Excess Molar Volumes and Viscosities of Binary Mixtures of *p*-Cresol with Ethylene Glycol and Methanol at Different Temperature and Atmospheric Pressure

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Densities and viscosities of the binary mixtures of *p*-cresol + ethylene glycol and *p*-cresol + methanol mixtures have been measured at different temperatures and atmospheric pressure, over the entire composition range. From these data, excess molar volumes and viscosity deviation were calculated for each of the systems. The densities and viscosities of pure components have been compared with those reported by other authors available in the literature. The excess molar volumes, V^E, and viscosity deviations, $\Delta\eta$, were fitted to a Redlich−Kister equation, and the coefficients of the Redlich−Kister equation and estimate the standard error are also presented.

Introduction

Hydroxy-substituted benzaldehydes are important intermediates for a large variety of chemical products used in consumables such as soaps, fragrances, pharmaceuticals, preservatives, plant protection chemicals, etc.[1] Hydroxy-substituted benzaldehydes can be produced either from phenols or from cresols.[2] Despite the disadvantage of the higher cost of a cresol feed as opposed to a phenol feed, there has been considerable interest in the use of oxidative technologies for the production of hydroxybenzaldehydes in view of the potential advantages in respect of selectivity and lower viscosity. The current oxidation route is oxidation of *p*-cresol to *p*-hydroxybenzaldehyde with molecular oxygen using methanol as solvent. Methanol was found to have high substrate conversions and high selectivity to the desired aldehyde. However, because methanol is a toxic, flammable liquid and has a low flash point and a wide flammability limit range, it remains inherently unsafe to use.

Ethylene glycol is a much safer solvent to use than methanol, being described as merely harmful (as opposed to toxic), having a much higher flash point and a lower flammability limit range relative to methanol. So ethylene glycol is considered as an alternative solvent in the reaction system.

The catalytic oxidation of cresol is a heterogeneous reaction. The mass transfer might be the rate-determined step, and the diffusion coefficient is the main factor for conversion and selectivity. So transport properties such as viscosity are required for the reaction as well as design of industrial process. On the other hand, experimental data of physical properties such as density and viscosity in binary mixtures give information on the existence of specific molecular interactions, but such data especially for the binary mixtures of the *p*-cresol with ethylene and methanol are scarce in the literature.

In this work, the densities and viscosities of *p*-cresol + ethylene glycol and methanol mixtures were measured at (308.15 to 353.15) K and atmospheric pressure. The results were used to calculate excess molar volumes and viscosity deviations over the entire mole fraction range. Experimental values were fitted by the Redlich−Kister equation. The standard deviations between the experimental data and values calculated from the Redilch−Kister equation are also presented.

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Table 1. Comparison of Experimental and Literature Values of Densities, ρ, and Viscosities, η, for Pure Compounds

		ρ/g·cm^{-3}		η/mPa·s	
liquid	*T*/K	exptl	lit	exptl	lit
p-cresol	308.15	1.02198		8.444	
	313.15	1.01805	1.0168[3]	6.745	6.661[3]
	318.15	1.01409		5.471	
	323.15	1.01012	1.0092[3]	4.532	4.480[3]
	333.15	1.00209	1.0017[3]	3.245	3.232[3]
	343.15	0.99207		2.431	
	353.15	0.98624		1.891	
ethylene glycol	308.15	1.10188	1.1031[5]	11.193	11.0505[5]
	313.15	1.09833	1.0936[4]	9.4444	9.443[4]
			1.0997[5]		9.2442[5]
					9.409[6]
	318.15	1.09475	1.0967[5]	7.972	7.9694[5]
	323.15	1.09116	1.09116[4]	6.794	6.992[4]
			1.0934[5]		6.7128[5]
	333.15	1.08394	1.0764[4]	5.065	5.060[4]
					5.076[6]
	343.15	1.07453	1.0676[4]	3.879	3.987[4]
	353.15	1.06972	1.0600[4]	3.058	3.021[4]
					3.078[6]
methanol	308.15	0.77692	0.77703[8]	0.479	0.476[8]
	313.15	0.77213	0.77201[7]	0.445	0.447[7]
			0.77236[8]		0.448[8]
	318.15	0.76731		0.421	
	323.15	0.76246		0.395	
	328.15	0.75785		0.372	

Experimental Section

Materials. The chemicals employed were of analytical grade and were purchased from Tianjin Reagent Company. The mass fraction purities, tested by gas chromatography, were *p*-cresol (> 0.992), ethylene glycol (> 0.994), and methanol (> 0.995). All chemicals were used without further purification. They were degassed ultrasonically and dried over 0.4 Å molecular sieves.

