

Thermodynamic Solvation Parameters for Saturated Benzoic Acid and some of its Derivatives in Binary Mixtures of Ethanol and Water

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Abstract

Different thermodynamic parameters of solvation were evaluated for the benzoic acid, orthotoluic acid, paratoluic acid, 2-chloro benzoic acid and 4-chloro benzoic acid by measuring the molal solubilities of their saturated solutions at different temperatures in binary mixtures of ethanol-water solvents. The solubility was increased when the mole fraction of ethanol increased in the binary mixtures, and also their solvation behaviors were increased by increasing temperature. The experimental solubility, the free energy, the entropy, the enthalpy and the other solvation parameters were determined, and the findings were discussed.

Keywords: Benzoic Acid, Ortho & Paratoluic Acids, 2- & 4-Chloro Benzoic Acids, Solubility, Thermodynamic Parameters.

Introduction

Benzoic acid and its derivatives have been produced in large quantities by chemical industries (Budavari, 1996; Robert and Jeanette 1985). To produce many substitutional organic materials, the benzoic acid has been widely used as a vital carrier that can be used in the fields of pharmaceuticals, preservatives, dyes, cosmetics, and plasticizers (Dian-Qing et al. 2002; Yang et al. 2007).

Many investigators have shown the solubility information of acid compounds in water (Dian-Qing et al. 2002; Apelblat et al. 2006) and in DMF (Dian-Qing et al. 2002). Investigations in different areas involving separation process (Poulson et al. 1999; Moore et al. 1997) have been done very well- as the estimated balance has been found about operating industrial equipment and designs (Marche et al. 2004; Pino-Garcia and Rasmuson 1998). The

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chemical industry with improvements in separation processes has been used to obtain information to understand the solubility of benzoic acid and other compounds (Park and Shechan, 1991). Thermo-physical characteristics of solutions have been more beneficial to understand vital data on the intermolecular interactions of the systems, and the thermodynamic properties have been important for the industrial processes. The accurate research studies on thermodynamic characteristics of solutions would be beneficial in theoretical investigations. The aim of this study was to estimate the effects of temperature and ethanol solvents on the solubility of benzoic acid and some of its derivatives to evaluate the thermodynamic parameters of solvation.

Experimental

Chemicals:

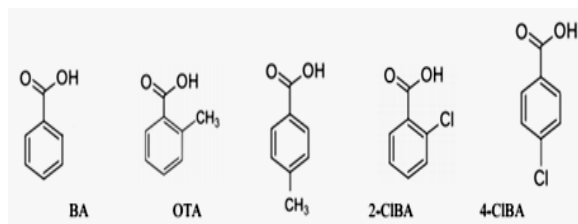


Figure 1. The chemical structure of acids

The benzoic acid (BA), ortho-toluic acid (OTA), para-toluic acid (PTA), 2-chloro-benzoic acid (2-CIBA), 4-chloro-benzoic acid (4-CIBA) (See Fig.1), and ethanol used in the experiments were purchased from Merck, they were of analytical grade reagents, and were used without any further purification.

Solubility measurement:

The solubility of acids in binary solvent mixtures of (ethanol-water) was determined by using the gravimetric method (El-Khouly et al. 2003). To each measurement, an excess mass of acid was added to about 30 mL binary solvent mixture. The equilibrium cell was heated to the desired temperature with continuous stirring for about 12 hours by using a magnetic stirrer

to fully mix the suspension, and a condenser was used to prevent the evaporation of solvent during the experiments. The temperature of the cell was controlled by circulating water from a thermostat (CF41, Julabo, Germany) through the jacket of the vessel, and measured by a thermometer with an accuracy of ± 0.02 K.

After 12 hours, stirring was stopped, and the solution was kept for 3 hours to approach equilibrium and allow the undissolved solid to settle down in the lower portion of the glass vessel. About 3 mL upper clear saturated solution was withdrawn by a syringe (10 mL), and filtered quickly with a membrane filter (0.22 μm) and poured into a pre-weighed glass dish. The total weight of the solution and the glass dish was measured immediately using an analytical balance (Mettler Toledo AB204-S, Switzerland) with an accuracy of ± 0.0001 g. When the mass of the residue reached a constant value, the final mass was recorded. At each temperature, the measurement was conducted three times, and the mean value was used to determine the solubility. The relative standard uncertainty of the solubility measurement based on the repeated observations was within 2%.

