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Salt Effect on the Enthalpy of Mixing of 1,4-Dioxane + Formic Acid at 303.15 K

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This paper presents the effect of five dissolved salts (sodium chloride, calcium chloride, zinc chloride, cadmium chloride, and ammonium chloride) on the endothermic enthalpy of mixing of the binary system 1,4-dioxane + formic acid measured at 303.15 K using an isothermal displacement calorimeter with vapor space. The salt sodium chloride increases the excess enthalpy values to an extent. A significantly increasing trend in excess enthalpy values for the salt calcium chloride, a remarkably decreased excess enthalpy for the salt ammonium chloride, and a comparatively smaller decrease in the excess enthalpy with the addition of zinc chloride and cadmium chloride were noticed. The experimental values of the enthalpy of mixing were fitted into a modified Redlich−Kister equation, and the deviations and parameters are reported. While the excess enthalpies of mixing (H^E) are more positive for the salts sodium chloride, cadmium chloride, and ammonium chloride, those for the salts calcium chloride and zinc chloride are more negative with increasing salt concentrations which exemplify the importance of solute−formic acid interaction.

Introduction

In recent years, there have been considerable increases in the experimental investigation of the excess thermodynamic properties of liquid mixtures. Excess thermodynamic functions have been used as a qualitative guide to predict the extent of complex formation in binary liquid mixtures. An important technique in extractive distillation is the use of salts as an extractive agent to modify the relative volatility of the binary mixtures. Furter and Cook[1] and Jaques and Furter[2] have studied the influence of added salt on the relative volatilities of two liquid components using 19 inorganic salts. The formation of the associated complexes or clusters of molecules of the volatile component around salt ions and its effect on relative volatilities have been studied extensively by Long and McDevit.[3] Furter[4]has reported that the salt effect is believed to be a complex function of salt and solvent interaction among all the components of the system. An equation has been derived by Sada et al.[5] indicating the changes in the chemical potentials of the solvent components when a salt is added to a mixed solvent. Meranda and Furter[6] have observed the gradual shift in the azeotropic composition in certain azeotropic systems by the addition of salts. Certain salts have been found to completely eliminate the azeotrope.

Very little information on the measurements of the excess enthalpy of binary mixtures in the presence of dissolved inorganic salts has been reported.[7–15] In this investigation, the effect of five dissolved salts (sodium chloride, calcium chloride, zinc chloride, cadmium chloride, and ammonium chloride) on the enthalpy of mixing of the binary 1,4-dioxane + formic acid system has been studied. The experiments were performed at 303.15 K.

Table 1. Physical Properties of the Solvents and Salts Used

solvent	this work	lit.[16]
	boiling point, T_b/K	
1,4-dioxane	374.2	374.0
formic acid	373.6	373.7
	ρ/(g·cm^{-3}), 298.15 K	
1,4-dioxane	1.0334	1.0337
formic acid	1.2210	1.2200

	as reported by the supplier	
salt	melting point, T_m/K	purity
sodium chloride	1073.45	≥ 99.8 %
calcium chloride	1044.85	≥ 99.9 %
zinc chloride	566.35	≥ 99.9 %
cadmium chloride	841.25	≥ 99.8 %
ammonium chloride	612.85	≥ 99.9 %

Experimental Section

Chemicals Used. 1,4-Dioxane, formic acid, and the salts used in this study were of analytical reagent grade supplied by E-Merck. The solvents were purified by fractional distillation, and their purity was checked by comparing measured densities (ρ) and boiling points (T_b) with those reported in the literature.[16] Their densities were measured using a bicapillary pycnometer, and their boiling were points measured using an ebulliometer giving an accuracy of ± 0.2 K. Some of the physical properties of solvents and salts used in this study are given in Table 1 along with the literature values. The melting point and purity (%) values of the salts were received from the supplier.

Experimental Apparatus. The calorimeter used in the present investigation is of the same type as that employed by Tsao and Smith[17] and Murti and Winkle.[18] The description of the apparatus, microprocessor design details, and the standard experimental procedures followed in this work has been given in the literature.[8] The total volume of the calorimeter is 215 cm^3, and the performance of the calorimeter and its reliability