Apparatus and Procedure. The densities of the pure components and their mixtures were measured with a high precision vibrating-tube digital density meter (Density/Specific Gravity

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Table 2. Densities ρ, Viscosities η, Excess Molar Volumes V^E, and Viscosity Deviations $\Delta\eta$ for the Binary Mixtures at Different Temperatures

x_1	ρ / g·cm^{-3}	η / mPa·s	V^E / cm^3·mol^{-1}	$\Delta\eta$ / mPa·s	x_1	ρ / g·cm^{-3}	η / mPa·s	V^E / cm^3·mol^{-1}	$\Delta\eta$ / mPa·s	x_1	ρ / g·cm^{-3}	η / mPa·s	V^E / cm^3·mol^{-1}	$\Delta\eta$ / mPa·s
(x) *p*-Cresol + (1 − x_1) Ethylene Glycol														
T = 308.15 K														
0.0000	1.10188	11.193	0.000	0.000	0.4001	1.05844	12.092	−0.071	1.999	0.8000	1.03162	9.310	−0.024	0.316
0.1001	1.08889	12.152	−0.046	1.234	0.4998	1.05066	11.540	−0.070	1.721	0.8994	1.02655	8.726	−0.008	0.005
0.1999	1.07745	12.427	−0.067	1.784	0.6002	1.04369	10.827	−0.066	1.284	1.0000	1.02198	8.444	0.000	0.000
0.2997	1.06732	12.434	−0.070	2.065	0.7000	1.03734	10.015	−0.045	0.747					
T = 313.15 K														
0.0000	1.09833	9.444	0.000	0.000	0.4001	1.05458	9.821	−0.066	1.457	0.8000	1.02772	7.526	−0.023	0.242
0.1001	1.08520	9.956	−0.043	0.782	0.4998	1.04678	9.353	−0.065	1.258	0.8994	1.02263	7.022	−0.007	0.005
0.1999	1.07368	10.185	−0.062	1.281	0.6002	1.03982	8.735	−0.063	0.911	1.0000	1.01805	6.745	0.000	0.000
0.2997	1.06352	10.134	−0.066	1.499	0.7000	1.03345	8.103	−0.044	0.548					
T = 318.15 K														
0.0000	1.09475	7.972	0.000	0.000	0.4001	1.05070	8.059	−0.061	1.088	0.8000	1.02379	6.177	−0.022	0.206
0.1001	1.08149	8.319	−0.040	0.598	0.4998	1.04288	7.662	−0.061	0.940	0.8994	1.01870	5.739	−0.008	0.016
0.1999	1.06989	8.436	−0.058	0.964	0.6002	1.03590	7.175	−0.060	0.704	1.0000	1.01409	5.471	0.000	0.000
0.2997	1.05967	8.358	−0.060	1.136	0.7000	1.02955	6.646	−0.044	0.425					
T = 323.15 K														
0.0000	1.09116	6.794	0.000	0.000	0.4001	1.04681	6.721	−0.056	0.832	0.8000	1.01985	5.136	−0.022	0.151
0.1001	1.07777	7.031	−0.037	0.464	0.4998	1.03898	6.378	−0.057	0.714	0.8994	1.01475	4.788	−0.008	0.028
0.1999	1.06609	7.069	−0.053	0.727	0.6002	1.03199	5.971	−0.058	0.535	1.0000	1.01012	4.532	0.000	0.000
0.2997	1.05581	6.976	−0.055	0.860	0.7000	1.02561	5.523	−0.041	0.313					
T = 333.15 K														
0.0000	1.08394	5.065	0.000	0.000	0.4001	1.03898	4.824	−0.047	0.487	0.8000	1.01192	3.711	−0.024	0.102
0.1001	1.07028	5.121	−0.031	0.239	0.4998	1.03111	4.607	−0.050	0.452	0.8994	1.00680	3.461	−0.013	0.033
0.1999	1.05844	5.137	−0.045	0.436	0.6002	1.02409	4.298	−0.054	0.326	1.0000	1.00209	3.245	0.000	0.000
0.2997	1.04806	5.029	−0.046	0.510	0.7000	1.01771	3.988	−0.041	0.197					
T = 343.15 K														
0.0000	1.07453	3.879	0.000	0.000	0.4001	1.02909	3.602	−0.039	0.302	0.8000	1.00199	2.786	−0.027	0.066
