Densities and Viscosities of *N*,*N*-Dimethylformamide + Formic Acid, and + Acetic Acid in the Temperature Range from (303.15 to 353.15) K

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Densities and viscosities for formic acid and acetic acid with dimethylformamide (DMF) were determined at atmospheric pressure from (303.15 to 353.15) K. The measurements were carried out over the whole range of compositions, using a vibrating-tube density meter and an Ubbelohde viscometer. The density and viscosity measurements were used to compute the excess molar volumes, V^E, and viscosity deviations, $\Delta\eta$. The excess molar volumes and viscosity deviations have been fit to the Redlich−Kister equation.

Introduction

N,*N*-Dimethylformamide (DMF) is a versatile solvent used in the synthesis of pharmaceuticals, in agricultural chemistry, and as a solvent for the preparation of polymers. As pure solvent, DMF has a large dipole moment and high dielectric constant. However, there are no proton donor groups in the molecule, and the hydrogen atom of the C−H group is able to provide only a weak hydrogen bond. DMF is associated to some extent by means of nonspecific dipole–dipole interactions due to the large dipole moment.[1] Nakabayashi et al.[2] have indicated that acetic acid−solvent complexes are also formed in the aprotic polar solvents DMF investigated by Raman spectra. Also, Umadevi et al.[3] investigated the structures of binary systems of acetic acid + *N*,*N*-dimethylformatide by Raman spectra.

A survey of the literature shows that there are no reports on the density and viscosity data of binary mixtures of DMF with formic acid and acetic acid. Studies on viscosity and density of binary mixtures along with other thermodynamic properties are being increasingly used as tools for the investigation of the properties of pure components and the nature of intermolecular interactions between the liquid mixtures. From the data of excess molar volumes and viscosity deviations, the information about molecular structure and interactions is in turn used to confirm the conclusion obtained from the Raman spectra. Therefore, in the present paper, we report densities and viscosities of these two binary mixtures over the entire range of compositions and in the temperature range (303.15 to 353.15) K. The results were used to calculate excess molar volumes and viscosity deviations. 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.

Experimental Section

Materials. The pure components were supplied by Tianjin Reagent Company. The components were degassed ultrasonically and dried over molecular sieves type 4 Å. The mass fraction purities, determined by gas chromatography, were as follows: DMF, > 0.998; formic acid, > 0.99; acetic acid, > 0.995. The densities and viscosities are compared with the literature values in Table 1.

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

		ρ/g·cm^{-3}		η/m·Pas	
liquid	T/K	exptl	lit.	exptl	lit.
DMF	298.15	0.94421	0.9445[6]	0.808	0.803[6]
			0.9442[7]		0.799[7]
			0.9439[8]		0.801[8]
			0.94387[9]		0.802[9]
	303.15	0.93945	0.9395[8]	0.760	0.756[6]
			0.9398[6]		0.754[8]
					0.752[10]
	313.15	0.92986	0.9302[6]	0.675	0.673[6]
			0.9298[11]		0.664[10]
formic acid	298.15	1.21135	1.2138[12]		
	303.15	1.20507	1.2075[12]	1.437	1.36[12]
	313.15	1.19281	1.1946[12]	1.205	1.15[12]
	298.15	1.04402	1.0440[12]		
acetic acid	303.15	1.03866	1.0383[12]	1.059	0.98[12]
			1.0380[13]		1.042[13]
	313.15	1.02737	1.0271[12]	0.920	0.86[12]
			1.0267[13]		0.907[13]

Apparatus and Procedure. The densities of the pure components and their mixtures were measured with a high accuracy vibrating-tube digital density meter. Before each series of measurements, the instrument was calibrated at atmospheric pressure with double distilled water and dry air. The uncertainty in density measurements was ± 5·10^{-5} g·cm^{-3}. Density measurements were reproducible to ± 3·10^{-5} g·cm^{-3}.

The liquid mixtures were prepared by mass using a BP210s balance reproducible to within ± 0.1 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.

The viscosities of pure liquids and the mixtures were measured at atmospheric pressure and at different temperatures using an Ubbelohde suspended-level viscometer which was calibrated with doubly distilled water. The uncertainty of the viscosity results was within ± 0.003 mPa·s.

