Dielectric Properties of Ethane-1,2-diol + 2-Methoxyethanol + Water Liquid Ternary Mixtures

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The dielectric properties of ethane-1,2-diol (1) + 2-methoxyethanol (2) + water (3) were measured in the range $30 \leq t/°C \leq 50$ and for 66 ternary mixtures covering the whole miscibility field ($0 \leq x_1/x_2/x_3 \leq 1$). The ϵ values were analyzed by some empirical equations based on the pure-component properties for the ternaries studied. A comparison of the experimental and calculated data shows that the selected equations can be safely employed to predict the dependence of ϵ on the temperature and on the composition of the systems studied when binary and/or ternary experimental data are not available.

Introduction

The dielectric constant is an important property of matters and of liquids in particular, in connection with the transport phenomena in solution and through biological membranes, responsible for particle solutes orientation and related to the structural order degree in solution. The dielectric properties of the liquid mixtures seem of particular interest for both theoretical and practical reasons.

In general, they cannot be predicted from the dielectric constants of individual components, and the solution of this problem seems to be very far, in spite of several models that have been proposed (*1–3*).

Among other purposes, the dielectric constant may be a useful tool for studying intermolecular interactions in real systems. Furthermore, in the literature it has been reported that the dielectric constant is useful in the determination of the stoichiometry of stable adducts in binary solvent mixtures (*4, 5*). However, very few works appeared in the past about ternary, or higher, liquid mixtures. Therefore, initially we have planned some investigations about the dielectric behavior of the ethane-1,2-diol (ED, component 1) + 2-methoxyethanol (ME, component 2) + water (W, component 3) solvent system in order to provide as much information as possible on the molecular interactions between unlike species.

It should be noted that all three pure species selected for this study are very polar molecules (μ_{ED} = 2.28 D, μ_{ME} = 2.36 D, μ_W = 1.85 D) and amphyprotic in nature.

The pure solvents and their binaries, obtained by mixing each component with each other, have been utilized in the past as potentially useful media for electrochemical investigations on ionization and dissociation reactions in solution (*6–9*). In an extension of our research about the electrolytic behavior into the ternary solvent mixtures, we are recording the experimental measurements on the thermomechanical properties of the above-mentioned ternaries.

Experimental Section

Materials. The solvents ethane-1,2-diol and 2-methoxyethanol (containing <0.10% and <0.05% by mass of water, respectively, as found by Karl-Fischer titrations) were high-purity grade reagents from Carlo Erba (Milan). These solvents were stored over 3-Å molecular sieves for many days before use, and the final purity was checked by gas chromatography (99.5% ED and 99.7% ME), confirming the absence of other significant organic components. Water for mixture preparation was deionized by a MilliQ-Plus apparatus (Millipore) and has a specific conductance ≤0.70 μS at 25 °C, measured at a frequency of 1 kHz.

Apparatus and Procedures. The mixtures were prepared just before use by weight on a Mettler PM 4800 Δ-range balance, operating in a drybox to avoid atmospheric moisture. The binaries without water were preserved on 3-Å molecular sieves. The probable error on each mole fraction x_1, x_2, and x_3 is estimated to be less than 1.5×10^{-4}.

Dielectric constant measurements were carried out at 2 MHz, by the heterodyne beat method, with a Wissenschaftlich-Technischen Werkstätten (WTW) GMBH dipolmeter, model DM01. The thermostated stainless steel measuring cells were MFL 2 ($7 \leq \epsilon \leq 21$) and MFL 3 ($21 \leq \epsilon \leq 90$) types. The sample cells are of the covered coaxial capacitor type with vacuum capacitances of 2 pF (MFL 2) and 0.5 pF (MFL 3). They are adequate to cover the dielectric constant range of ED + ME + W mixtures over the whole investigated composition range. It was checked that a good overlap was obtained when passing from the MFL 2 to MFL 3 measuring cell, the difference lying always within the experimental accuracy of the equipment. The cells were calibrated with standard pure liquids, such as dichloromethane, pyridine, 1-butanol, and acetone (MFL 2) and methanol, ethanol, glycerol and bidistilled water (MFL 3). With the exception of the bidistilled water, all these solvents were spectrograde quality, or better. The dielectric constants for the standards were taken from the literature (*10*).

