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Excess Molar Volumes and Viscosities for Binary Mixtures of γ-Butyrolactone with Methyl Formate, Ethyl Formate, Methyl Acetate, Ethyl Acetate, and Acetonitrile at 298.15 K

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Densities and viscosities for the binary mixtures of γ-butyrolactone with methyl formate, ethyl formate, methyl acetate, ethyl acetate, and acetonitrile have been determined over the whole mole fraction range of the mixtures at 298.15 K. From these measurements, the excess molar volumes (V_m^E) and viscosity deviations ($\Delta\eta$) were calculated. These results were fitted to Redlich−Kister type polynomials. It is shown that the values of V_m^E and $\Delta\eta$ are both negative over the entire composition range for each binary system investigated. Results have been discussed briefly on the basis of molecular interactions in these mixtures.

Introduction

As a dipolar aprotic solvent, γ-butyrolactone possesses a medium dielectric constant ($\epsilon = 41.77$ at 298.15 K) and a fairly broad liquid range (231−477 K). It can react with a lithium electrode, forming a compact and stable passivating film on the surface of lithium electrodes and prohibiting the further reaction between γ-butyrolactone and electrodes. Thus it is widely applied in primary lithium batteries. Especially, it is found in the recent years that γ-butyrolactone is a perfect plasticizer for the solid electrolyte of lithium batteries.[1,2] The solid electrolytes involving γ-butyrolactone have high conductivity and good elasticity at room temperature, and can be rather compatible with metallic lithium. Therefore, the study of thermodynamic and transport properties of the mixtures containing γ-butyrolactone should be very interesting.

It is well-known that the composition of nonaqueous organic electrolyte solutions plays a major role in determining cell performance, operating temperature, shelf life, and self-discharging rating.[3,4] Thus, selection of the solvents, as an important part of organic electrolyte solutions in lithium batteries, is very crucial. However, many researchers have shown that it is very difficult for any single solvent to show all these excellent performance points synchronically. Acetonitrile and some linear esters, such as methyl formate, ethyl formate, methyl acetate, and ethyl acetate, have low viscosities, low melting points, and high solubilities for lithium salts.[3] Mixed solvents containing these components not only show high conductivities and stability, but also possess excellent high/low temperature performance and high cycling efficiency.[5−7]

The binary mixed systems of γ-butyrolactone with alkanols and aromatic compounds have been investigated extensively. Some thermodynamic and other properties, such as V_m^E, $\Delta\eta$, and dielectric constants have been reported.[8−12] To the best of our knowledge, there are no reports on these properties for the systems concerned in the present paper. To obtain a better understanding of the molecular interactions in these mixtures, the densities and viscosities have been determined for these mixtures. From these measurements, the excess molar volumes and viscosity deviations have been calculated and fitted to Redlich−Kister polynomials. Attempts have been made to interpret the behavior of the liquid mixtures on the basis of these excess properties.

Table 1. Comprision of Experimental Density (ρ) and Absolute Viscosity (η) for the Pure Solvents Investigated at 298.15 K

	$\rho/(\mathrm{g\cdot cm^{-3}})$		$\eta/(\mathrm{mPa\cdot s})$	
solvent	expt	lit.	expt	lit.
γ-butyrolactone	1.124 55	1.1242[15] 1.12365[16]	1.722	1.7356[15] 1.6529[16]
methyl formate	0.964 77	0.9663[17]	0.328	0.3280[17] 0.328[19]
ethyl formate	0.915 98		0.379	
methyl acetate	0.928 48	0.9285[18] 0.9279[6]	0.364	0.384[18] 0.368[6]
ethyl acetate	0.894 51	0.8946[18]	0.426	0.430[18]
acetonitrile	0.776 69	0.7768[19] 0.77654[15]	0.341	0.3409[19] 0.3399[15]

Experimental Section

Chemicals. γ-Butyrolactone (Beijing Chemical Reagent Co., China, A. R.) was dried with anhydrous $CaSO_4$ and then over 4A molecular sieves for several days and distilled twice under reduced pressure, and the middle fraction was collected. Acetonitrile (Beijing Yili Fine Chemicals, China, A. R.) was dried over 4A molecular sieves for 3 days and then distilled twice. The middle fraction was collected and stored over 4A molecular sieves prior to use. Methyl formate (Shanghai Chemical. Co., China, A. R.) was rinsed with concentrated aqueous Na_2CO_3 and saturated aqueous NaCl for three times, dried with Na_2CO_3 and P_2O_5, respectively, and distilled for three times. The middle fraction was collected. Ethyl formate, methyl acetate (both Beijing Xudong Chemicals, China, A. R.), and ethyl acetate (XinXiang Huafeng Chemicals, China, A. R.) were rinsed with saturated aqueous Na_2CO_3 and saturated aqueous NaCl solutions, dried over anhydrous K_2CO_3 and P_2O_5, and then distilled twice under atmospheric pressure. All these purified solvents were stored over P_2O_5 in a desiccator before use. The mole fraction of water was <0.0005 in these solvents, as indicated by Karl−Fisher titration.