The details of the methods and techniques used to determine densities and viscosities have been described

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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 / $g \cdot cm^{-3}$	η / mPa·s	V^E / $cm^3 \cdot mol^{-1}$	$\Delta\eta$ / mPa·s	x_1	ρ / $g \cdot cm^{-3}$	η / mPa·s	V^E / $cm^3 \cdot mol^{-1}$	$\Delta\eta$ / mPa·s
(x_1) Formic Acid + (1 − x_1) DMF									
T = 303.15 K									
0.0000	0.93945	0.760	0.0000	0.000	0.6003	1.05317	1.466	−0.0574	0.300
0.1001	0.95365	0.831	−0.0364	0.004	0.7005	1.08171	1.555	−0.0227	0.321
0.2000	0.96923	0.930	−0.0550	0.034	0.7998	1.11525	1.562	0.0063	0.261
0.3001	0.98666	1.048	−0.0715	0.085	0.9002	1.15702	1.533	−0.0365	0.163
0.4000	1.00616	1.186	−0.0769	0.155	1.0000	1.20507	1.437	0.0000	0.000
0.4999	1.02799	1.324	−0.0632	0.226					
T = 313.15 K									
0.0000	0.92986	0.675	0.0000	0.000	0.6003	1.04303	1.253	−0.0892	0.260
0.1001	0.94397	0.738	−0.0408	0.010	0.7005	1.07128	1.313	−0.0509	0.267
0.2000	0.95957	0.815	−0.0723	0.034	0.7998	1.10439	1.342	−0.0147	0.243
0.3001	0.97707	0.920	−0.1045	0.086	0.9002	1.14558	1.330	−0.0494	0.178
0.4000	0.99643	1.028	−0.1111	0.141	1.0000	1.19281	1.205	0.0000	0.000
0.4999	1.01812	1.143	−0.0990	0.203					
T = 323.15 K									
0.0000	0.92092	0.608	0.0000	0.000	0.6003	1.03304	1.076	−0.1060	0.220
0.1001	0.93486	0.662	−0.0408	0.012	0.7005	1.06099	1.121	−0.0684	0.223
0.2000	0.95034	0.725	−0.0777	0.034	0.7998	1.09373	1.140	−0.0316	0.201
0.3001	0.96777	0.808	−0.1191	0.076	0.9002	1.13405	1.117	−0.0515	0.137
0.4000	0.98686	0.898	−0.1221	0.125	1.0000	1.18063	1.022	0.0000	0.000
0.4999	1.00839	0.992	−0.1153	0.177					
T = 333.15 K									
0.0000	0.91064	0.552	0.0000	0.000	0.6003	1.02194	0.933	−0.1311	0.185
0.1001	0.92458	0.590	−0.0543	0.005	0.7005	1.04951	0.971	−0.0872	0.190
0.2000	0.93991	0.649	−0.0922	0.032	0.7998	1.08177	0.982	−0.0434	0.168
0.3001	0.95733	0.720	−0.1462	0.070	0.9002	1.12174	0.959	−0.0659	0.113
0.4000	0.97615	0.793	−0.1437	0.111	1.0000	1.16744	0.879	0.0000	0.000
0.4999	0.99750	0.867	−0.1386	0.151					
T = 343.15 K									
0.0000	0.90172	0.502	0.0000	0.000	0.6003	1.01192	0.823	−0.1528	0.163
0.1001	0.91536	0.538	−0.0446	0.010	0.7005	1.03913	0.850	−0.1076	0.164
0.2000	0.93070	0.587	−0.0999	0.032	0.7998	1.07096	0.857	−0.0623	0.144
0.3001	0.94793	0.643	−0.1566	0.062	0.9002	1.11003	0.835	−0.0695	0.095
0.4000	0.96658	0.708	−0.1589	0.100	1.0000	1.15499	0.766	0.0000	0.000
0.4999	0.98775	0.768	−0.1589	0.134					
T = 353.15 K									
0.0000	0.89144	0.460	0.0000	0.000	0.6003	1.00084	0.732	−0.1750	0.141
0.1001	0.90511	0.492	−0.0600	0.010	0.7005	1.02778	0.754	−0.1279	0.142
0.2000	0.92026	0.534	−0.1129	0.030	0.7998	1.05918	0.756	−0.0754	0.122
0.3001	0.93730	0.582	−0.1686	0.056	0.9002	1.09786	0.736	−0.0817	0.081
0.4000	0.95590	0.635	−0.1799	0.088	1.0000	1.14208	0.677	0.0000	0.000
0.4999	0.97691	0.693	−0.1823	0.125					
(x_1) Acetic Acid + (1 − x_1) DMF									
T = 303.15 K									
0.0000	0.93945	0.760	0.0000	0.000	0.6014	1.00774	1.613	−1.0359	0.673
0.1004	0.95010	0.847	−0.2440	0.057	0.7011	1.01812	1.656	−0.9801	0.686
0.2009	0.96118	0.967	−0.4702	0.147	0.8008	1.02713	1.582	−0.8039	0.583
0.3012	0.97265	1.108	−0.6752	0.258	0.9006	1.03447	1.375	−0.4997	0.346
0.4014	0.98449	1.283	−0.8551	0.403	1.0000	1.03866	1.059	0.0000	0.000
0.5014	0.99633	1.457	−0.9843	0.547					
T = 313.15 K									
0.0000	0.92986	0.675	0.0000	0.000	0.6014	0.99798	1.357	−1.1046	0.535
0.1004	0.94075	0.750	−0.2788	0.051	0.7011	1.00811	1.390	−1.0416	0.543
0.2009	0.95176	0.848	−0.5134	0.124	0.8008	1.01684	1.331	−0.8552	0.460
0.3012	0.96318	0.973	−0.7281	0.224	0.9006	1.02384	1.166	−0.5359	0.270
0.4014	0.97495	1.112	−0.9160	0.339	1.0000	1.02737	0.920	0.0000	0.000
0.5014	0.98668	1.249	−1.0496	0.451					
T = 323.15 K									
0.0000	0.92092	0.608	0.0000	0.000	0.6014	0.98811	1.157	−1.1462	0.431
0.1004	0.93127	0.669	−0.2537	0.042	0.7011	0.99802	1.180	−1.0837	0.435
0.2009	0.94227	0.752	−0.5079	0.104	0.8008	1.00649	1.132	−0.8937	0.368
0.3012	0.95363	0.850	−0.7379	0.183	0.9006	1.01313	1.002	−0.5633	0.217
0.4014	0.96533	0.965	−0.9398	0.278	1.0000	1.01608	0.804	0.0000	0.000
0.5014	0.97695	1.071	−1.0840	0.365					