In all the cases, the experiments were generally performed at least for 10 replicate runs for each composition and at each temperature, and the results were averaged. The reproducibility of measurements (standard deviation $\sigma(\epsilon)$) was approximately equal to ±0.2%, and the accuracy was $\pm 2 \times 10^{-3}$, with a confidence interval of 95%.

The thermostated measuring cell was encased in a polyurethane protective jacket, and the temperature control was provided by a Lauda K2R thermostatic bath maintained to ±0.02 °C.

The temperature constance was checked by a thermoresistance Pt 100 (Tersid, Milan) immersed into the measuring cell, and by collecting the resistance values with a Wayne Kerr 6425 precision component analyzer.

Karl-Fischer titrations were performed with an automatic titration system (Crison Model KF 431) equipped with a digital buret (Crison Model 738).

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Table I. Ternary Composition and Dielectric Constants of Ethane-1,2-diol (1) + 2-Methoxyethanol (2) + Water (3)

		ϵ							ϵ				
x_1	x_2	30 °C	35 °C	40 °C	45 °C	50 °C	x_1	x_2	30 °C	35 °C	40 °C	45 °C	50 °C
1.0000	0.0000	39.95	38.89	37.99	36.98	36.07	0.1513	0.0705	57.44	55.95	54.65	53.30	51.99
0.9272	0.0728	37.41	36.60	35.57	34.80	33.76	0.1342	0.0312	62.90	61.26	59.92	58.43	57.09
0.7442	0.0000	44.90	43.88	42.68	41.60	40.62	0.1213	0.0000	68.35	66.87	65.30	63.69	62.30
0.8499	0.1501	35.11	34.28	33.36	32.57	31.65	0.2614	0.7386	20.55	19.94	19.38	18.79	18.29
0.6760	0.0601	42.62	41.67	40.52	39.49	38.47	0.1916	0.4634	30.08	29.27	28.40	27.62	26.87
0.5638	0.0000	49.56	48.45	47.09	46.05	44.79	0.1618	0.3411	36.20	35.26	34.35	33.39	32.51
0.7676	0.2324	32.68	31.99	31.09	30.31	29.41	0.1362	0.2394	42.68	41.63	40.72	39.53	38.67
0.6053	0.1243	40.34	39.44	38.32	37.35	36.42	0.1208	0.1635	49.40	48.10	46.98	45.70	44.51
0.4952	0.0589	46.99	45.79	44.61	43.59	42.38	0.1018	0.1071	55.53	54.02	52.82	51.45	50.03
0.4274	0.0000	53.92	52.60	51.39	50.10	48.73	0.0959	0.0634	60.78	59.28	57.85	56.37	55.01