0.1001	1.06064	3.886	−0.025	0.152	0.4998	1.02120	3.424	−0.045	0.269	0.8994	0.99684	2.588	−0.016	0.011
0.1999	1.04868	3.849	−0.037	0.260	0.6002	1.01417	3.201	−0.051	0.192	1.0000	0.99207	2.431	0.000	0.000
0.2997	1.03824	3.752	−0.039	0.307	0.7000	1.00779	2.995	−0.043	0.129					
T = 353.15 K														
0.0000	1.06972	3.058	0.000	0.000	0.4001	1.02359	2.776	−0.032	0.185	0.8000	0.99633	2.173	−0.034	0.049
0.1001	1.05558	2.934	−0.022	−0.008	0.4998	1.01562	2.648	−0.039	0.174	0.8994	0.99110	2.022	−0.021	0.014
0.1999	1.04336	2.990	−0.027	0.166	0.6002	1.00855	2.488	−0.049	0.131	1.0000	0.98624	1.891	0.000	0.000
0.2997	1.03281	2.915	−0.030	0.206	0.7000	1.00215	2.326	−0.045	0.085					
(x) *p*-Cresol + (1 − x$_1$) Methanol														
T = 308.15 K														
0.0000	0.77692	0.476	0.000	0.000	0.4000	0.94054	1.762	−0.634	−1.902	0.8000	1.00353	5.072	−0.304	−1.779
0.1000	0.83727	0.701	−0.338	−0.572	0.4999	0.96159	2.316	−0.634	−2.143	0.8999	1.01373	6.535	−0.188	−1.112
0.1999	0.88121	0.977	−0.521	−1.092	0.5999	0.97795	3.021	−0.530	−2.235	1.0000	1.02198	8.444	0.000	0.000
0.2998	0.91453	1.333	−0.612	−1.532	0.6999	0.99204	3.928	−0.445	−2.124					
T = 313.15 K														
0.0000	0.77213	0.445	0.000	0.000	0.4000	0.93635	1.588	−0.654	−1.377	0.8000	0.99957	4.251	−0.315	−1.234
0.1000	0.83269	0.652	−0.348	−0.423	0.4999	0.95748	2.044	−0.653	−1.550	0.8999	1.00979	5.364	−0.195	−0.750
0.1999	0.87678	0.901	−0.537	−0.803	0.5999	0.97390	2.631	−0.547	−1.593	1.0000	1.01805	6.745	0.000	0.000
0.2998	0.91025	1.218	−0.632	−1.116	0.6999	0.98804	3.360	−0.460	−1.494					
T = 318.15 K														
0.0000	0.76731	0.421	0.000	0.000	0.4000	0.93216	1.435	−0.676	−1.006	0.8000	0.99558	3.603	−0.326	−0.858
0.1000	0.82809	0.608	−0.360	−0.318	0.4999	0.95335	1.817	−0.674	−1.128	0.8999	1.00583	4.440	−0.202	−0.526
0.1999	0.87235	0.834	−0.554	−0.597	0.5999	0.96984	2.310	−0.566	−1.140	1.0000	1.01409	5.471	0.000	0.000
0.2998	0.90593	1.112	−0.651	−0.823	0.6999	0.98403	2.894	−0.476	−1.061					
T = 323.15 K														
0.0000	0.76246	0.395	0.000	0.000	0.4000	0.92793	1.305	−0.698	−0.745	0.8000	0.99158	3.092	−0.338	−0.612
0.1000	0.82345	0.568	−0.371	−0.241	0.4999	0.94921	1.633	−0.695	−0.830	0.8999	1.00184	3.754	−0.208	−0.363
0.1999	0.86789	0.772	−0.572	−0.450	0.5999	0.96576	2.043	−0.585	−0.833	1.0000	1.01012	4.532	0.000	0.000
0.2998	0.90161	0.998	−0.673	−0.637	0.6999	0.98000	2.527	−0.493	−0.764					
T = 328.15 K														
0.0000	0.75785	0.372	0.000	0.000	0.4000	0.92403	1.192	−0.718	−0.586	0.8000	0.98791	2.688	−0.343	−0.497
0.1000	0.81909	0.533	−0.382	−0.191	0.4999	0.94539	1.477	−0.713	−0.653	0.8999	0.99820	3.212	−0.207	−0.324
0.1999	0.86371	0.719	−0.589	−0.356	0.5999	0.96201	1.821	−0.600	−0.660	1.0000	1.00657	3.888	0.000	0.000
0.2998	0.89758	0.942	−0.692	−0.484	0.6999	0.97630	2.229	−0.504	−0.604					