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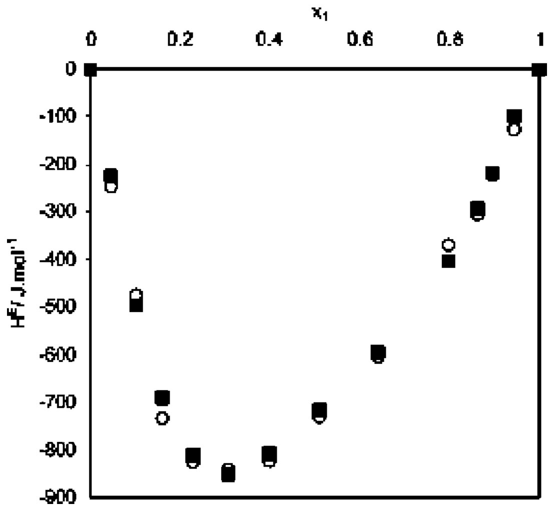

Figure 1. Enthalpy of mixing of water + methanol at 303.15 K: ○, this work; ■, Battler and Rowley.[19]

Table 2. Enthalpy of Mixing Data of the System 1,4-Dioxane (1) + Formic Acid (2) at 303.15 K for the Salt NaCl

no salt			5 % NaCl			
x_1	H^E_{exptl} / $J \cdot mol^{-1}$	H^E_{calcd} / $J \cdot mol^{-1}$	x_1	x_3	H^E_{exptl} / $J \cdot mol^{-1}$	H^E_{calcd} / $J \cdot mol^{-1}$
0.056	−211	−205.6	0.059	$3.54 \cdot 10^{-2}$	−235.0	−229.3
0.114	−401.5	−402	0.115	$2.90 \cdot 10^{-2}$	−446.2	−451.1
0.225	−813.4	−826.2	0.225	$2.11 \cdot 10^{-2}$	−898.9	−903.6
0.301	−1152	−1149.8	0.304	$1.60 \cdot 10^{-2}$	−1275.1	−1268.3
0.383	−1466	−1442.1	0.381	$1.18 \cdot 10^{-2}$	−1622.9	−1606.8
0.472	−1677	−1690.5	0.47	$8.13 \cdot 10^{-3}$	−1849.4	−1863.6
0.58	−1747	−1761.5	0.586	$5.08 \cdot 10^{-3}$	−1929.5	−1920.2
0.705	−1480	−1469	0.701	$2.64 \cdot 10^{-3}$	−1598.3	−1613.4
0.762	−1148	−1137.1	0.769	$1.53 \cdot 10^{-3}$	−1277.9	−1268.8
0.836	−762	−770.5	0.831	$7.15 \cdot 10^{-4}$	−838.6	−844.1
0.917	−240	−232.9	0.918	$1.65 \cdot 10^{-4}$	−267.5	−274.3

for the measurement of the enthalpy of mixing values was ascertained by determining the enthalpy of mixing values of the binary system water + methanol at 303.15 K. The data obtained in the present work compare well within 2 % with the literature data[19] (Figure 1). The salt is first dissolved to the required mass in the solvent in which it is preferentially soluble at the experimental temperature. The concentrations of salts reported in this work [(5 and 10) %] are the initial concentrations by mass [mass of salt/(mass of salt + mass of solvent)] before mixing with the other solvent.

Results and Discussion

It is well-known that the excess enthalpy of mixing of nonpolar 1,4-dioxane with polar formic acid is negative, and it illustrates the strong association of formic acid with 1,4-dioxane through hydrogen bonding. The influence of salts NaCl, $CaCl_2$, $ZnCl_2$, $CdCl_2$, and NH_4Cl on the excess enthalpy of mixing of this system is illustrated in Tables 2 to 5. Figure 2shows the enthalpy values of the system with 5 % mass of the salts NaCl, $CaCl_2$, $ZnCl_2$, $CdCl_2$, and NH_4Cl. The salt-free data are also given in the figure for the purpose of comparison. The dddition of univalent salt NH_4Cl decreases the area of heterogeneity to a considerable extent. As observed from Figure 2, the addition of NaCl increases the original negative values of excess enthalpy to an extent, which is more pronounced in the mole fraction region of 1,4-dioxane between 0.5 and 0.6. This trend may be attributed to the contribution from solvation energy of the salts without reducing intermolecular forces in solvents. The addition of NH_4Cl considerably decreases the negative enthalpy of mixing compared to those of the other salts and the salt-free system, and the resultant solvents interaction is expected to be less important than solute−formic acid interaction. But this interaction would not contribute much to the excess enthalpy of a system, as ammonium ions are not expected to have very strong interaction with formic acid.