0.6798	0.3202	30.43	29.59	28.81	28.01	27.34	0.0822	0.0285	65.61	63.81	62.28	60.68	58.96
0.5296	0.1903	38.09	37.12	36.20	35.23	34.35	0.0749	0.0000	70.75	68.90	67.42	65.69	64.19
0.4422	0.1044	45.08	43.92	42.79	41.82	40.62	0.1359	0.8641	18.66	18.11	17.75	17.18	16.82
0.3756	0.0447	51.72	50.45	49.12	47.88	46.65	0.1138	0.5655	26.84	26.01	25.44	24.68	23.94
0.3267	0.0000	57.84	56.49	55.06	53.76	52.34	0.0871	0.4011	34.29	33.40	32.43	31.54	30.77
0.5860	0.4140	28.00	27.34	26.60	25.95	25.20	0.0732	0.2915	40.81	39.62	38.73	37.54	36.74
0.4554	0.2595	35.79	34.89	33.97	33.19	32.29	0.0599	0.2083	47.34	45.95	44.90	43.68	42.59
0.3692	0.1647	42.75	41.78	40.61	39.57	38.55	0.0549	0.1459	53.23	51.74	50.53	49.11	47.99
0.3198	0.0850	50.07	48.77	47.60	46.47	45.32	0.0457	0.0971	58.89	57.48	55.92	54.67	53.19
0.2755	0.0405	55.63	54.27	52.96	51.72	50.42	0.0393	0.0590	63.85	62.27	60.67	59.15	57.48
0.2432	0.0000	61.32	59.99	58.41	57.04	55.73	0.0366	0.0265	68.56	66.95	65.46	63.72	62.09
0.4855	0.5145	25.50	24.88	24.21	23.54	22.96	0.0346	0.0000	73.55	71.63	70.03	68.25	66.56
0.3668	0.3273	33.86	32.93	32.06	31.27	30.41	0.0000	1.0000	16.54	16.17	15.76	15.38	15.02
0.3038	0.2193	40.73	39.74	38.75	37.76	36.81	0.0000	0.6756	23.52	22.89	29.21	21.52	20.77
0.2583	0.1388	47.43	46.23	44.95	43.81	42.80	0.0000	0.4792	32.18	31.39	30.60	29.68	28.81
0.2220	0.0816	53.55	52.24	50.97	49.76	48.45	0.0000	0.3494	38.08	37.26	36.22	35.16	34.23
0.1956	0.0360	59.41	58.09	56.52	55.19	53.79	0.0000	0.2564	44.76	43.69	42.42	41.35	40.22
0.1775	0.0000	64.93	63.39	61.92	60.31	58.97	0.0000	0.1865	50.81	49.40	48.12	46.68	45.36
0.3776	0.6224	23.00	22.22	21.69	20.99	20.45	0.0000	0.1327	56.96	55.42	54.20	52.77	51.56
0.2889	0.4101	31.16	30.37	29.60	28.79	27.94	0.0000	0.0892	59.42	57.65	55.84		
0.2367	0.2797	38.37	37.34	36.35	35.53	34.50	0.0000	0.0545	64.67				
0.1970	0.1873	45.24	44.07	43.05	41.95	40.77	0.0000	0.0249	70.76	68.86	67.01	65.25	63.55
0.1694	0.1189	51.86	50.44	49.27	48.06	46.84	0.0000	0.0000	76.56	74.80	73.23	71.46	70.00

