

Ethylene Glycol as Entrainer in 1-Propanol Dehydration: Scrutiny of Physicochemical Properties of Ethylene Glycol+1-Propanol Binary Mixture at Different Temperatures

Sanghamitra Pradhan, Sujata Mishra, Lopamudra Acharya

Abstract: Density (ρ), and dielectric permittivity (ϵ) of the binary mixture of ethylene glycol + 1-propanol have been determined as a function of mole fraction of ethylene glycol at $T = (298, 308 \text{ and } 313)\text{K}$. These measurements have been employed to compute thermodynamic properties like molar volume (V), and molar polarization (P) of the liquid mixtures. Excess properties such as VE , ϵE and PE for the binary mixture have also been calculated at different temperatures. In order to calculate the standard deviations of the excess properties, Redlich – Kister polynomial equation was used. The negative and positive values of the excess properties predicted the type and strength of molecular interactions between the components in the binary mixture. The excess molar volume for the mixture at all temperatures is found to be negative. The ϵE values for ethylene glycol + 1-propanol has been found to be negative at 298K and but positive values are obtained at 308 and 313K. The divergent magnitudes of ϵE values at different temperatures have been attributed to different strengths of hydrogen bond interactions as well as alignments of net dipoles.

Keywords: Binary mixture, Ethylene glycol, Excess properties, 1-propanol, Temperature.

I. INTRODUCTION

Investigations on various thermo physical and thermodynamic properties of binary mixtures on the basis of a wide range of mole fractions and temperatures furnish useful information concerning the type of intermolecular associations [1]. Studies on volumetric properties of solutions help in interpreting various types of solute-solvent interactions. Additional parameters such as apparent molar volumes, limiting apparent molar volumes also serve as important tools for analyzing the various thermodynamic interactions in the chosen binary mixture.

Pal and Singh have reported excess molar volumes and viscosities of different kind of ethylene glycol binary mixtures at 298.15 and 308.15K [2]. Investigations on deviation of refractive index, viscosity of mixture and deviation in molar volume for aqueous solution of alcohol and ethylene glycol at 291.15K and atmospheric pressure have also been reported [3]. Cruz et al. carried out viscosity

studies for the first six members of poly (ethylene glycols) (PEGs) at different temperatures and established a generalized correlation basing on the number of carbon atoms [4]. The physicochemical properties of ethylene glycol and water mixture at various temperatures have been investigated by Yang et al [5]. The negative excess molar volumes over the entire range of composition at low temperatures predicted the existence of specific interactions between unlike molecules.

Dielectric investigations elucidate the intermolecular interactions in the binary mixtures which occur due to dipole-dipole, dipole-induced dipole interactions and hydrogen bonding. Several researchers have identified the homo and hetero interactions in various liquid systems with the help of dielectric measurements [6]. Ray et al. have evaluated the excess Gibb's energy of mixing in the polar-nonpolar binary mixture using dielectric probe and proposed an equation to find out excess Gibb's energy [7]. Dielectric permittivity reports of binary mixtures of ethylene glycol mono phenyl ether with alcohols at varied temperatures have been investigated by Rana and Chaube [8]. Dielectric properties of binary mixtures of phenyl ether of ethylene glycol with methanol have also been examined. Rana et al. have studied the physical, electrical and optical behaviour of mixtures of anisole and alcohols at different temperatures [9].

Alcohols serve as an important class of organic compounds which have industrial and scientific significance. These are strongly associated in solution because of dipole-dipole interaction and hydrogen bonding (Figure 1). Increasing attention has recently been paid to find alternative source of energy due to adverse effect of fossil fuels on the environment. One of these proposals is use alcohols as biofuels from renewable energy sources [10]. With the help of esterification process the conversion of 1-propanol to diesel fuel can be carried out [11]. With the azeotropic mixture of 1-propanol and water, the dehydration of 1-propanol becomes very tedious with conventional distillation process. The separation of the mixture becomes feasible with the aid of extractive distillation process by employing a third component in the process to break the azeotrope [12]. Franco et al. have discussed the prospective of ethylene glycol as entrainer in 1-propanol dehydration [13].

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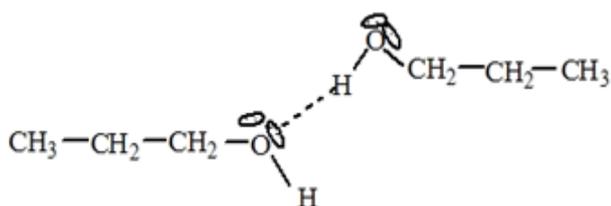


Figure 1: Representation of intermolecular hydrogen bonding in propanol.