Meter DA 505, KEM, Japan) whose measurement cell temperature was controlled automatically within ± 0.01 K of the selected value. Before each series of measurements, the instrument was calibrated at atmospheric pressure with double-distilled water and dry air. Densities of both water and dry air at the various working temperatures were given by the manufacturer in the instruction manual. The calibration was accepted if the measurements were within $\pm 5 \times 10^{-5}$ g·cm^{-3} of the published values. The uncertainty in density measurements was $\pm 5 \times 10^{-5}$ g·cm^{-3}. Density measurements were reproducible to $\pm 3 \times 10^{-5}$ g·cm^{-3}.

The liquid mixtures were prepared by mass using a BP210s balance accurate to within ± 0.01 mg. The average uncertainty in the mole fraction of the mixtures was estimated to be less than ± 0.0001. The molar excess volumes were calculated from composition−density data with an uncertainty better than ± 0.002 cm^3·mol^{-1}. All molar quantities were based on the IUPAC relative atomic mass table.

Table 3. Coefficients of the Redlich−Kister Equation and Standard Deviation for Excess Molar Volumes and Viscosity Deviations of Mixtures

T/K	property	A_0	A_1	A_2	A_3	A_4	σ
			(x) *p*-Cresol + (1 − x_1) Ethylene Glycol				
308.15	V^E/cm^3·mol^{-1}	−0.278	0.116	−0.027	0.245		0.0145
	$\Delta\eta$/mPa·s	6.759	−8.137				0.2871
313.15	V^E/cm^3·mol^{-1}	−0.262	0.093	−0.016	0.251		0.0140
	$\Delta\eta$/mPa·s	4.766	−5.444				0.2653
318.15	V^E/cm^3·mol^{-1}	−0.245	0.071	−0.027	0.243		0.0151
	$\Delta\eta$/mPa·s	3.642	−4.038				0.1576
323.15	V^E/cm^3·mol^{-1}	−0.229	0.052	−0.027	0.238		0.0168
	$\Delta\eta$/mPa·s	2.787	−3.050				0.0752
333.15	V^E/cm^3·mol^{-1}	−0.201	0.009	−0.053	0.194		0.0186
	$\Delta\eta$/mPa·s	1.660	−1.588				0.1450
343.15	V^E/cm^3·mol^{-1}	−0.182	−0.039	−0.065	0.168		0.0182
	$\Delta\eta$/mPa·s	1.006	−1.004				0.0684
353.15	V^E/cm^3·mol^{-1}	−0.159	−0.124	−0.107	0.212		0.0115
	$\Delta\eta$/mPa·s	0.534	−0.219				0.3814
			(x) *p*-Cresol + (1 − x_1) Methanol				
308.15	V^E/cm^3·mol^{-1}	−2.479	1.090	0.169	−0.057	−1.336	0.0631
	$\Delta\eta$/mPa·s	−8.546	−3.422	−1.229	−0.486		0.0351
313.15	V^E/cm^3·mol^{-1}	−2.556	1.113	0.157	−0.049	−1.359	0.0629
	$\Delta\eta$/mPa·s	−6.163	−2.231	−0.548	−0.053		0.0289
308.15	V^E/cm^3·mol^{-1}	−2.640	1.138	0.157	−0.042	−1.408	0.0638
	$\Delta\eta$/mPa·s	−4.458	−1.359	−0.322	−0.109		0.0394
323.15	V^E/cm^3·mol^{-1}	−2.725	1.163	0.120	−0.021	−1.372	0.0648
	$\Delta\eta$/mPa·s	−3.304	−0.798	−0.078	−0.082		0.0302
328.15	V^E/cm^3·mol^{-1}	−2.798	1.199	0.134	0.052	−1.361	0.0641
	$\Delta\eta$/mPa·s	−2.560	−0.621	−0.414	−0.452		0.0553