Table II. Coefficients α_i and Standard Deviations $\sigma(\ln \epsilon)$ of Equation 1 for Ethane-1,2-diol (1) + 2-Methoxyethanol (2) + Water (3)

x_1	x_2	α_0	$10^3\,\alpha_1$	$10^3\,\sigma(\ln \epsilon)$	x_1	x_2	α_0	$10^3\,\alpha_1$	$10^3\,\sigma(\ln \epsilon)$
1.0000	0.0000	5.231 86	−5.095 18	0.92	0.1513	0.0705	5.555 17	−4.964 24	0.70
0.9272	0.0728	5.174 25	−5.114 94	2.5	0.1342	0.0312	5.602 22	−4.821 23	1.1
0.7442	0.0000	5.345 10	−5.079 53	1.3	0.1213	0.0000	5.644 78	−4.682 47	0.90
0.8499	0.1501	5.130 00	−5.180 54	1.4	0.2614	0.7386	4.801 51	−5.868 11	1.1
0.6760	0.0601	5.323 07	−5.176 55	1.4	0.1916	0.4634	5.122 23	−5.668 05	0.89
0.5638	0.0000	5.441 29	−5.070 30	1.8	0.1618	0.3411	5.225 15	−5.395 38	0.68
0.7676	0.2324	5.094 74	−5.295 65	2.4	0.1362	0.2394	5.262 62	−4.976 40	1.9
0.6053	0.1243	5.267 64	−5.176 23	1.5	0.1208	0.1635	5.473 63	−5.190 71	1.1
0.4952	0.0589	5.399 22	−5.111 09	1.2	0.1018	0.1071	5.578 21	−5.150 56	1.6
0.4274	0.0000	5.510 89	−5.022 92	1.5	0.0959	0.0634	5.623 40	−5.000 97	0.43
0.6798	0.3202	5.048 67	−5.389 67	1.2	0.0822	0.0285	5.783 70	−5.278 45	1.3
0.5296	0.1903	5.209 77	−5.177 81	0.51	0.0749	0.0000	5.728 83	−4.850 50	1.2
0.4422	0.1044	5.368 04	−5.144 67	1.5	0.1359	0.8641	4.502 16	−5.201 67	3.3
0.3756	0.0447	5.513 23	−5.169 10	0.46	0.1138	0.5655	4.992 07	−5.616 30	2.6
0.3267	0.0000	5.571 22	−4.990 32	0.88	0.0871	0.4011	5.192 42	−5.468 26	1.3
0.5860	0.4140	4.925 83	−5.252 93	1.7	0.0732	0.2915	5.309 11	−5.282 81	2.6
0.4554	0.2595	5.127 68	−5.113 33	1.1	0.0599	0.2083	5.444 69	−5.240 81	1.6
0.3692	0.1647	5.340 19	−5.223 77	1.3	0.0549	0.1459	5.546 50	−5.188 54	1.6
0.3198	0.0850	5.414 66	−4.954 50	0.64	0.0457	0.0971	5.615 12	−5.077 14	1.3
0.2755	0.0405	5.504 85	−4.902 31	0.42	0.0393	0.0590	5.743 31	−5.231 75	1.2
0.2432	0.0000	5.581 25	−4.831 08	1.2	0.0366	0.0265	5.730 99	−4.955 29	1.5
0.4855	0.5145	4.847 80	−5.305 54	1.0	0.0346	0.0000	5.800 44	−4.957 67	0.99
0.3668	0.3273	5.139 60	−5.336 92	0.87	0.0000	1.0000	4.277 97	−4.854 66	0.66
0.3038	0.2193	5.245 95	−5.075 04	0.44	0.0000	0.6756	5.042 69	−6.208 92	2.6
0.2583	0.1388	5.430 26	−5.183 76	1.4	0.0000	0.4792	5.155 88	−5.549 68	2.4
0.2220	0.0816	5.488 53	−4.973 40	0.82	0.0000	0.3494	5.286 46	−5.423 72	2.5
0.1956	0.0360	5.598 58	−4.991 73	1.2	0.0000	0.2564	5.434 43	−5.383 93	1.3
0.1775	0.0000	5.644 28	−4.851 54	0.93	0.0000	0.1865	5.647 79	−5.670 91	1.1
0.3776	0.6224	4.902 62	−5.836 13	2.6	0.0000	0.1327	5.547 66	−4.968 63	1.4
0.2889	0.4101	5.085 66	−5.427 22	1.6	0.0000	0.0892	5.966 53	−6.207 08	0.62
0.2367	0.2797	5.238 69	−5.250 90	1.5	0.0000	0.0545			
0.1970	0.1873	5.371 50	−5.143 07	1.4	0.0000	0.0249	5.888 64	−5.375 66	0.36
0.1694	0.1189	5.474 83	−5.038 03	1.1	0.0000	0.0000	5.699 88	−4.493 23	0.96

Results and Discussion

Table I shows the ternary composition (mole fraction) and the experimental ϵ values measured for 66 mixtures at 5 different temperatures ranging from 30 to 50 °C, at 5 °C intervals. It should be noted that Table I is lacking of some ϵ values because the high conductivity of the corresponding

Table III. Coefficients and Average Deviations ($\overline{\Delta\epsilon}$) of Equation 4 for Ethane-1,2-diol + 2-Methoxyethanol + Water

t/°C	$a_1 \times 10$	$a_2 \times 10$	$b_1 \times 10$	$b_2 \times 10$	$\overline{\Delta\epsilon}$
30	5.342 27	3.827 33	1.443 27	1.371 06	0.28
35	5.419 68	3.842 57	1.472 79	1.396 25	0.25
40	5.447 30	3.922 71	1.492 37	1.438 08	0.27
45	5.542 16	3.821 33	1.528 49	1.430 61	0.23
50	5.617 66	3.904 76	1.561 93	1.477 69	0.26

ME + W binary mixtures in the rich water region.