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Table 2. Experimental Density (ρ), Absolute Viscosity (η), Excess Molar Volume (V_m^E), and Viscosity Deviations ($\Delta\eta$) for Binary Mixtures of γ-Butyrolactone with Methyl Formate, Ethyl Formate, Methyl Acetate, Ethyl Acetate, and Acetonitrile at 298.15 K

x_1	ρ/g·cm^{-3}	η/mPa·s	V_m^E/cm^3·mol^{-1}	$\Delta\eta$/mPa·s	x_1	ρ/g·cm^{-3}	η/mPa·s	V_m^E/cm^3·mol^{-1}	$\Delta\eta$/mPa·s
Methyl Formate (1) + γ-Butyrolactone (2)									
0.0000	1.12455	1.722	0.0000	0.000	0.5599	1.052 08	0.678	−0.5713	−0.263
0.0499	1.11934	1.589	−0.0903	−0.063	0.6177	1.042 47	0.611	−0.5913	−0.250
0.1025	1.11341	1.457	−0.1643	−0.122	0.7071	1.027 00	0.515	−0.5370	−0.221
0.1530	1.10766	1.367	−0.2398	−0.142	0.7556	1.017 82	0.456	−0.4893	−0.213
0.2017	1.10190	1.262	−0.3062	−0.179	0.8000	1.009 10	0.426	−0.4358	−0.181
0.2492	1.09597	1.175	−0.3580	−0.200	0.8515	0.998 63	0.380	−0.3636	−0.155
0.3030	1.08910	1.080	−0.4162	−0.220	0.9005	0.988 56	0.344	−0.3010	−0.123
0.4004	1.07598	0.913	−0.5038	−0.251	0.9511	0.976 75	0.331	−0.1596	−0.065
0.4594	1.06767	0.821	−0.5511	−0.261	1.0000	0.964 77	0.328	0.0000	0.000
0.5053	1.06055	0.747	−0.5550	−0.271					
Ethyl Formate (1) + γ-Butyrolactone (2)									
0.0000	1.12455	1.722	0.0000	0.000	0.5488	1.017 07	0.732	−0.7613	−0.253
0.0501	1.11504	1.571	−0.1031	−0.084	0.6028	1.005 70	0.677	−0.7557	−0.235
0.0994	1.10551	1.446	−0.1908	−0.143	0.7138	0.981 80	0.576	−0.6845	−0.187
0.1504	1.09599	1.333	−0.3029	−0.187	0.7502	0.973 78	0.547	−0.6404	−0.167
0.2017	1.08629	1.230	−0.4050	−0.221	0.7894	0.965 16	0.510	−0.5906	−0.152
0.2492	1.07720	1.143	−0.4899	−0.244	0.8503	0.951 35	0.470	−0.4704	−0.110
0.3001	1.06720	1.057	−0.5594	−0.262	0.9000	0.939 77	0.439	−0.3374	−0.074
0.4017	1.04718	0.895	−0.6862	−0.288	0.9501	0.928 04	0.412	−0.1896	−0.034
0.4509	1.03725	0.845	−0.7255	−0.271	1.0000	0.915 98	0.379	0.0000	0.000
0.5030	1.02661	0.783	−0.7540	−0.263					
Methyl Acetate (1) + γ-Butyrolactone (2)									
0.0000	1.12455	1.722	0.0000	0.000	0.5486	1.022 82	0.678	−0.6002	−0.299