The viscosities of pure liquids and the mixtures were measured at atmospheric pressure and at different temperatures using several Ubbelohde suspended-level viscometers. The viscometer was immersed in a well-stirred water bath (Lauda, Germany) with temperature control to within ± 0.01 K. An electronic digital stopwatch with a readability of ± 0.01 s was used for flow time measurement. Experiments are repeated a minimum of four times at each temperature for all compositions, and the results were averaged. The viscosity η of the liquid was then calculated from the following relationship:

$$\nu = \frac{\eta}{\rho} = k(t - \theta) \tag{1}$$

where t is the flow time; ν is the kinematic viscosity; and k and θ are the viscometer constant and the Hagenbach correction factor, respectively.

According to the viscosity of the mixtures, two types of viscometers at different diameter and length were used. For the system of *p*-cresol + ethylene glycol, the viscometer with the capillaries (0.8 to 0.9) mm in diameter and 100 mm in length was used. For the system of *p*-cresol + methanol, one of the capillaries was (0.8 to 0.9) mm in diameter and 100 mm in length for mole fraction of *p*-cresol from (0.6 to 1.0); the another one was (0.2 to 0.3) mm in diameter and 120 mm in length for mole fraction from (0 to 0.5). The calibration of the viscometer was carried out with double-distilled water and standard oil (supplied by Chinese Standard Bureau). During the heating, to minimize the evaporation losses, the viscometer's limbs were closed with Teflon caps. During the measurement of flow times, the caps of the limbs were removed. The overall uncertainty of the viscosity measurements is dependent on the temperature control of the viscometer, the time of the flow, and the concentration, which are of the order of 1×10^{-2}, 1×10^{-2}, and 3×10^{-4}, respectively. The uncertainty of viscosity results was within ± 0.003 mPa·s.

In the experiments, the density and viscosity for one composition sample were measured at different temperatures. Densities and viscosities of pure compounds are reported in Table 1, together with the corresponding literature data.[3−8]

Results and Discussion

Excess volumes and viscosity deviations were calculated from our measurements according to the following equations:[9]

$$V^E = \frac{x_1M_1 + x_2M_2}{\rho} - \frac{x_1M_1}{\rho_1} - \frac{x_2M_2}{\rho_2} \tag{2}$$

where x_1 and x_2 are mole fractions; M_1 and M_2 are the molar masses; and ρ_1 and ρ_2 are the densities of pure components 1 and 2, respectively. Quantities without subscripts refer to the mixture.

The viscosity deviations were calculated from the following relation:[10,11]

$$\Delta\eta = \eta - (x_1\eta_1 + x_2\eta_2) \tag{3}$$

where η is the viscosity of mixtures and η_1 and η_2 are the viscosity of components 1 and 2, respectively. The values of V^E and $\Delta\eta$ for each mixture were fitted to the Redlich−Kister equation:[11]

$$Y = x_1(1 - x_1)\sum_{i=0}^{n} A_i(2x_1 - 1)^i \tag{4}$$

where Y = V^E or $\Delta\eta$, A_i is adjustable parameters, and x_1 is the fraction of component 1.

In each case, the optimum number of coefficients A_i was determined from an examination of the variation of the standard derivation:

$$\sigma(Y) = [\sum(Y_{cal} - Y_{exp})^2/(n - m)]^{(1/2)} \tag{5}$$

where n is the total number of experimental values and m is the number of parameters. The excess molar volume data and the viscosity deviations are presented in Table 2. Table 3 lists the values of the parameters A_i together with the standard deviations.