The dependence of ϵ on the temperature has been checked by the equation

$$\ln \epsilon = \alpha_0 + \alpha_1(T/\mathrm{K}) \tag{1}$$

whose α_i coefficients are listed in Table II. This equation reproduces the experimental values within an average deviation, evaluated as following:

$$\overline{\Delta\epsilon} = (1/N)\sum_{N}|\epsilon_{\mathrm{calcd}} - \epsilon_{\mathrm{exptl}}| \tag{2}$$

where N is the number of experimental points; $\overline{\Delta\epsilon} = \pm 0.03$.

In an attempt to establish the dependence $\epsilon = \epsilon(x)$ for binary mixtures, King and Queen (*11*) employed a rational function like this one:

$$\ln \epsilon = \frac{1 + a_1 x_1}{(\ln \epsilon_2)^{-1} + b_1 x_1} \tag{3}$$

which is known as one form of the reciprocal Padé approximant (*11*), the constants $a_0 = 1$ and $b_0 = (\ln\epsilon_2)^{-1}$ being employed. It is noteworthy that eq 3 needs only two adjustable parameters, i.e., a_1 and b_1, to fit any set of binary data. Now, the use of eq 3 has been extended to this ternary solvent system in the form

$$\ln \epsilon = \frac{1 + a_1 x_1 + a_2 x_2}{\left(\sum_{i=1}^{3} x_i \ln \epsilon_i\right)^{-1} + b_1 x_1 + b_2 x_2} \tag{4}$$

where ϵ_i are the dielectric constants of the pure species, and the other symbols have their usual significance. The isothermal fitting coefficients a_i and b_i of eq 4 are summarized in Table III, along with the average uncertainty $\overline{\Delta\epsilon}$ at each temperature. The overall average deviation $\overline{\Delta\epsilon}$ for this correlation procedure is equal to ±0.26. Figure 1 shows the plot of eq 4 for ϵ values at 40 °C.

In order to quicken the interpolation procedures, eq 1 and 4 could be combined to provide a general relation of the type $\epsilon = \epsilon(T,x_1,x_2,x_3)$ and that may be explicated in the form

$$\ln \epsilon = \frac{\sum_{h=0}^{1}\sum_{k=0}^{1}\sum_{l=0}^{1}\gamma_{hkl}(T/K)^h x_1^k x_2^l}{\left(\sum_{i=1}^{3} x_i \ln \epsilon_i\right)^{-1} + \delta_1 x_1 + \delta_2 x_2} \quad \text{with } k + l = 0, 1 \tag{5}$$

This equation, whose coefficients are listed in Table IV, fits the experimental ϵ values of Table I within $\overline{\Delta\epsilon} = \pm 0.24$.

Kolling (*12*) suggested a suitable relation to evaluate the deviation from a mole fraction average of the dielectric constant ($\delta\epsilon$) for a binary mixture. On the basis of those argumentations, the equation has now been extended to the

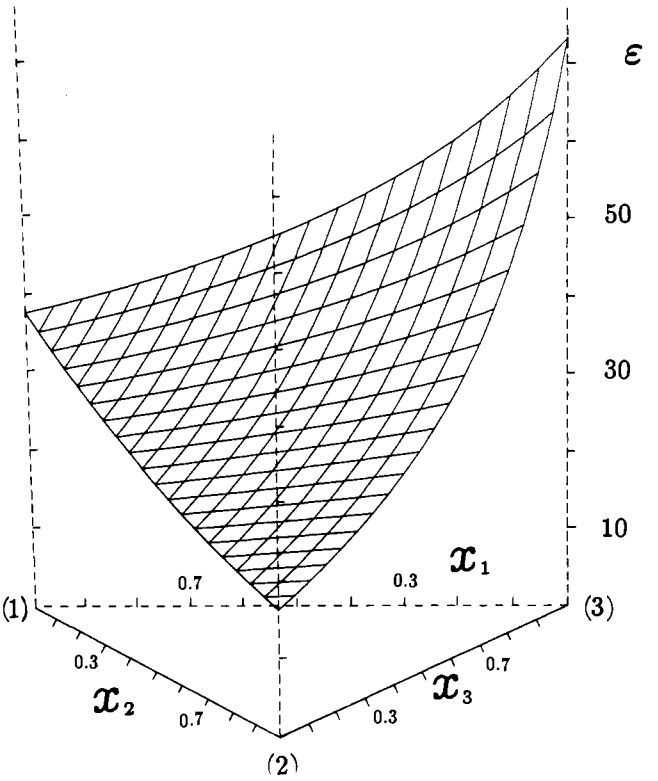

Figure 1. Computer-generated ϵ–x_i surface for the ED (1) + ME (2) + W (3) ternary solvent system at 40 °C.