Results and Discussion

The solubility of the saturated solutions at different temperatures (293.15, 298.15, 303.15, 308.15 and 313.15K) was calculated using equation (1):

$$\text{Molalsolubility } (S) = \frac{W \times 1000}{d_0 \times M} \text{ g.mol}/1000\text{g.solven} \quad (1)$$

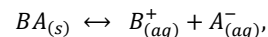
Where W is the weight of one ml. of saturated solution, after its evaporation in the aluminum dish under the effect of the tungsten lamp, M is the molecular weight of BA acid, and d_0 is the density of the solvent used (ethanol and its mixture with water). The results have been listed in Table (1), graphically demonstrated in Figure (2). As can be seen in Figure 2, both the solvent composition and temperature might cause the solubility of acids. It can be observed that when the temperature was increased, the solubility was increased to give composition in all solvent mixtures. Moreover, the solubility of acids was increased by increasing the mole fraction of ethanol (X_s) which might cause the increase in the solvation and dissolvent-solvent interactions. From these findings, it could be indicated that the dissolving process of acids has been endothermic (Gomaa et al. 2013). It was found that the solubility of benzoic acid (BA) in pure ethanol had the biggest solubility among all the used monocarboxylic acids. Also, it could be concluded that the trend of solubility in ethanol-rich solvents was: S (molal), (BA) > (2CIBA) > (OTA) > (PTA) > (4CIBA).

Table 1- Molal solubility (S) of BA, OTA, PTA, 2-CIBA and 4-CIBA in (ETOH-H₂O) mixed solvents at different temperatures.

ETOH vol. %	X_s	BA					OTA				
		293.15K	298.15K	303.15K	308.15K	313.15K	293.15K	298.15K	303.15K	308.15K	313.15K
0	0	0.0227	0.0263	0.0272	0.0299	0.0326	0.0068	0.0084	0.0091	0.0107	0.0123
20	0.0717	0.0367	0.0445	0.0538	0.0541	0.0598	0.0260	0.0276	0.0304	0.0336	0.0426
40	0.1708	0.0517	0.0591	0.0626	0.0649	0.0694	0.0574	0.0705	0.074	0.0739	0.0872
60	0.3166	0.0983	0.1531	0.1587	0.1656	0.1720	0.1011	0.1059	0.1108	0.115	0.1242
80	0.5527	0.8815	0.9706	1.0740	1.2610	1.2985	0.4264	0.4497	0.5674	0.8059	1.0543
100	1	2.7441	2.802	3.1016	3.3563	3.6672	2.3228	2.4091	2.5359	2.5847	2.6595
		PTA					2-CIBA				
0	0	0.0053	0.0084	0.0107	0.013	0.0146	0.0157	0.0185	0.0199	0.0213	0.0227
20	0.0717	0.0084	0.0108	0.0148	0.0203	0.0229	0.0266	0.0301	0.0332	0.0347	0.0371
40	0.1708	0.0448	0.0491	0.0529	0.0599	0.0681	0.0287	0.0331	0.0403	0.0443	0.0479
60	0.3166	0.0574	0.0691	0.0756	0.0805	0.1357	0.2355	0.2496	0.2608	0.3116	0.3527
80	0.5527	0.1267	0.1422	0.2108	0.2696	0.4616	1.0107	1.0443	1.1124	1.1644	1.1955
100	1	0.462	0.5339	0.6972	0.752	0.8462	1.4866	1.5454	1.6974	1.8689	1.9996
		4-CIBA									
0	0	0.0013	0.0026	0.0046	0.0100	0.0120					
20	0.0717	0.0040	0.0060	0.0061	0.0116	0.0137					
40	0.1708	0.0137	0.0145	0.0163	0.0186	0.0238					
60	0.3166	0.0373	0.0388	0.0413	0.0423	0.0463					
80	0.5527	0.0872	0.0908	0.0941	0.0971	0.1033					
100	1	0.2515	0.2796	0.2854	0.2955	0.3477					