Keeping in mind the potential use of ethylene glycol as entrainer, the present investigation focuses on the molecular interactions resulting in modifications of molecular and dipolar orientation between 1-propanol and ethylene glycol at different temperatures (T= 298, 308 and 313K). Few thermo physical data are available in the literature concerning this system at variable temperatures. The excess volume and dielectric permittivity have been evaluated for clear understanding of the thermodynamics of the pure components as well as mixture. Mixing rules proposed by different researchers for dielectric measurements have been used which are significant from theoretical and applied research point of view.

II. EXPERIMENTAL

A. Materials

Ethylene glycol and 1-propanol were obtained from Merck. The state of purity was greater than 0.99 mass percent. The volume loss of chemicals has been eluded with the use of air tight containers. The assessment of pureness of the chemicals has been done by relating the measured densities and dielectric permittivity given in Table 1 with the available literature and the matching of data was observed to satisfactory level.

Table1-List of experimental and literature values of densities and dielectric permittivity of ethylene glycol and 1- propanol at 298, 308 and 313K.

Components	T/K	ρ (g/cm ³) (expt)	ρ (g/cm ³) (lit)
Ethylene glycol (X ₂)	298 K	1.10500	1.10950[13]
	308 K	1.10200	1.10250[14]
	313 K	1.09800	1.09835[14]
1-propanol (X ₁)	298 K	0.80300	0.79940[15]
	308 K	0.79880	0.79161[15]
	313 K	0.79632	0.78777[15]

B. Methods

The binary mixtures of 1-propanol and ethylene glycol of varied mole fractions were prepared by taking desired amount of ethylene glycol in 50 cm³ volumetric flask and adjusting the volume with required amount of 1- propanol. Values of different mole fractions of the binary mixtures were obtained with the help of density and volume data. Density of the pure liquids and binary mixtures have been recorded with the help of (DSA 5000M, Anton Paar). The operating frequency of the densitometer is 2 MHz having accuracy of ± 0.00001 g/cm³. The built in thermostat

maintains the temperature up to ± 0.01 K. Standardization has been carried out with distilled water. The dielectric constants were recorded with dipole meter (Mittal Enterprises, New Delhi). The dipole meter was calibrated with air and reference liquid. The required temperature has been maintained with the aid of digital constant temperature water bath which facilitates the circulation of water through the measuring cell which is double walled. 500 KHz frequency is maintained by the circuit built audio oscillator present inside the equipment. The dipole meter holds an accuracy of ± 0.005 . For measurement of dielectric permittivity of the binary mixtures maximum 50 cm³ for all the mole fractions has been used.

III. THEORETICAL STUDIES

The VE of the binary mixture was calculated with the help of the given equation:

$$V^E = \sum_{i=1}^N X_i M_i (\rho^{-1} - \rho_i^{-1}) \quad (1)$$

Where ρ represents the density of the mixture, ρ_i is the density of the pure component, X_i is the mole fraction and N indicates the number of unlike components.

The molar polarization (P) of the binary mixture was obtained utilizing the equation given below:

$$P = \frac{(\epsilon - 1)}{(\epsilon + 2)} \cdot V \quad (2)$$

Where ϵ is the dielectric permittivity and V is the molar volume of the binary mixture.

The excess dielectric constant and molar polarization were calculated using

$$A^E = A - (A_1 X_1 + A_2 X_2) \quad (3)$$

AE represents the excess properties (ϵ^E , P^E), A1 and A2 represent the same parameters for individual components.

Excess molar volume, V^E (cm³.mol⁻¹), dielectric permittivity, ϵ^E and molar polarization, P^E (cm³.mol⁻¹) were fitted to Redlich- Kister polynomials of the form:

$$V^E, \epsilon^E, P^E = X_1 X_2 \sum_{i=0}^m A_i (2X_2 - 1)^i \quad (4)$$

Resolving a set of linear equations using MATLAB R2015b, the coefficients Ai were obtained and the polynomial degree is represented as m.

Using equation 5, the standard deviation (σ) was calculated; n and P represents the data points and coefficients.

$$\sigma(AE) = \left[\frac{\sum (A_{exp}^E - A_{cal}^E)^2}{(n - p)} \right]^{1/2} \quad (5)$$

IV. RESULTS AND DISCUSSION

Analysis of excess properties of liquid is important to study the interaction in binary mixtures [19]. The densities (ρ), excess molar volume (VE), excess dielectric permittivity (ϵE) and excess molar polarization (PE) on mixing ethylene glycol and 1- propanol at (298, 308 and 313 K) are given in Table 2.