Table 2. Continued

x_1	ρ / $g\cdot cm^{-3}$	η / $mPa\cdot s$	V^E / $cm^3\cdot mol^{-1}$	$\Delta\eta$ / $mPa\cdot s$	x_1	ρ / $g\cdot cm^{-3}$	η / $mPa\cdot s$	V^E / $cm^3\cdot mol^{-1}$	$\Delta\eta$ / $mPa\cdot s$
				T = 333.15 K					
0.0000	0.91064	0.552	0.0000	0.000	0.6014	0.97759	0.998	−1.1832	0.352
0.1004	0.92119	0.597	−0.2815	0.030	0.7011	0.98728	1.015	−1.1096	0.353
0.2009	0.93218	0.673	−0.5448	0.089	0.8008	0.99548	0.976	−0.9037	0.298
0.3012	0.94349	0.755	−0.7800	0.155	0.9006	1.00178	0.870	−0.5510	0.176
0.4014	0.95511	0.843	−0.9846	0.228	1.0000	1.00504	0.709	0.0000	0.000
0.5014	0.96655	0.935	−1.1232	0.304					
				T = 343.15 K					
0.0000	0.90172	0.502	0.0000	0.000	0.6014	0.96755	0.873	−1.2175	0.292
0.1004	0.91156	0.547	−0.2410	0.032	0.7011	0.97706	0.885	−1.1479	0.292
0.2009	0.92254	0.607	−0.5264	0.079	0.8008	0.98499	0.852	−0.9381	0.245
0.3012	0.93382	0.674	−0.7813	0.133	0.9006	0.99093	0.764	−0.5740	0.145
0.4014	0.94535	0.749	−1.0003	0.195	1.0000	0.99369	0.632	0.0000	0.000
0.5014	0.95668	0.824	−1.1509	0.256					
				T = 353.15 K					
0.0000	0.89144	0.460	0.0000	0.000	0.6014	0.95719	0.771	−1.2573	0.245
0.1004	0.90158	0.499	−0.2766	0.027	0.7011	0.96649	0.780	−1.1744	0.243
0.2009	0.91255	0.551	−0.5698	0.069	0.8008	0.97417	0.751	−0.9467	0.202
0.3012	0.92382	0.608	−0.8317	0.115	0.9006	0.97973	0.677	−0.5539	0.118
0.4014	0.93528	0.670	−1.0520	0.166	1.0000	0.98295	0.570	0.0000	0.000
0.5014	0.94648	0.730	−1.1983	0.215					