Molal solubility (S) in (g. mole / 1000 g. solvent)

For carboxylic acid, with the formula of BA, the balance occurred in its saturated solution at a given constant temperature.



$$K_{sp} = a_+ \cdot a_- \dots \quad (2)$$

Where K_{sp} indicated the thermodynamic solubility of the constant product, and a_+ and a_- pointed out to the activity of B^+ and A^- in the solution; respectively. At high concentration, the solubility product constant (K_{sp}) has been given by Gomaa et al. 2014 b:

$$K_{sp} = S^2 \gamma_{\pm}^2 \quad (3)$$

$$pK_{sp} = -\log S^2 \gamma_{\pm}^2 \quad (4)$$

Alternatively, in the high concentration, the electrostatic interaction was highly large, and the activity coefficient might be estimated utilizing the extended Debye-Hückel law (Debye and Hückel, 1923; Bjerrum, 1926; Davies, 1962):

$$\log \gamma_{\pm} = -\frac{Z_+ Z_- A \sqrt{I}}{I + B r^{\circ} \sqrt{I}} \quad \text{for } I < 10^{-1} M \quad (5)$$

Where Z_+ and Z_- are the freightage of ions, $A = 1.823 \times 10^6 (\epsilon T)^{-3/2}$, $B = 50.29 \times 10^8 (\epsilon T)^{-1/2}$, and r° is the solvated radius, the ionic strength, I , (m_i is the molality of ion i) $I = \frac{1}{2} \sum_i m_i z_i^2$

The solvation free energy changes (ΔG_s) of acid were calculated from the solubility measurements (Gomaa. and AbouElleef 2013; Gomaa et al. 2014a) by using the following equation (6):

$$(\Delta G_s) = -2.303RT \log K_{sp} \quad (6)$$

The results have been listed in Table (2), and the solvation free energy changes of acids were decreased by the in the increase in mole fraction of ethanol, and also the increasing temperature indicated favoring more solvation of the acid.