Table 3. Enthalpy of Mixing Data of the System 1,4-Dioxane (1) + Formic Acid (2) at 303.15 K for the Salt $CaCl_2$

5 % $CaCl_2$				10 % $CaCl_2$			
x_1	x_3	H^E_{exptl} / $J \cdot mol^{-1}$	H^E_{calcd} / $J \cdot mol^{-1}$	x_1	x_3	H^E_{exptl} / $J \cdot mol^{-1}$	H^E_{calcd} / $J \cdot mol^{-1}$
0.051	$6.952 \cdot 10^{-2}$	−297.6	−294.9	0.057	$3.55 \cdot 10^{-2}$	−216.2	−216.1
0.117	$5.632 \cdot 10^{-2}$	−555	−574.3	0.117	$2.89 \cdot 10^{-2}$	−513.7	−494.6
0.221	$4.180 \cdot 10^{-2}$	−991.5	−972.8	0.221	$2.12 \cdot 10^{-2}$	−1065.9	−1086.6
0.305	$3.150 \cdot 10^{-2}$	−1342	−1339.6	0.309	$1.60 \cdot 10^{-2}$	−1611.8	−1627.8
0.382	$2.341 \cdot 10^{-2}$	−1707	−1701	0.38	$1.17 \cdot 10^{-2}$	−2048.2	−2022.1
0.471	$1.631 \cdot 10^{-2}$	−2052	−2076.1	0.472	$8.13 \cdot 10^{-3}$	−2385.1	−2379.1
0.582	$1.010 \cdot 10^{-2}$	−2319	−2318.1	0.581	$5.08 \cdot 10^{-3}$	−2432.1	−2445.9
0.701	$5.412 \cdot 10^{-3}$	−2119	−2103	0.705	$2.63 \cdot 10^{-3}$	−1965.6	−1956.4
0.76	$3.170 \cdot 10^{-3}$	−1803	−1788.3	0.769	$1.53 \cdot 10^{-3}$	−1491.2	−1498.1
0.835	$1.390 \cdot 10^{-3}$	−1194	−1226.6	0.832	$7.15 \cdot 10^{-4}$	−969.7	−974.1
0.919	$3.304 \cdot 10^{-4}$	−527	−514.3	0.918	$1.65 \cdot 10^{-4}$	−314.5	−305.2

Table 4. Enthalpy of Mixing Data of the System 1,4-Dioxane (1) + Formic Acid (2) at 303.15 K for the Salt $ZnCl_2$

5 % $ZnCl_2$				10 % $ZnCl_2$			
x_1	x_3	H^E_{exptl} / $J \cdot mol^{-1}$	H^E_{calcd} / $J \cdot mol^{-1}$	x_1	x_3	H^E_{exptl} / $J \cdot mol^{-1}$	H^E_{calcd} / $J \cdot mol^{-1}$
0.057	$3.54 \cdot 10^{-2}$	−143	−141.7	0.117	$5.72 \cdot 10^{-2}$	−412.5	−394.8
0.119	$2.90 \cdot 10^{-2}$	−332.6	−326.4	0.226	$4.20 \cdot 10^{-2}$	−1007	−1018.1
0.225	$2.11 \cdot 10^{-2}$	−696	−700.8	0.302	$3.08\text{v}10^{-2}$	−1372	−1394.3
0.302	$1.60 \cdot 10^{-2}$	−978	−986.6	0.382	$2.21 \cdot 10^{-2}$	−1667	−1653.1
0.386	$1.17 \cdot 10^{-2}$	−1269	−1254.3	0.476	$1.48 \cdot 10^{-2}$	−1763	−1739.3
0.475	$8.10 \cdot 10^{-3}$	−1475	−1465.1	0.585	$9.14 \cdot 10^{-3}$	−1546	−1563.4
0.589	$5.07 \cdot 10^{-3}$	−1495	−1502.8	0.704	$5.70 \cdot 10^{-3}$	−1138	−1138.6
0.703	$2.63 \cdot 10^{-3}$	−1248	−1247.8	0.773	$2.48 \cdot 10^{-3}$	−843	−843.5
0.772	$1.52 \cdot 10^{-3}$	−974	−966	0.832	$1.32 \cdot 10^{-3}$	−590	−592.4
0.831	$7.15 \cdot 10^{-4}$	−663	−677.8	0.916	$1.37 \cdot 10^{-4}$	−276	−267.9
0.918	$1.65 \cdot 10^{-4}$	−266	−255				

Table 5. Excess Enthalpy of the System 1,4-Dioxane (1) + Formic Acid (2) at 303.15 K for the Salts $CdCl_2$ and NH_4Cl