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_1) Formic Acid + (1 − x_1) DMF				
303.15	$V^E/cm^3\cdot mol^{-1}$	−0.3023	0.4613	0.9688	−0.6788	−1.7482	0.0717
	$\Delta\eta/mPa\cdot s$	0.937	1.431	0.061	−0.523	−0.129	0.0468
313.15	$V^E/cm^3\cdot mol^{-1}$	−0.4450	0.5051	0.9878	−0.8406	−1.6616	0.0732
	$\Delta\eta/mPa\cdot s$	0.833	1.068	−0.146	0.139	0.738	0.0319
323.15	$V^E/cm^3\cdot mol^{-1}$	−0.5023	0.4464	0.8175	−0.7800	−1.2845	0.0655
	$\Delta\eta/mPa\cdot s$	0.716	0.905	−0.095	−0.076	0.422	0.0150
333.15	$V^E/cm^3\cdot mol^{-1}$	−0.6074	0.4850	0.9670	−0.8485	−1.6315	0.0834
	$\Delta\eta/mPa\cdot s$	0.612	0.709	0.014	0.051	0.083	0.0123
343.15	$V^E/cm^3\cdot mol^{-1}$	−0.6764	0.4436	0.7508	−0.9236	−1.0497	0.0773
	$\Delta\eta/mPa\cdot s$	0.543	0.601	−0.048	−0.015	0.174	0.0116
353.15	$V^E/cm^3\cdot mol^{-1}$	−0.7725	0.3956	0.9498	−0.8120	−1.4993	0.0776
	$\Delta\eta/mPa\cdot s$	0.488	0.508	−0.117	−0.037	0.226	0.0123
			(x_1) Acetic Acid + (1 − x_1) DMF				
303.15	$V^E/cm^3\cdot mol^{-1}$	−3.9035	−1.8156	−0.3289	0.0436		0.0428
	$\Delta\eta/mPa\cdot s$	2.203	2.727	0.400	−1.121	−0.526	0.0328
313.15	$V^E/cm^3\cdot mol^{-1}$	−4.1525	−1.8911	−0.5312	0.1418		0.0628
	$\Delta\eta/mPa\cdot s$	1.806	2.047	0.161	−0.800	−0.288	0.0074
323.15	$V^E/cm^3\cdot mol^{-1}$	−4.3027	−2.0108	−0.3375	−0.2309		0.0532
	$\Delta\eta/mPa\cdot s$	1.463	1.595	0.104	−0.578	−0.211	0.0094
333.15	$V^E/cm^3\cdot mol^{-1}$	−4.4795	−1.9928	−0.2109	0.1656		0.0363
	$\Delta\eta/mPa\cdot s$	1.203	1.234	0.123	−0.334	−0.325	0.0160
343.15	$V^E/cm^3\cdot mol^{-1}$	−4.6113	−2.1214	0.1116	−0.3124		0.0353
	$\Delta\eta/mPa\cdot s$	1.014	1.005	0.025	−0.332	−0.108	0.0097
353.15	$V^E/cm^3\cdot mol^{-1}$	−4.8153	−2.0985	0.2785	0.2375		0.0257
	$\Delta\eta/mPa\cdot s$	0.853	0.811	0.036	−0.284	−0.161	0.0073

previously.[4,5] The measured density and viscosity values, for all the pure compounds, are listed along with those from the literature in Table 1.