0.0471	1.11639	1.585	−0.0993	−0.073	0.5907	1.014 51	0.632	−0.5979	−0.288
0.1055	1.10617	1.467	−0.2143	−0.112	0.7010	0.992 23	0.520	−0.5410	−0.250
0.1514	1.09788	1.365	−0.2857	−0.151	0.7450	0.983 19	0.485	−0.5009	−0.225
0.2015	1.08874	1.254	−0.3559	−0.194	0.7921	0.973 46	0.446	−0.4499	−0.200
0.2517	1.07954	1.160	−0.4216	−0.220	0.8471	0.961 78	0.416	−0.3591	−0.156
0.2930	1.07191	1.091	−0.4701	−0.233	0.9005	0.950 26	0.378	−0.2497	−0.121
0.4005	1.05161	0.902	−0.5582	−0.276	0.9509	0.939 44	0.369	−0.1445	−0.062
0.4297	1.04604	0.858	−0.5762	−0.280	1.0000	0.928 48	0.364	0.0000	0.000
0.4981	1.03283	0.746	−0.6031	−0.300					
Ethyl Acetate (1) + γ-Butyrolactone (2)									
0.0000	1.12455	1.722	0.0000	0.000	0.5528	0.991 38	0.755	−0.7218	−0.251
0.0501	1.11170	1.575	−0.1236	−0.082	0.6019	0.980 29	0.707	−0.7030	−0.235
0.1023	1.09869	1.453	−0.2551	−0.136	0.6994	0.958 72	0.622	−0.6335	−0.194
0.1528	1.08612	1.345	−0.3604	−0.179	0.7500	0.947 76	0.583	−0.5807	−0.167
0.2005	1.07440	1.249	−0.4498	−0.213	0.7981	0.937 26	0.547	−0.4971	−0.141
0.2542	1.06130	1.152	−0.5324	−0.241	0.8490	0.926 34	0.513	−0.4001	−0.109
0.3039	1.04933	1.071	−0.5965	−0.257	0.9020	0.915 13	0.481	−0.2851	−0.072
0.4046	1.02561	0.926	−0.6972	−0.272	0.9504	0.905 34	0.450	−0.2005	−0.040
0.4546	1.01388	0.864	−0.7145	−0.269	1.0000	0.894 51	0.426	0.0000	0.000
0.5006	1.00332	0.810	−0.7281	−0.263					
Acetonitrile (1) + γ-Butyrolactone (2)									
0.0000	1.12455	1.722	0.0000	0.000	0.5515	0.971 59	0.728	−0.4416	−0.232
0.0503	1.11332	1.591	−0.0705	−0.062	0.5979	0.955 36	0.676	−0.4584	−0.220
0.0938	1.10280	1.471	−0.0978	−0.121	0.7014	0.916 35	0.571	−0.4561	−0.182
0.1387	1.09174	1.375	−0.1338	−0.155	0.7522	0.895 89	0.525	−0.4477	−0.158
0.2001	1.07633	1.253	−0.2008	−0.193	0.8006	0.875 23	0.485	−0.4156	−0.131
0.2490	1.06353	1.164	−0.2507	−0.214	0.8359	0.859 67	0.456	−0.3836	−0.112
0.3005	1.04954	1.074	−0.3017	−0.233	0.8994	0.830 18	0.409	−0.3320	−0.071
0.3969	1.02154	0.925	−0.3722	−0.249	0.9532	0.802 92	0.365	−0.2106	−0.041
0.4459	1.00642	0.860	−0.4005	−0.246	1.0000	0.776 69	0.341	0.0000	0.000
0.4998	0.98909	0.790	−0.4283	−0.241					