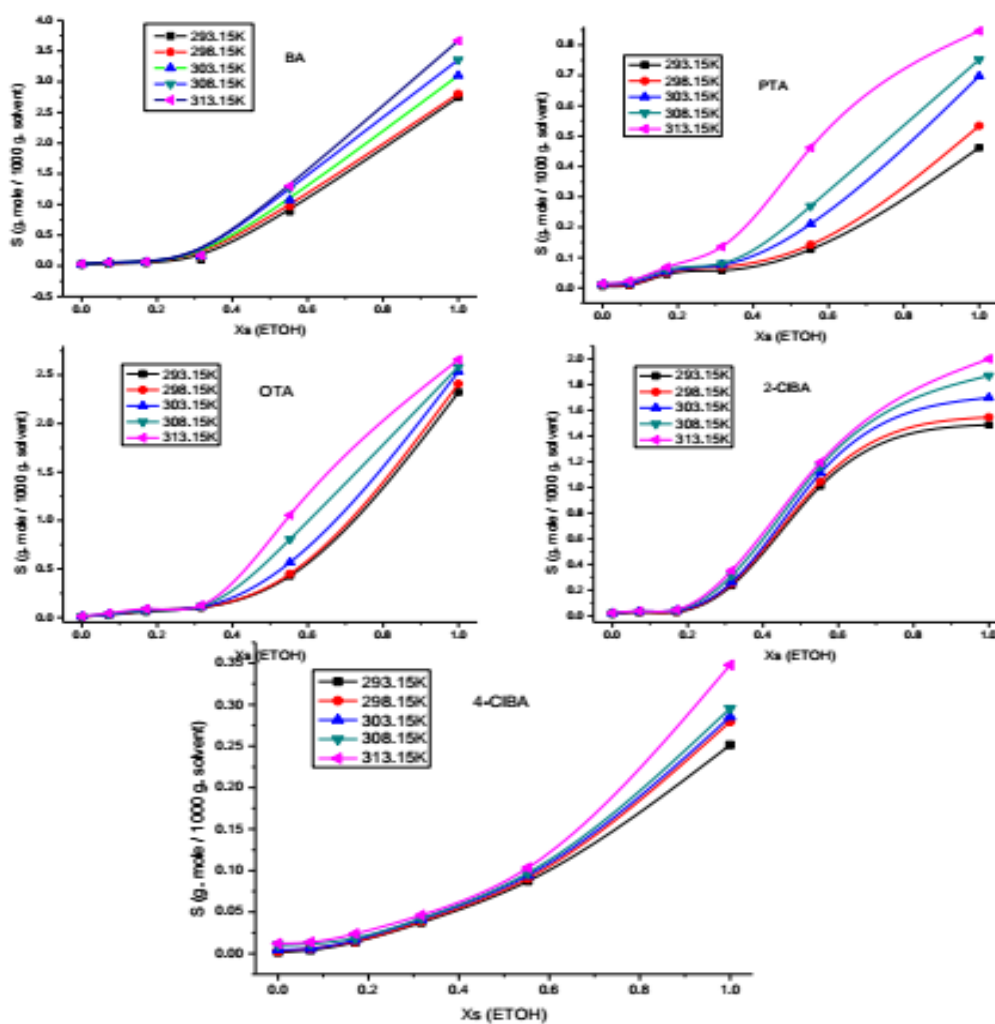


Figure 2: Relation between the molal solubility (S) of saturated BA, OTA, PTA, 2-CIBA and 4-CIBA acid solution and the mole fraction of EtOH (X_s) in (EtOH–H₂O) mixed solvents at different temperatures.

Table 2: Gibbs free energy changes of solvation (ΔG_s) for BA, OTA, PTA, 2-CIBA and 4-CIBA in (ETOH-H₂O) mixed solvents at different temperatures.

ETOH vol. %	X _s	BA					OTA				
		293.15K	298.15K	303.15K	308.15K	313.15K	293.15K	298.15K	303.15K	308.15K	313.15K
0	0	19.112	18.748	18.904	18.772	18.658	24.746	24.166	24.140	23.767	23.469
20	0.0717	16.916	16.307	15.693	15.967	15.766	18.495	18.530	18.402	18.251	17.412
40	0.1708	15.388	15.032	15.022	15.167	15.139	14.913	14.222	14.233	14.557	14.044
60	0.3166	12.555	10.751	10.780	10.997	11.159	12.431	12.435	12.447	12.696	12.688
80	0.5527	3.144	2.777	2.379	2.309	2.407	6.366	6.249	5.311	4.377	3.383
100	1	2.465	2.537	2.243	2.135	2.172	3.176	3.193	3.133	3.309	3.636
		PTA					2-CIBA				
0	0	25.931	24.166	23.394	22.815	22.615	20.809	20.394	20.397	20.408	20.424
20	0.0717	23.742	22.984	21.838	20.681	20.451	18.388	18.137	17.979	18.1	18.09
40	0.1708	16.04	15.893	15.813	15.551	15.231	18.075	17.730	17.088	16.992	16.927
60	0.3166	14.983	14.386	14.222	14.361	12.271	8.639	8.525	8.483	8.057	7.790
80	0.5527	11.717	11.407	9.822	9.369	7.214	2.533	2.444	2.217	2.678	2.795
100	1	9.772	9.4652	8.632	8.647	8.657	5.053	5.091	4.883	4.745	4.917
		4-CIBA					ΔG_s in (kJ mol ⁻¹)				
0	0	32.552	29.698	27.441	24.112	23.576					
20	0.0717	27.252	25.755	26.125	23.429	22.970					
40	0.1708	21.501	21.605	21.403	21.168	20.316					
60	0.3166	16.943	17.044	17.049	17.38	17.347					
80	0.5527	13.367	13.422	13.492	13.99	14.062					