Table IV. Coefficients of Equation 5 for Ethane-1,2-diol (1) + 2-Methoxyethanol (2) + Water (3)

symbol	variable quantity	coefficient	symbol	variable quantity	coefficient
γ_{000}		1.018 58	γ_{110}	Tx_1/K	$-6.375\,63 \times 10^{-4}$
γ_{010}	x_1	$6.824\,82 \times 10^{-1}$	γ_{101}	Tx_2/K	$-1.082\,52 \times 10^{-3}$
γ_{001}	x_2	$6.968\,94 \times 10^{-1}$	δ_1	x_1	$1.311\,30 \times 10^{-1}$
γ_{100}	T/K	$-7.047\,15 \times 10^{-5}$	δ_2	x_2	$1.307\,75 \times 10^{-1}$

ternary mixtures of this work in the form

$$\delta\epsilon = \epsilon - \sum_{i=1}^{3} x_i \epsilon_i \tag{6}$$

In the literature it has been suggested that for $\delta\epsilon > |5|$ we are in the presence of strong interactions, of any kind, which take place between components. In the case of this ternary solvent system and taking into account the molecular properties of the selected components, it is probable that the hydrogen bond formation could be the prevailing interaction pattern between like and/or unlike species. For the ternaries investigated, $\delta\epsilon$ is always negative and ranges from −0.81 to −17.48 in the limits of the investigated temperatures.

In order to confirm the presence in these mixtures of three-component stable adducts, we have followed the suggestions provided by other researchers (*13*, *14*), and the $\delta\epsilon$ values were isothermally fitted to an equation of the type

$$\delta\epsilon = d_0 x_1 x_2 + d_1 x_2 x_3 + d_2 x_1 x_3 + d_3 x_1 x_2 (x_2 - x_1) + d_4 x_2 x_3 (x_3 - x_2) + d_5 x_1 x_3 (x_3 - x_1) + d_6 x_1 x_2 x_3 \tag{7}$$

whose adjustable coefficients d_i are listed in Table V. A similar equation, derived from the Redlich–Kister one (*15*), was used by Pedrosa et *al.* (*16*) to fit other thermomechanical excess properties, and on this work we have extended its use to the $\delta\epsilon$ quantity too. It should be noted that eq 7 mainly represents the $\delta\epsilon$ quantity for a ternary system as a sum of contributions

Table V. Coefficients d_i and Standard Deviations $\sigma(\delta\epsilon)$ of Equation 7 for Ethane-1,2-diol + 2-Methoxyethanol + Water

t/°C	d_0	d_1	d_2	d_3	d_4	d_5	d_6	$10\sigma(\delta\epsilon)$
30	−10.39	−66.03	−27.79	−0.07	−46.64	−10.04	42.57	7.8
35	−10.32	−64.96	−27.19	−1.11	−45.41	−11.27	40.11	7.3
40	−10.30	−64.19	−27.23	−0.62	−45.12	−10.55	42.41	7.8
45	−10.32	−63.60	−26.86	−1.25	−41.35	−12.17	41.72	6.0
50	−10.30	−63.01	−26.79	−0.66	−40.67	−12.28	43.30	6.5