5 % $CdCl_2$				5 % NH_4Cl			
x_1	x_3	H^E_{exptl} / $J\cdot mol^{-1}$	H^E_{calcd} / $J\cdot mol^{-1}$	x_1	x_3	H^E_{exptl} / $J\cdot mol^{-1}$	H^E_{calcd} / $J\cdot mol^{-1}$
0.055	$3.59\cdot10^{-2}$	−206	−209	0.057	$3.512\cdot10^{-2}$	−163.9	−163.3
0.112	$2.90\cdot10^{-2}$	−335.9	−342.8	0.119	$2.780\cdot10^{-2}$	−356.6	−360.9
0.226	$2.09\cdot10^{-2}$	−639.8	−627.9	0.225	$2.090\cdot10^{-2}$	−673.5	−680.2
0.304	$1.55\cdot10^{-2}$	−896.5	−867.2	0.304	$1.322\cdot10^{-2}$	−876.1	−858.9
0.386	$1.12\cdot10^{-2}$	−1156.9	−1182.5	0.385	$1.022\cdot10^{-2}$	−970.6	−963.7
0.471	$7.72\cdot10^{-3}$	−1445.9	−1466.4	0.45	$7.920\cdot10^{-3}$	−965.6	−983.4
0.58	$4.53\cdot10^{-3}$	−1621.6	−1587.9	0.579	$4.326\cdot10^{-3}$	−852.4	−859
0.701	$2.26\cdot10^{-3}$	−1213.4	−1221.9	0.703	$2.230\cdot10^{-3}$	−600.8	−584.2
0.765	$1.14\cdot10^{-3}$	−815.6	−825.2	0.769	$1.014\cdot10^{-3}$	−411.5	−409.6
0.837	$3.81\cdot10^{-4}$	−385.6	−390.2	0.835	$3.914\cdot10^{-4}$	−230.5	−240.6

Table 6. Estimated Parameters of Equation 1 and Percentage of Standard Deviation % $\sigma(H^E)$ of the System 1,4-Dioxane (1) + Formic Acid (2) at 303.15 K

salt conc. (wt %)	% $\sigma(H^E)$	A_0	A_1	A_2	A_3
no salt	1.56	−6445.84	−2669.88	4004.15	4038.46
5 % NaCl	1.28	−6692.39	−2666.41	4724.18	4034.53
5 % $CaCl_2$	1.65	−7441.75	−2473.29	6072.99	3716.92
10 % $CaCl_2$	1.72	−8864.22	−5896.35	3328.53	7441.02
5 % $ZnCl_2$	1.63	−5987.47	−2051.41	4125.88	2450.65
10 % $ZnCl_2$	1.79	−6895.83	1874.41	5284.67	−3133.74
5 % $CdCl_2$	1.84	−6132.11	−3857.62	7378.18	10167.8
5 % NH_4Cl	1.57	−3838.72	1602.54	2510.01	−359.52

Among the bivalent salts, the behavior of $CaCl_2$ is rather different from that of $ZnCl_2$ and $CdCl_2$. Addition of $CaCl_2$ (5 % by mass) to 1,4-dioxane + formic acid increases the exothermic nature of the system, and a much more pronounced effect is seen for 10 % $CaCl_2$ (by mass) as observed from Table 3 and Figure 3. The negative H^E is generally attributed to the dipole-induced dipole interis $Ca^{2+} = 1.0$ Å ≈ $Cd^{2+} = 0.95$ Å > $Zn^{2+} \approx 0.74$ Å. Ca^{2+} and Cd^{2+} ions having large ionic radii can facilitate coordination of solvents with minimum interligand (solvent) repulsive forces. However, around smaller Zn^{2+}, comparatively more interligand forces are expected on its coordination sphere, providing a less negative enthalpy of mixing.[4] In our study, an increase in the negative values of H^E would be possible only if the solvated formic acid exhibited enhanced intermolecular forces with 1,4-dioxane. When formic acid coordinates with Ca^{2+} through its carboxyl oxygen, the hydroxyl proton in the same carboxyl group will be more acidic than the hydroxyl proton of the free formic acid. Therefore, the hydroxyl protons of the metal ion-coordinated formic acid can strongly interact with 1,4-dioxane with the consequent increase in the negative excess enthalpy. In the case of $CdCl_2$, repulsion among formic acid in the solvation sphere of Cd^{2+} is also expected, and it would also reduce the total solvation energy released. The same behavior is also expected to occur in the case of $ZnCl_2$.