The enthalpy alteration of solvation (ΔH) was studied from the plots of $\log K_{sp,v.s} 1/T$, (slope = $-\Delta H/2.303 R$) using equation (7) (Kim 1978):

$$\log K_{sp} = \frac{-(\Delta H)}{RT} + Constant \quad (7)$$

The values of (ΔG_s) and (ΔH) can be utilized to study the values of $T\Delta S$ at various temperatures as stated by Gibbs-Helmholtz statement:

$$(\Delta G_s) = (\Delta H) - (T\Delta S) \quad (8)$$

All the findings have been tabulated in Table (3). The enthalpies and entropies of solvation had positive quantities indicating the endothermic and spontaneous process of solvation. All the solvation thermodynamic parameters including ΔG_s and ΔS have been increased by increasing temperature. The Gibbs free energies and enthalpies of solvation for benzoic acid had low endothermic properties reaction which increased its solvation demeanors, and lowered the positivity with the high ethanol content referring to a more solvation process. Entropies of solvation were decreased with an increase in ethanol content due to increasing solute-solvent interaction, but the irregular behavior was due to solvent-solvent interaction between water and ethanol. The transfer of free energy change of solvation (ΔG_t) of acid from water (w) to (ETOH-H₂O) mixture, at different temperatures, were calculated from the following equation (Schwitzgebel G and Barthel J 1980):

$$(\Delta G_t) = (\Delta G_s) - (\Delta G_w) \quad (9)$$

Where (s), (w) denote solvent and water; respectively.

The volume of ΔG_t (solute, w \rightarrow w + s) as listed in Table (4) has been a measure of the total modification in the solvation (energy) of an ion from water to ethanol-aqueous mixture. These amounts have been generally "well behaved", that is, they varied easily based on the mission of solvent constituents, however, they might show highly variety. The transfer of free energies included negative values, and the increase in negativity increased the mole fraction of ethanol favoring more ion-solvent interaction.

The Gibbs free energies of solubilization of acids in mixture solvents might be decreased, and the increases in the mole fraction of ethanol in the mixed solvent and the carryout Gibbs free energies from water to mixed solvents increased the negative values. These confirmed that increasing organic solvent in the mixture could considerably cause variations in the solubility of benzoic acid and its derivatives in blends of ethanol-water. For that reason, the extension of an organic solvent in water is favorable for the solubilization of benzoic acid and its derivatives in mixtures of ethanol-water with increasing temperature.

Conclusions

The solubility of the saturated solution of benzoic acid and its derivatives was increased by increasing the mole portion of ethanol in blended solvents, and also it was increased with increasing temperature. Gibbs energy for the acids solution was studied to determine the solute-solvent influences concerning the electrostatic characteristics of the solvent, solute and ion solvation and also the ionic force of the average. The saturated

solution of benzoic acid and its derivatives in the blended ethanol-water solvent at various temperatures was completely non-ideal which might cause the average coefficient of ions'

activities in the solution, and it might cause the ion association phenomenon.

Table 3: Enthalpy change (ΔH) and entropy change (ΔS) of solvation for BA, OTA, PTA, 2-CIBA and 4-CIBA benzoic acid in (EtOH-H₂O) mixed solvents at different temperatures.