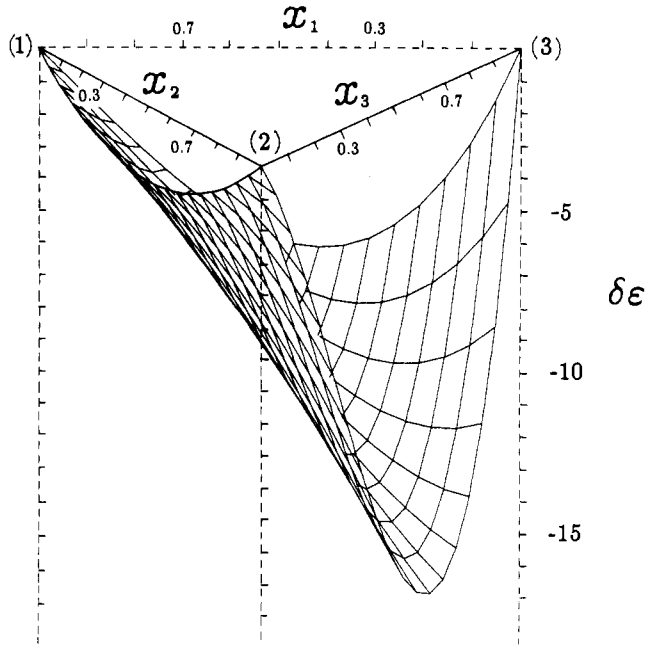

Figure 2. Pictorial view of the $\delta\epsilon$–composition (x_i) surface for the ED (1) + ME (2) + W (3) ternary solvent system at 40 °C.

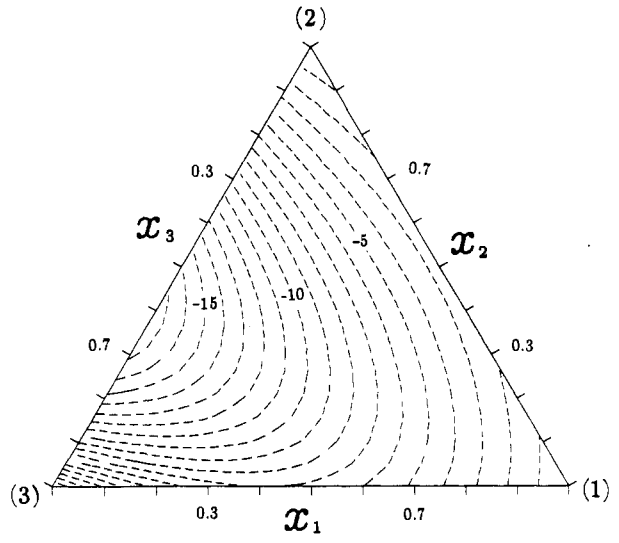

Figure 3. Computer-generated contour diagram showing lines of constant $\delta\epsilon$ on a liquid mole fraction grid for ED (1) + ME (2) + W (3) at 40 °C.

relative to the three binary subsystems, the overall ternary mixing effects being contained in the d_6 term only. Equation 7 reproduces the experimental data to within $\Delta(\delta\epsilon) = \pm 0.41$.

Furthermore, the $\delta\epsilon$ function has been plotted, at each investigated temperature, in the ternary domain $[x_1, x_2, x_3]$, and the results at 40 °C are displayed in Figures 2 and 3. A careful examination of these figures shows that no stable three-component adducts are formed in these nonelectrolytic solutions, any relative minimum in the ternary domain being absent. However, as one can see from Figure 4, it is possible

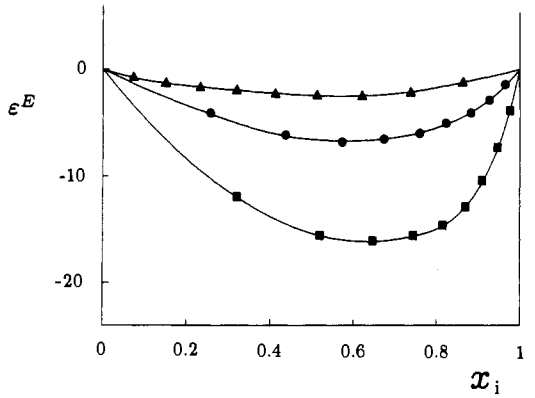

Figure 4. Trend of ϵ^E vs the mole fraction x_i of binary systems at 40 °C: (Δ) ED + ME vs x_{ME}; (●) ED + W vs x_W; (■) ME + W vs x_W.

to deduce that probably binary adducts may be formed through specific solvent–cosolvent interactions by hydrogen bondings or dipolar interactions; in particular the most stable moiety should be constituted by ME and W, at the stoichiometric ratio ME:W = 1:2 at all the investigated temperatures.

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