Result and Discussion

Excess volumes were calculated from our measurements according to the following equations[4]

$$V^E = \frac{x_1M_1 + x_2M_2}{\rho} - \frac{x_1M_1}{\rho_1} - \frac{x_2M_2}{\rho_2} \qquad (1)$$

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[4]

$$\Delta\eta = \eta - (x_1\eta_1 + x_2\eta_2) \qquad (2)$$

where η is the viscosity of mixtures and η_1 and η_2 are the viscosity of components 1 and 2, respectively.

The densities, viscosities, excess molar volume data, and the viscosity deviations are presented in Table 2.

The values of V^E and $\Delta\eta$ for each mixture were fitted to the Redlich–Kister equation.[5]

$$Y = x_1(1 - x_1)\sum_{i=0}^{n} A_i(2x_1 - 1)^i \qquad (3)$$

where Y = V^E or $\Delta\eta$; A_i are adjustable parameters; and x_1 is

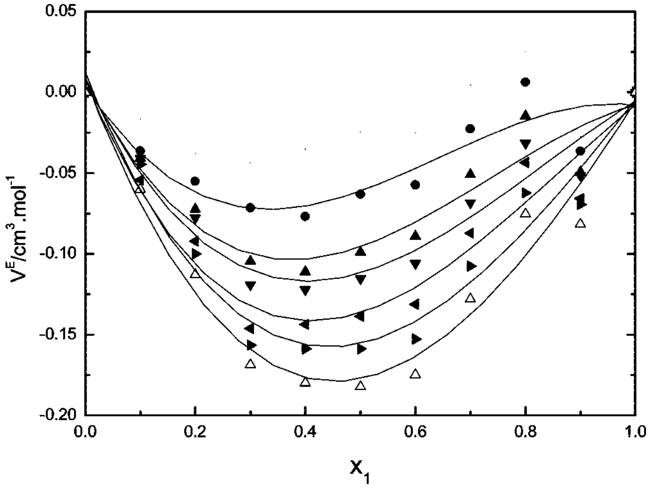

Figure 1. Excess volumes, V^E, versus the mole fraction, x_1, for the system formic acid (1) + DMF (2) at different temperatures T: ●, 303.15 K; ▲, 313.15 K; ▼, 323.15 K; solid triangle pointing left, 333.15 K; solid triangle pointing right, 343.15 K; Δ, 353.15 K; solid curves, calculated with Redlich–Kister equations; symbols, experimental values.

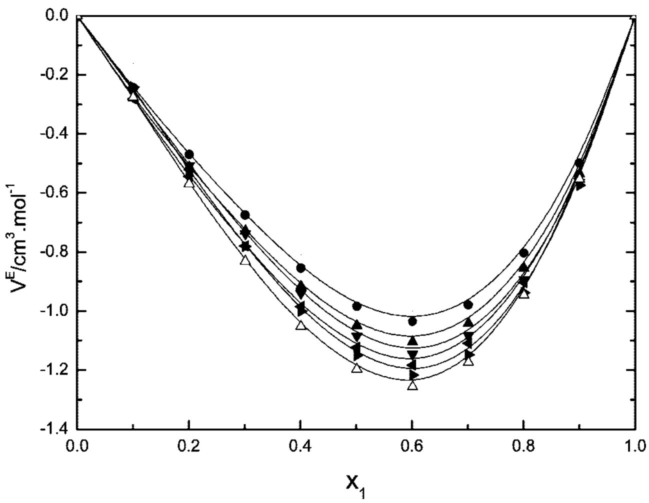

Figure 2. Excess volumes, V^E, versus the mole fraction, x_1, for the system acetic acid (1) + DMF (2) at different temperatures T: ●, 303.15 K; ▲, 313.15 K; ▼, 323.15 K; solid triangle pointing left, 333.15 K; solid triangle pointing right, 343.15 K; Δ, 353.15 K; solid curves, calculated with Redlich−Kister equations; symbols, experimental values.