EtOH vol. %	BA						OTA											
	ΔH	ΔS					ΔH	ΔS										
		298.15K	303.15K	308.15K	313.15K	313.15K		298.15K	303.15K	308.15K	313.15K	313.15K						
0	24.254	5.141	5.506	5.349	5.482	5.596	42.016	17.269	17.849	17.876	18.249	18.547						
20	32.476	15.561	16.170	16.784	16.509	16.711	32.695	14.200	14.166	14.294	14.445	15.284						
40	17.519	2.131	2.487	2.497	2.352	2.381	23.042	8.129	8.820	8.809	8.484	8.998						
60	27.523	14.968	16.772	16.743	16.526	16.365	7.883	-4.548	-4.551	-4.563	-4.812	-4.805						
80	14.631	11.487	11.853	12.251	12.321	12.224	5.296	45.929	46.046	46.984	47.918	48.912						
100	8.331	5.866	5.794	6.088	6.196	6.159	-2.822	-5.999	-6.015	-5.956	-6.131	-6.459						
EtOH vol. %	PTA						2-CIBA											
	ΔH	ΔS					ΔH	ΔS										
		298.15K	303.15K	308.15K	313.15K	313.15K		298.15K	303.15K	308.15K	313.15K	313.15K						
0	24.254	114.224	88.293	90.057	90.829	91.409	25.236	4.428	4.843	4.839	4.828	4.812						
20	32.476	120.995	97.253	98.011	99.157	100.314	22.129	3.741	3.992	4.150	4.029	4.039						
40	17.519	47.346	31.306	31.453	31.533	31.795	35.972	17.896	18.242	18.883	18.979	19.044						
60	27.523	85.320	70.337	70.933	71.098	70.959	21.290	12.651	12.765	12.806	13.232	13.500						
80	14.631	147.176	135.459	135.769	137.354	137.807	11.833	-4.367	-4.278	-4.050	-4.512	-4.628						
100	8.331	71.296	61.524	61.831	62.664	62.649	8.756	3.702	3.664	3.873	4.010	3.839						
EtOH vol. %	4-CIBA						ΔH and ΔS in (kJ mol ⁻¹)											
	ΔH	ΔS																
		298.15K	303.15K	308.15K	313.15K	313.15K												
0	170.879	138.327	141.181	143.439	146.767	147.303												
20	90.935	63.683	65.180	64.810	67.506	67.965												
40	37.829	16.328	16.225	16.427	16.662	17.513												
60	10.237	-6.706	-6.807	-6.811	-7.142	-7.109												
80	1.893	-11.474	-11.529	-11.600	-12.098	-12.169												
100	7.019	-5.111	-5.001	-5.212	-5.444	-5.320												

Table 4: Transfer Gibbs free energies change of solvation (ΔG_i) for BA, OTA, PTA, 2-CIBA and 4-CIBA in (EtOH-H₂O) mixed solvents at different temperatures.

EtOH vol. %	X_s	BA					OTA										
		293.15K	298.15K	303.15K	308.15K	313.15K	293.15K	298.15K	303.15K	308.15K	313.15K						
0	0	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000						
20	0.0717	-2.196	-2.441	-3.211	-2.804	-2.892	-6.251	-5.637	-5.739	-5.516	-6.057						
40	0.1708	-3.724	-3.716	-3.882	-3.604	-3.519	-9.834	-9.944	-9.907	-9.210	-9.425						
60	0.3166	-6.558	-7.996	-8.124	-7.775	-7.499	-12.315	-11.732	-11.693	-11.071	-10.781						
80	0.5527	-15.968	-15.970	-16.524	-16.463	-16.251	-18.380	-17.917	-18.829	-19.389	-20.085						
100	1	-16.647	-16.211	-16.661	-16.637	-16.486	-21.569	-20.973	-21.006	-20.458	-19.832						
EtOH vol. %	X_s	PTA					2-CIBA										
		293.15K	298.15K	303.15K	308.15K	313.15K	293.15K	298.15K	303.15K	308.15K	313.15K						
0	0	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000						
20	0.0717	-2.189	-1.182	-1.556	-2.134	-2.164	-2.420	-2.256	-2.418	-2.308	-2.334						
40	0.1708	-9.891	-8.273	-7.582	-7.264	-7.383	-2.733	-2.664	-3.309	-3.416	-3.497						
60	0.3166	-10.948	-9.780	-9.172	-8.454	-10.343	-12.170	-11.868	-11.914	-12.351	-12.634						
80	0.5527	-14.213	-12.759	-13.572	-13.446	-15.400	-18.275	-17.949	-18.180	-17.729	-17.629						
100	1	-16.158	-14.701	-14.762	-14.167	-13.958	-15.755	-15.302	-15.514	-15.663	-15.507						
EtOH vol. %	X_s	4-CIBA					ΔH and ΔS in (kJ mol ⁻¹)										
		293.15K	298.15K	303.15K	308.15K	313.15K											
		293.15K	298.15K	303.15K	308.15K	313.15K											
0	0	0.000	0.000	0.000	0.000	0.000											
20	0.0717	-5.299	-3.943	-1.316	-0.683	-0.606											
40	0.1708	-11.050	-8.093	-6.038	-2.945	-3.260											

60	0.3166	-15.609	-12.654	-10.392	-6.733	-6.230
80	0.5527	-19.185	-16.276	-13.948	-10.122	-9.515
100	1	-20.422	-17.678	-15.210	-11.650	-11.237

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