Apparatus and Measurements. The binary mixtures were prepared by mass on the molality scale. The possible error in the mole fraction is estimated to be $<\pm 1 \times 10^{-4}$. Density measurements of pure liquids and binary solutions were carried out using an Anton Paar vibrating-tube digital densimeter (model DMA 60/602, Austria). The uncertainty in density is estimated to be $\pm 2.0 \times 10^{-5}$ g·cm^{-3}. The temperature of the measurements was controlled by circulating water from a constant-temperature bath (Schott, Germany). A CT-1450 temperature controller and a CK-100 ultracryostat were used to regulate the bath temperature to within ±0.005 K. The densimeter was calibrated with dehumidized air and redistilled water from time to time. The density of water at 298.15 K was obtained from the literature.[13] The density of dehumidized air was calculated according to the formula

$$\rho_a/\text{g}\cdot\text{cm}^{-3} = 0.0012930P/\text{kPa}(1 + 0.00367t/°\text{C})/101.325 \tag{1}$$

where p is the atmospheric pressure and t is the room temperature.

Solution viscosities were measured with a suspended-leveled Ubbelohde viscometer, which was mounted in a water thermostat bath (Schott, Germany). The temperature of the water thermostat was controlled to be as precise as

Table 3. Derived Parameters and Standard Deviations of the Fit for the Excess Molar Volumes and Viscosity Deviations at 298.15 K

Y^E	B_0	B_1	B_2	B_3	B_4	S_R[a]
		Methyl Formate (1) + γ-Butyrolactone (2)				
V_m^E/cm^3·mol^{-1}	−2.258 16	−0.703 90	−0.089 27	0.182 95	−0.755 03	0.0091
$\Delta\eta$/mPa·s	−1.042 49	−0.047 62	−0.293 75			0.0064
		Ethyl Formate (1) + γ-Butyrolactone (2)				
V_m^E/cm^3·mol^{-1}	−3.004 08	−0.736 37	−0.029 82	−0.402 01		0.0041
$\Delta\eta$/ mPa·s	−1.061 97	0.430 70	−0.206 25			0.0045
		Methyl Acetate (1) + γ-Butyrolactone (2)				
V_m^E/cm^3·mol^{-1}	−2.395 46	−0.398 81	−0.219 00			0.0036
$\Delta\eta$/ mPa·s	−1.167 57	−0.040 24	−0.107 06			0.0069
		Ethyl Acetate (1) + γ-Butyrolactone (2)				
V_m^E/ cm^3·mol^{-1}	−2.897 22	−0.125 27	0.196 49	−0.418 54		0.0112
$\Delta\eta$/ mPa·s	−1.048 74	0.388 53	−0.160 73			0.0027
		Acetonitrile (1) + γ-Butyrolactone (2)				
V_m^E/ cm^3·mol^{-1}	−1.725 44	0.720 25	0.007 11	−1.239 91	−1.695 64	0.0088
$\Delta\eta$/ mPa·s	−0.963 47	0.317 38	−0.166 97			0.0030

[a] Standard deviations of the fit.

the density measurements. The viscometer was calibrated against the efflux time of redistilled water at 298.15 and 308.15 K. The desired density and viscosity data for water were taken from the literature.[14] Flow time measurements were performed by a Schott photoelectric time unit (AVS-310) with a resolution of ±0.01 s. The absolute viscosity (η) of the solutions is given by the following equation

$$\eta/\rho = At' - B/t' \tag{2}$$

where η is the solution density and t' is the flow time. A and B are the viscometer constants, and their values, 0.002 838 cm^2·s^{-2} and 2.0668 cm^2, respectively, are determined by calibration of the viscometer. The estimated accuracy of the experimental viscosity is ±0.3%. In all cases, the experiments were performed in triplicate at each mole fraction and the results were averaged.

For comparison, the experimental results of densities and viscosities for the pure solvents are listed in Table 1 along with the literature values. It can be seen that these experimental values are in good agreement with the literature ones.

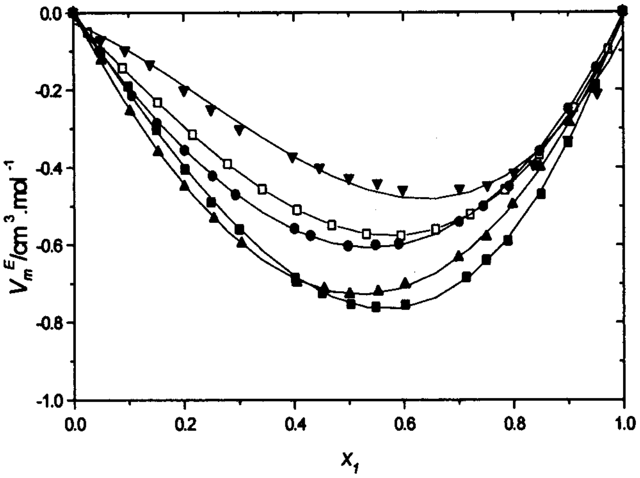

Figure 1. Excess Molar Volumes V_m^E for mixtures of component (1) + γ-butyrolactone (2) at 298.15 K as a function of mole fraction of component (1) x_1: acetonitrile (▼); methyl formate (□); methyl acetate (●); ethyl acetate (▲); ethyl formate (■).