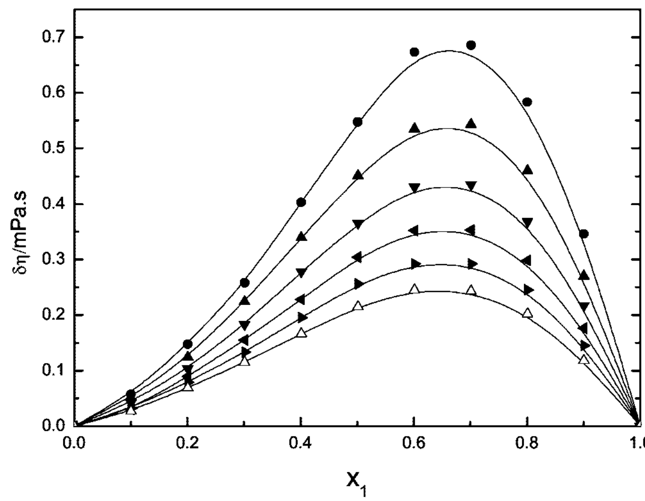

Figure 3. Deviation of viscosity, $\Delta\eta$, versus the mole fraction, x_1, for the system formic acid (1) + DMF (2) at different temperatures T: ●, 303.15 K; ▲, 313.15 K; ▼, 323.15 K; solid triangle pointing left, 333.15 K; solid triangle pointing right, 343.15 K; Δ, 353.15 K; solid curves, calculated with Redlich−Kister equations; symbols, experimental values.

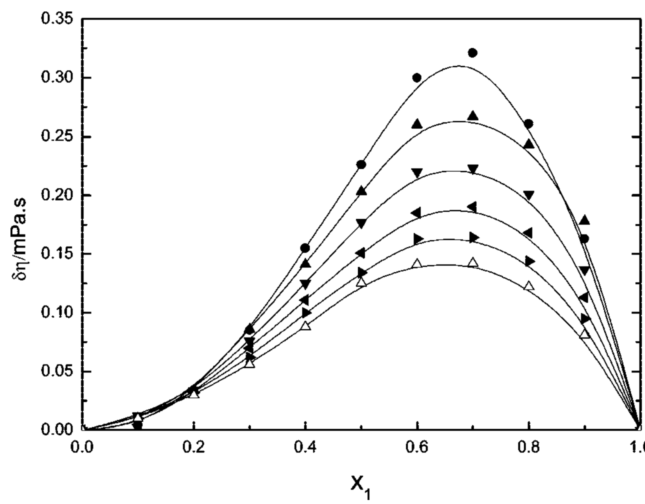

Figure 4. Deviation of viscosity, $\Delta\eta$, versus the mole fraction, x_1, for the system acetic acid (1) + DMF (2) at different temperatures T: ●, 303.15 K; ▲, 313.15 K; ▼, 323.15 K; solid triangle pointing left, 333.15 K; solid triangle pointing right, 343.15 K; Δ, 353.15 K; solid curves, calculated with Redlich−Kister equations; symbols, experimental values.

the mole fraction of component 1. The values of coefficients, A_i, were determined by a nonlinear regression analysis based on the least-squares method. The binary parameters A_i along with the standard deviations σ of these fits are summarized in Table 3.

The expression used to calculate σ was

$$\sigma(Y) = \left[\sum (Y_{\text{calcd}} - Y_{\text{exptl}})^2/(n-m)\right]^{\frac{1}{2}} \qquad (4)$$

where n is the total number of experimental values and m is the number of parameters.

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

Figures 1 and 2 show the values of V^E for the binary mixtures of formic acid (1) + DMF (2) and acetic acid (1) + DMF (2), respectively. Both binary systems have small negative V^E values, the formic acid + DMF system having about a fifth of the absolute values compared to the acetic acid + DMF system.

Figures 3 and 4 show the viscosity deviations for these two binary mixtures, respectively. The viscosity deviations are positive for these two systems.

Conclusion

Densities and viscosities for the formic acid and acetic acid with DMF binary systems have been measured in the temperature range (303.15 to 363.15) K at atmospheric pressure. The excess molar volume and viscosity deviations were correlated using the Redlich–Kister polynomial equation. The excess molar volumes for these two binary systems were negative over the whole composition range and at all temperatures, while the deviations of viscosity for these two systems were positive.

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