Table 2 Representation of density ρ , excess molar volume excess dielectric permittivity ϵE and excess molar polarization PE of binary mixture of ethylene glycol and 1- propanol at 298, 308 and 313K.

X_2	ρ (g/cm^3)	V^E ($cm^3.m\epsilon^E$)	P^E
Temp = 298 K			
0	0.80300	0	0
0.178	0.84739	-0.144	-1.557
0.278	0.87424	-0.220	-1.458
0.403	0.91068	-0.364	-2.066
0.519	0.94588	-0.420	-2.219
0.654	0.99032	-0.502	-2.010
0.700	1.00365	-0.518	-1.810
0.811	1.04112	-0.44	-1.290
0.981	1.10056	-0.162	-0.261
1	1.10500	0	0
Temp = 308 K			
0	0.7988	0	0
0.178	0.84431	-0.233	-1.980
0.278	0.87168	-0.341	-2.044
0.403	0.90804	-0.464	-3.132
0.519	0.94361	-0.531	-3.242
0.654	0.98723	-0.542	-3.090
0.700	1.00003	-0.521	-2.621
0.811	1.03714	-0.411	-1.571
0.981	1.09551	-0.06	-0.211
1		0	0
Temp = 313 K			
0	0.79632	0	0
0.178	0.84283	-0.335	-2.583
0.278	0.87011	-0.441	-2.605
0.403	0.90864	-0.728	-3.466
0.519	0.94316	-0.717	-3.900
0.654	0.98655	-0.713	-3.580
0.700	0.99964	-0.711	-3.211
0.811	1.03851	-0.700	-2.011
0.981	1.09311	-0.141	-0.233
1	1.098	0	0

The densities of the binary mixtures of ethylene glycol and 1-propanol were recorded at varied temperatures. The difference between the observed thermodynamic parameter of a mixture and the parameter for an ideal liquid is called as the excess function or property. Study of excess thermodynamic properties of liquid mixtures is vital to predict the nature of molecular interaction. The sign and magnitude of excess molar volume gives idea about the strength of molecular interaction. Negative VE indicate chemical interactions whereas positive VE results from unfavorable interactions.

A. Excess molar volume

Excess molar volumes represented in (Figure 2) and the values of VE follow a negative trend over the entire range of mole fraction at the measured temperature [20]. The main factors that affect the excess molar volume are the hydrogen bonding, molecular sizes and shapes. Alcohol molecules present in the binary mixture are strongly associated through

hydrogen bonding. The values of VE are found to depend on temperature and become negative with increase in temperature $T = (298-313)$ K. VE becomes more negative with rise in mole fraction of glycol, reaches a minimum then gradually increases at higher mole fraction. Intermolecular association at higher concentration of propanol is more whereas it decreases with increase in concentration of glycol. Attractive interactions between like molecules become weak with increase in temperature resulting in expansion of volume. The space between larger molecules becomes available for smaller molecules resulting in interstitial accommodation and efficient packing.

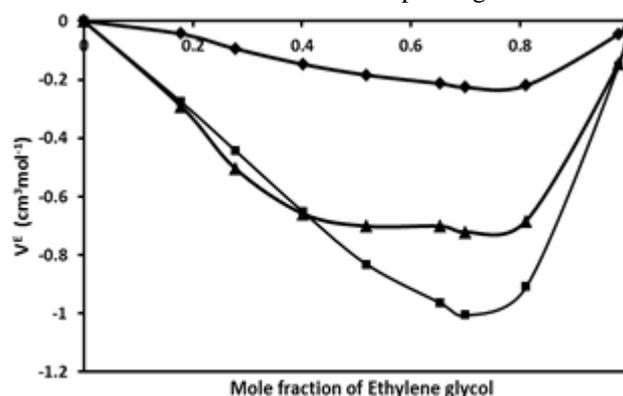


Figure 2: Variation of VE versus mole fraction of ethylene glycol for the binary system of ethylene glycol and 1-propanol at T = (-♦- 298 K, -■- 308 K and -▲- 313 K).

B. Permittivity and mixing rules

Dielectric studies of the binary mixture of polar-nonpolar, polar-polar components are vital for understanding various inter and intra molecular interactions. In the present investigation, three mixing rules have been used for evaluation of dielectric permittivity suggested by Looyenga, Kraszewski and Lichtenecker-Rother [21]. Permittivity values of binary mixtures have been compared with the following mixing rules;

(i) Looyenga

$$\epsilon = \left[\epsilon_1^{1/3} + \phi_2 \left(\epsilon_2^{1/3} - \epsilon_1^{1/3} \right) \right]^3 \tag{6}$$

(ii) Kraszewski

$$\epsilon^{1/2} = \phi_1 \epsilon_1^{1/2} + \phi_2 \epsilon_2^{1/2} \tag{7}$$

(iii) Lichtenecker-Rother

$$\epsilon = \epsilon_1^{\phi_1} \epsilon_2^{\phi_2} \tag{8}$$

where $\epsilon_1, \epsilon_2, \phi_1, \phi_2$, are dielectric constants and volume fractions of 1-propanol and ethylene glycol, respectively.