Results and Discussion

Experimental values of density and absolute viscosity for the five binary mixtures were collected in Table 2 over the whole composition range. The excess molar volumes have been calculated by the following equation

$$V_m^E = [x_1M_1 + (1 - x_1)M_2]/\rho - x_1M_1/\rho_1 - (1 - x_1)M_2/\rho_2 \tag{3}$$

where ρ_1 and ρ_2 refer to the densities of components 1 and 2, respectively, in the various mixtures, as given in Table 2, and ρ is the density of the mixtures. M_1 and M_2 refer to the molar masses of the two components, and x_1 is the mole fraction of the first component. The viscosity deviations were calculated from the equation

$$\Delta\eta = \eta - x_1\eta_1 - (1 - x_1)\eta_2 \tag{4}$$

where η_1 and η_2 are the absolute viscosities of components 1 and 2, respectively. η refers to the viscosity of the mixtures. Excess molar volumes and viscosity deviations are also included in Table 2, and their uncertainties are estimated to be ±0.0006 cm^3·mol^{-1} and ±0.003 mPa·s, respectively. These properties were mathematically represented by the Redlich−Kister polynomials[20]

$$Y^E = x_1(1 - x_1)\sum_{j\geq 0} B_j(1 - 2x_1)^j \tag{5}$$

where Y^E refers to V_m^E or $\Delta\eta$. B_j are adjustable parameters, and they can be obtained by least-squares analysis. Values of the fitted parameters along with the standard deviations of the fit are presented in Table 3.

Dependences of excess molar volumes, V_m^E, for the mixtures investigated on the mole fraction of component 1, x_1, are displayed in Figure 1. It can be seen that values of V_m^E are all negative for the five binary mixtures over the whole mole fraction range, indicating negative deviations from ideal behaviors. This is similar to the case for mixtures of γ-butyrolactone with aromatic compounds.[10,11] The largest deviations are all located at $x_1 \approx 0.5−0.6$, except for the case of acetonitrile. The deviations at equimolar composition follow the sequence ethyl formate ≈ ethyl acetate > methyl acetate > methyl formate > acetonitrile. From the molar volumes of the compounds studied at 298.15 K (methyl formate, 62.24; ethyl formate, 80.88; methyl acetate, 79.79; ethyl acetate, 98.50; acetonitrile, 52.85; γ-butyrolactone, 76.55 cm^3·mol^{-1}),[21,23] it appears that the

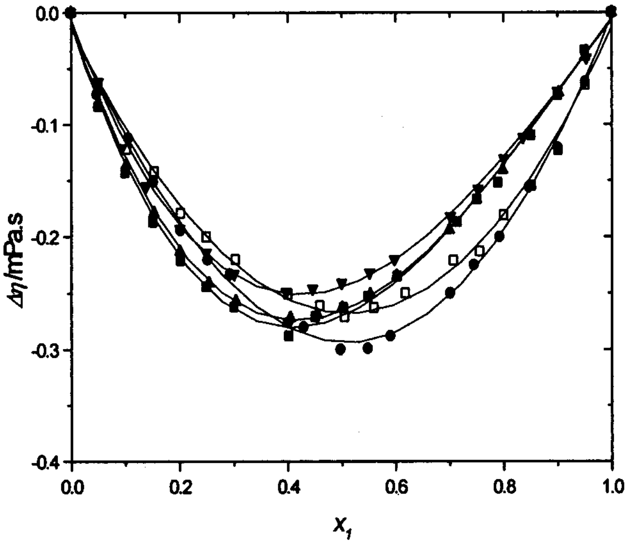

Figure 2. Viscosity deviations $\Delta\eta$ for mixtures of component (1) + γ-butyrolactone (2) at 298.15 K as a function of mole fraction of component (1) x_1: acetonitrile (▼); methyl formate (□); methyl acetate (●); ethyl acetate (▲); ethyl formate (■).

interstitial accommodation of one component into the other component is not favorable and so might have a weak contribution toward the negative V_m^E except for acetonitrile and ethyl acetate. The vapor pressure studies suggest that γ-butyrolactone molecules have a very weak specific interaction.[24] Because of its large dipole moment (μ = 4.12 D),[21] γ-butyrolactone can interact with component 1 strongly through the dipole−dipole interactions in the mixtures and hence make a significant negative contribution toward V_m^E. Thus, it may be concluded that dipole−dipole interaction and the dispersion force between unlike molecules are primarily responsible for the negative values of V_m^E in the binary systems studied.[25,26] According to Joshi et al.,[27] the large difference between the relative permittivities[21,22] of the unlike components could also be responsible for the observed negative values of V_m^E.

The variation of viscosity deviations, $\Delta\eta$, with the mole fraction of component 1 is presented in Figure 2 for the five binary mixtures. It can be seen that the viscosity deviations are also negative for every binary system investigated over the entire mole fraction composition range, and all the minims were located at $x \approx 0.4-0.5$. The negative viscosity deviations may be attributed to the existence of dispersion and dipolar forces between unlike molecules and related to the difference in size and shape of the unlike molecules.

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