$CaCl_2$ and $ZnCl_2$ systems were observed to exhibit more negative excess enthalpy with increasing salt concentration (10 % by mass), emphasizing the presence of strong intermolecular forces in formic acid compared to solvation of their ions (Figure 3). When 10 % $ZnCl_2$ is used, the excess enthalpy becomes more negative than the salt-free system (Table 4). As explained for the $CaCl_2$ system, the energy released is more than the energy required to disrupt the intermolecular forces for solvation of ions. Hence the excess enthalpy is negative for the addition of 10 % $ZnCl_2$ salt.

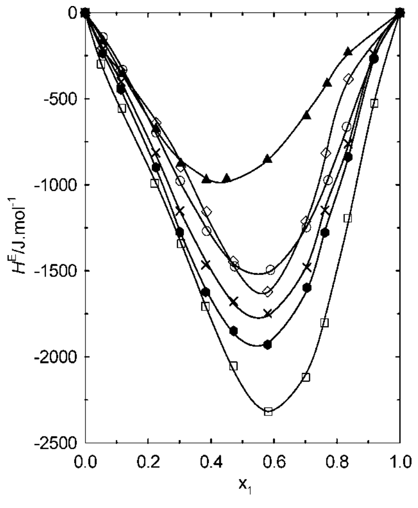

Figure 2. Effect of 5 % mass of the salts ●, NaCl; □, $CaCl_2$; ○, $ZnCl_2$; ◇, $CdCl_2$; and ▲, NH_4Cl and ×, salt-free solution on the enthalpy of mixing of 1,4-dioxane (1) + formic acid (2) at 303.15 K. Solid lines indicate the calculated value.

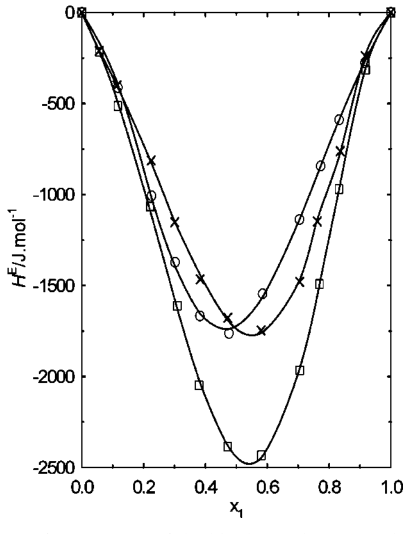

Figure 3. Effect of 10 % mass of the bivalent salts □, $CaCl_2$ and ○, $ZnCl_2$ and ×, salt-free solution on the enthalpy of mixing of 1,4-dioxane (1) + formic acid (2) at 303.15 K. Solid lines indicate the calculated value.

Data Correlation and Statistical Analysis. The Redlich–Kister model expresses the excess free energy of mixing as the sum of terms which consist of a function of the mole fractions of each component and an interaction parameter.[20] In this study, the effect of added salts on the experimental enthalpy data (H^E) was correlated with values calculated using the Redlich–Kister polynomial.

$$H^E/(\mathrm{J \cdot mol^{-1}}) = x_1 x_2 \sum_{i=0}^{j} A_i (2x_1 - 1)^i \qquad (1)$$

where H^E is the enthalpy of mixing; x_1 and x_2 are the mole fractions of components 1 and 2, respectively; and the A_i are the constants that are functions of temperature and the system properties.

The experimental H^E values were fitted to the following equation by the method of least-squares, with each point weighted equally, and the binary parameters (A_i) have been evaluated. The modeling results are listed in Table 6 along with the percentage standard deviation (σ). The values of the percentage standard deviation σ are computed from the following equation and shown in Table 6.

$$\%\ \sigma(H^E) = \left[\frac{\sum \left(\frac{H^E_{\mathrm{exptl}} - H^E_{\mathrm{calcd}}}{H^E} \cdot 100 \right)^2}{(N - K)} \right]^{1/2} \qquad (2)$$

where N and K are the number of data points and the number of parameters, respectively.

Conclusion

The experimental H^E study and its statistical analysis show that, among the five salts studied, the salts $CaCl_2$ and $ZnCl_2$ exhibit more negative values of enthalpy of mixing for the binary mixture. However, the salts NaCl, $CdCl_2$, and NH_4Cl show more positive values of excess enthalpy with increasing salt concentrations which implies that the solvent interactions become less important than solute−formic acid interaction. The model represented the experimental data with the required accuracy for the studied system, and therefore this model is suitable to predict the excess enthalpy of mixing in a 1,4-dioxane + formic acid binary mixture in the presence of the selected inorganic salts.

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