The plots of V^E against x_1 for the *p*-cresol with ethylene glycol and methanol are shown in Figure 1 and Figure 2,

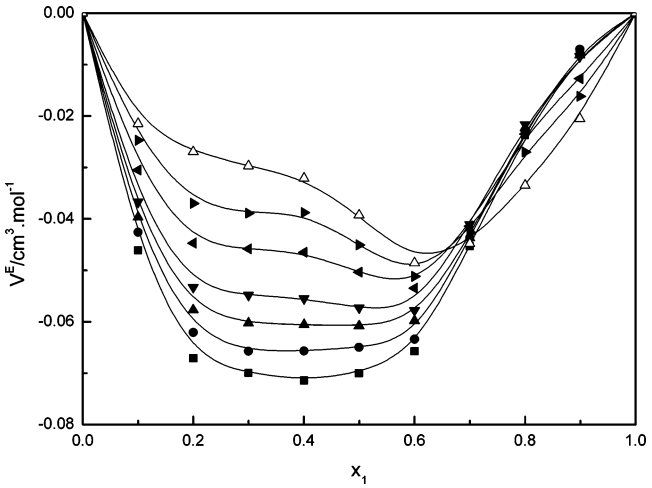

Figure 1. Excess volumes, V^E, vs the mole fraction, x_1, for system *p*-cresol (1) + ethylene glycol (2) at different temperatures *T*: ■, 308.15 K; ●, 313.15 K; ▲, 318.15 K; ▼, 323.15 K; right-facing solid triangle, 333.15 K; left-facing solid triangle, 343.15 K; △, 353.15 K; solid curves, calculated with Redlich−Kister equations; symbols, experimental values.

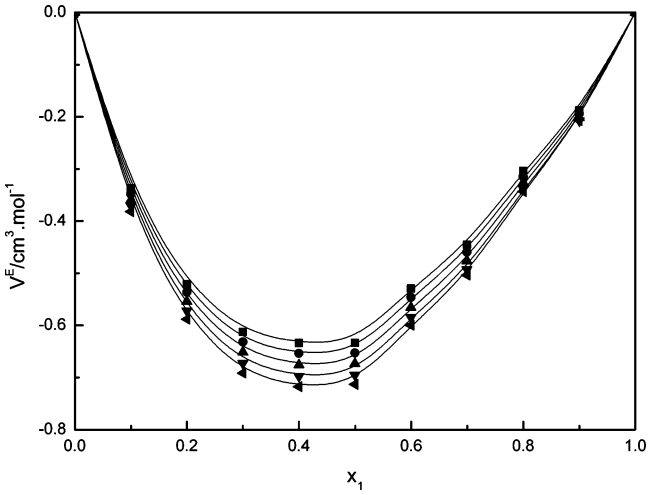

Figure 2. Excess volumes, V^E, vs the mole fraction, x_1, for system *p*-cresol (1) + methanol (2) at different temperatures *T*: ■, 308.15 K; ●, 313.15 K; ▲, 318.15 K; ▼, 323.15 K; right-facing solid triangle, 328.15 K; solid curves, calculated with Redlich−Kister equations; symbols, experimental values.

respectively. The values of the excess molar volumes are negative over the whole mole fraction range for these two binary systems. For the mixture of *p*-cresol + ethylene glycol, the excess molar volumes increase with increase in temperature, and all lines of excess molar volumes to composition intersect as x_1 is greater than 0.7. On the contrary, the excess molar volumes decrease with increasing temperature and the parabolic composition dependence is found for the system of *p*-cresol + methanol. The absolute values of excess molar volumes at the same composition for the mixture of *p*-cresol + methanol are greater than the values for the mixture of *p*-cresol + ethylene glycol mixture. To a certain degree, the excess molar volumes reflect the type of interactions taking place in the mixture. For the *p*-cresol + alcohol mixtures, the main factors that affects the excess volumes are the hydrogen bonding and the molecular sizes and shapes. Alcohol molecules are strongly self-associated through hydrogen bonding. The cresol with alcohol can also associate by hydrogen bonding. The interaction strengthen of hydrogen bonding for the system of *p*-cresol + methanol may be stronger than the interaction of *p*-cresol + ethylene glycol mixture. So the excess molar volumes in absolute values of