(iv) The volume fractions, are calculated as,

$$\varphi_i = \frac{X_i V_i}{\sum_{i=1}^2 X_i V_i} \tag{9}$$

Where, the *i*th component has molar volume *V_i*. The root mean square deviation (RMSD) values for the above stated mixing rules have been calculated with the help of equation 10.

$$\text{RMSD} = \left(\frac{1}{n \sum (\epsilon_{\text{exp}} - \epsilon_{\text{cal}})^2} \right)^{1/2} \tag{10}$$

Where, *n* is the number of data points and ϵ_{exp} represents the observed data and ϵ_{cal} signifies the calculated value obtained using the different mixing rules. The experimental values of dielectric constants measured at 298, 308 and 313K are compared with the predicted values and are presented in Table 3.

Table 3 List of experimental and predicted values of dielectric permittivity (ε) Looyenga, Kraszewski and Lichtenecker-Rother at 298, 308 and 313K.

X ₂	ε (Expt)	ε (Looyenga)	ε (Kraszewski)	ε (Kraszewski) ε (Lichtenecker-Rother)
Temp = 298 K				
0	20.017	20.017	20.017	20.017
0.178	21.820	22.308	22.890	22.215
0.278	23.919	23.777	24.629	23.658
0.403	25.812	25.839	26.967	25.789
0.519	27.979	28.000	29.263	27.992
0.654	30.889	30.881	32.153	31.054
0.700	32.209	31.794	33.027	32.029
0.811	34.950	34.564	35.543	34.863
0.981	39.3799	39.470	39.631	39.746
1	40.021	40.021	40.021	40.021
Temp = 308 K				
0	18.750	18.750	18.750	18.750
0.178	20.195	21.044	21.662	20.927
0.278	22.161	22.521	23.425	22.362
0.403	23.610	24.599	25.794	24.487
0.519	25.856	26.785	28.122	26.701
0.654	28.748	29.709	31.053	29.792
0.700	30.339	30.638	31.939	30.782
0.811	33.642	33.462	34.495	33.681
0.981	38.453	38.485	38.650	38.740
1	39.050	39.050	39.050	39.050
Temp = 313 K				
0	18.000	18.000	18.000	18.000
0.178	18.707	20.203	20.797	20.089
0.278	20.635	21.622	22.491	21.466
0.403	22.211	23.618	24.766	23.505
0.519	24.040	25.717	27.003	25.630
0.654	26.992	28.526	29.818	28.596
0.700	28.438	29.419	30.669	29.546
0.811	31.802	32.132	33.124	32.332
0.981	36.896	36.956	37.115	37.197
1	37.500	37.500	37.500	37.500

The calculated values of dielectric permittivity obtained using different mixing rules are substantially equivalent to the experimental values. Kraszewski model provides the best prediction for static permittivity. RMSD values of different theoretical mixture rules are presented in Table 4.

Table 4 RMSD values of different mixing rules for ethylene glycol + 1- propanol at 298, 308 and 313K.

Mixing rule	RMSD		
	Dielectric permittivity (ε)		
	298K	308K	313K
Looyenga	0.084	0.013	0.004
Kraszewski	0.007	0.002	0.001
Lichtenecker-Rother	0.117	0.014	0.004

Dielectric permittivities of the mixtures of polar liquids are significant for understanding types of molecular interactions such as dipole-dipole, dipole-induced dipole and hydrogen bonding interactions. As a consequence, significant deviations are noted from the ideal ones and also the excess permittivity (εE) furnishes information about the associations present in the liquid mixture. It can be seen from the (Figure 3) that negative εE values for ethylene glycol +1-propanol binary mixture are negative recorded at 298, 308 and 313 K. This indicates the presence of polar association between the components leading to the formation of multimers through hydrogen bonding resulting in reduction of effective dipole moment. The variation of εE at various temperatures can be attributed to different power of hydrogen bond interactions and net alignments resulting from dipolar interactions in polar binary mixture [7].

