.

The apparent molal volume (V_ϕ) of a binary solution is related to density by

$$V_\phi = \frac{M_i}{\rho} + \frac{(\rho_w - \rho)}{m_i \rho \rho_w} \tag{7}$$

where m_i is the molality of solute i, ρ is the density of the binary solution, and ρ_w is the density of water.

In general, the apparent molar volumes V_ϕ of aqueous electrolyte solutions are extrapolated to zero concentration to yield apparent molal volumes at infinite dilution $V^{\infty}{}_{\phi}$ using the Conway et al. [11,16] equation:

$$V_\phi = V_\phi^{\infty} + km^{1/2} + bm + cm^{3/2} \tag{8}$$

where b and c are empirical parameters and k is the limiting theoretical slope, the values for which are (1.698, 1.782, 1.868, 1.955, and 2.046) cm^{-3} L$^{1/2}$ mol$^{-3/2}$ at (288.15, 293.15, 298.15, 303.15, and 308.15) K, respectively.[17]

Experimental data on the solutions of sodium nitrate reported by Isono[15] at (15, 20, 25 30, 35, and 45) °C were used to obtain values for V_ϕ. These values were fit to eq 8 to obtain the values for $V^{\infty}{}_{\phi}$ given in Table 3.

To determine the values of $V^{\infty}{}_{\phi}$ for the polymer, it was necessary to make prior measurements of density on PEG-4000 + H_2O. These experimental values are given in Table 4 for different molalities of PEG-4000. Kirincic and Klofutar[18] give some data on density for other concentrations, which we adjusted to a polynomial equation with an absolute deviation of 0.000 01 g·cm^{-3}. For our compositions, this equation predicts densities with average deviations of $\pm$0.000 02 g·cm^{-3} for our experimental data.

Various authors [11,18,20] have suggested that the dependency of V_ϕ on the molality of the polymer is a linear function typified by

$$V_\phi = V_\phi^{\infty} + hm \tag{9}$$

where h is an empirical constant.

Using the values of density from Table 4 and eq 7, values of V_ϕ were obtained for the polymer, which were fitted to eq 9. Table 3 shows the values of $V^{\infty}{}_{\phi}$ for PEG-4000 obtained using this fit.

Once the apparent molar volumes at infinite dilution of sodium nitrate and PEG-4000 are known, as well as the molar volume of water and the density of the ternary mixture, values for V^E are determined by means of eq 6. These values are listed in the sixth column of Table 1.

By observing the values for the excess volume V^E at a given temperature, it can be observed that V^E increases as the concentrations of PEG or $NaNO_3$ increase. However, a differentiating factor can be observed between the two situations. When the concentration of PEG remained fixed, it was noted that the increase in V^E from the low value for $NaNO_3$ of 3% was more than 20 times that for the case when its concentration was increased to 24%. In contrast, it was noted that V^E only increased slightly more than twice when the concentration of PEG in the mixture was increased from 3 to 24%. This behavior was observed at all temperatures studied.

This situation may indicate that the increase of $NaNO_3$ in the mixture produces a greater perturbation (from the structural point of view of the dissolution) than an increase in PEG.

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