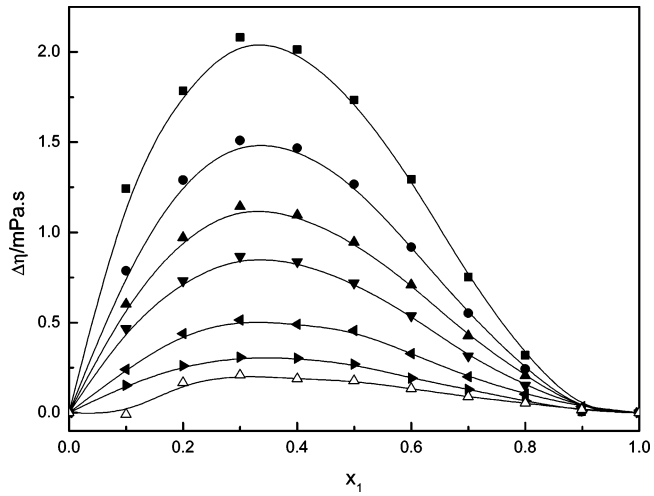

Figure 3. Deviation of viscosity, $\Delta\eta$, vs the mole fraction, x_1, for system *p*-cresol (1) + ethylene glycol (2) at different temperatures *T*: ■, 308.15 K; ●, 313.15 K; ▲, 318.15 K; ▼, 323.15 K; right-facing solid triangle, 333.15 K; left-facing solid triangle, 343.15 K; △, 353.15 K; solid curves, calculated with Redlich−Kister equations; symbols, experimental values.

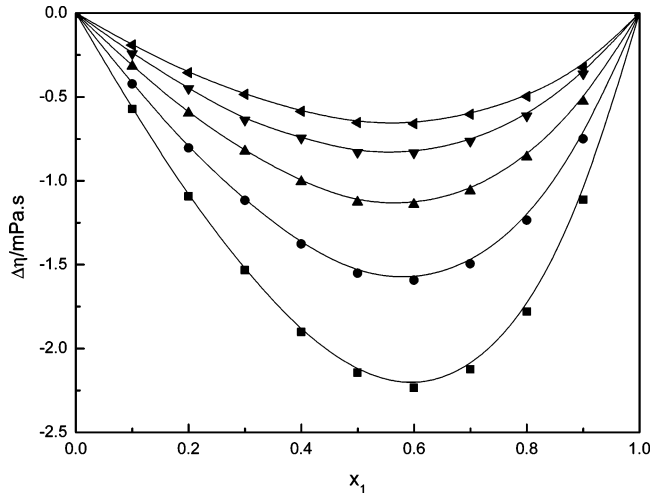

Figure 4. Deviation of viscosity, $\Delta\eta$, vs the mole fraction, x_1, for system for system *p*-cresol (1) + methanol (2) at different temperatures *T*: ■, 308.15 K; ●, 313.15 K; ▲, 318.15 K; ▼, 323.15 K; right-facing solid triangle, 328.15 K; solid curves, calculated with Redlich−Kister equations; symbols, experimental values.

p-cresol + methanol mixtures are larger than the values for *p*-cresol + ethylene glycol mixture.

Figure 3 shows viscosity deviations against mole fraction for the binary mixture *p*-cresol with ethylene glycol together with the fitted curve, obtained from the Redlich−Kister equation. It can be seen that the viscosity deviations for the system of *p*-cresol with ethylene glycol at selected temperatures are positive over the entire composition, and the curves are asymmetrical in nature and skewed to the ethylene glycol-rich range. The dependence of viscosity deviation on mole fraction for *p*-cresol with methanol is displayed in Figure 4. The viscosity deviations are negative at all mole fractions and temperatures and the curves skewed to the *p*-cresol-rich range. From the our previous research results,[10−12] it can be concluded that if one solution with higher viscosity is mixed with lower viscosity solution to form a binary mixture, usually the viscosity deviations are negative and the curve of the viscosity deviation on mole fraction is skewed to the higher viscosity solution.

Conclusion

Densities and viscosities for the *p*-cresol + (ethylene glycol, methanol) binary systems have been experimentally determined at (308.15 to 353.15) K and normal atmospheric pressure, over the entire mole fraction range. The excess molar volume and viscosity deviations were correlated using the Redlich−Kister equation. The excess molar volumes for these two binary systems were negative over the whole composition range and at all temperatures. The values of viscosity deviations for *p*-cresol + ethylene glycol were positive, and the values for *p*-cresol + methanol are negative.

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