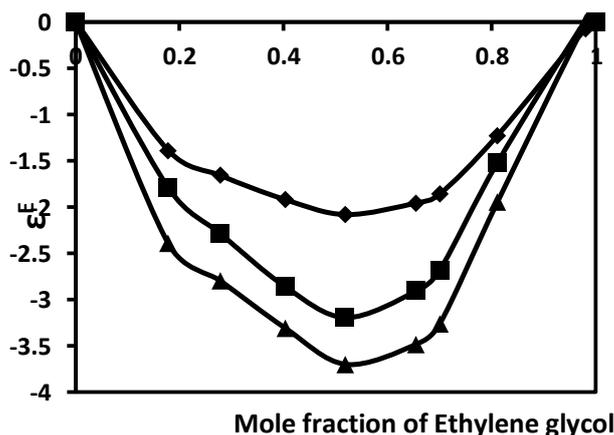


Figure 3: Variation of εE versus mole fraction of ethylene glycol for the binary system of ethylene glycol and 1-propanol at T = (-♦- 298 K, -■- 308 K and -▲- 313 K).

C. Excess molar polarization

Values of excess molar polarization (PE) give good evidence about the interactions in the liquid mixture. Molar polarization provides information about the electrical properties of the molecule and is described by electrical properties per unit volume. PE identifies the range of



interactions which can be short or long between like and unlike molecules. Positive PE indicates the associated region due to parallel alignment of molecular dipoles [22]. Figure 4 shows the variation of PE with different mole fractions of ethylene glycol. The shape of PE curves for T= 308 and 313 K are almost identical. But positive PE is observed at 298K in 1- propanol rich region indicating self-association but with rise in mole fraction of ethylene glycol, it tends to become negative due to interaction between unlike components in the binary mixture. If the variations of PE in terms of temperature are considered, an increase of extrema is observed indicating favourable association between the unlike components. This may be due to geometrical fitting as a result of favourable association between like molecules which decreases with increase in temperature leading to expansion in volume. The coefficients (A_i) of the fitting for VE, ϵ^E and PE with their corresponding standard deviation (σ) are listed in Table 5.

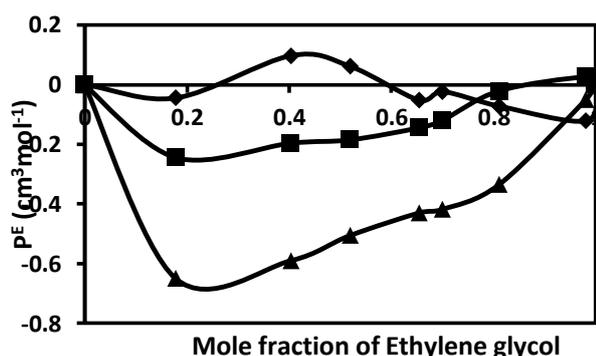


Figure 4: Variation of PE versus mole fraction of ethylene glycol for the binary system of ethylene glycol and 1-propanol at T = (-♦- 298 K, -■- 308 K and -▲- 313 K).

Table 5 Calculated values of Redlich Kister coefficients and standard deviations for the excess functions for ethylene glycol + 1- propanol.

Excess property	Temp (K)	A_0	A_1	A_2	A_3	σ
V^E	298	-0.7306	-0.5916	-0.3869	-0.7779	0.0298
	308	-3.2885	-3.0062	-1.6824	-0.5292	0.0676
	313	-2.8513	-0.3973	-1.0888	-3.8848	0.0009
ϵ^E	298	-8.4271	-2.0946	-0.7144	8.3391	0.0129
	308	-12.9125	-3.9075	4.8125	14.9175	0.0220
	313	-14.9256	-5.4196	1.3281	21.2291	0.0174
P^E	298	0.3967	-1.4961	-1.2188	2.7544	0.0024
	308	-0.7613	0.1858	-0.2787	2.5883	0.0006
	313	-2.1156	1.3374	-2.9869	1.0611	0.0008

V. CONCLUSIONS

From the experimental densities, molar volumes, dielectric permittivity values, excess molar volumes,

dielectric constants and molar polarizations were calculated at T = 298, 308 and 313 K. Negative deviations were observed in all the excess properties mostly over higher composition range of ethylene glycol at T = 298, 308 and 313 K and this could be attributed to the presence of intermolecular interactions in the system studied. Out of the three mixing rules used for theoretical calculation of dielectric permittivity, Kraszewski model worked best. Redlich-Kister polynomial equation was used successfully for the correlation of VE, ϵ^E and PE. The basic findings of this work can be a source of supporting information regarding physicochemical behaviour of ethylene glycol and 1- propanol binary mixture where ethylene glycol is one of the active components used in extractive distillation due to its low toxicity and reduced vapour pressure.

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