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Solubility of Gallic Acid, Vanillin, Syringic Acid, and Protocatechuic Acid in Aqueous Sulfate Solutions from (293.15 to 318.15) K

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The solubility of gallic acid, vanillin, syringic acid, and protocatechuic acid in aqueous solutions of various concentrations of sodium sulfate was determined from (293.15 to 318.15) K at atmospheric pressure using a thermostatted reactor and HPLC analysis. Results showed a salting-out effect on the solubility of the tested phenolic compounds. The solubility data were accurately correlated to a semiempirical equation. The temperature dependence of the solubility data were used to estimate thermodynamic properties such as Gibbs free energy ($\Delta_{sol}G^o$), molar enthalpy of dissolution ($\Delta_{sol}H^o$), and molar entropy of dissolution ($\Delta_{sol}S^o$).

Introduction

In the olive oil production process, the disposal of olive oil mill waste waters (OMWW) represents one of the main environmental problems. The phenolic compounds (PhC), which are very abundant in the OMWW and are the major contributor of their polluting load, are characterized by a strong antioxidant activity.[1,2]

Aqueous solubility is a particularly important parameter for assessing environmental partitioning of different compounds. It was reported in the literature that the low solubilities in water of some solutes can be modified by the presence of cosolutes such as salts or by increasing the temperature. Two phenomena related to solubility changes caused by the presence of cosolutes can be observed: salting-in and salting-out effects.[3–7] On the other hand, increasing temperature generally increases aqueous solubility over the range of temperatures encountered at the earth's surface.[8,9] For example, Lu and Lu[10] and Daneshfar et al.[11] mentioned that the aqueous solubility of gallic acid and its esters increases with increasing temperature.

This work is a continuous effort concerning the salt addition effect on PhC solubility.[12,13] We report here on the solubility–temperature dependence data for four phenolic compounds contained in OMWW: gallic acid (3,4,5-trihydroxybenzoic acid), vanillin (4-hydroxy-3-methoxybenzaldehyde), syringic acid (4-hydroxy-3,5-dimethoxybenzoic acid), and protocatechuic acid (3,4-dihydroxybenzoic acid). This study is carried out in sulfate salt aqueous solutions.

The data obtained for each sodium sulfate concentration were used to calculate appropriate thermodynamic properties, such as Gibbs free energy ($\Delta_{sol}G^o$), molar enthalpy of dissolution ($\Delta_{sol}H^o$), and molar entropy of dissolution ($\Delta_{sol}G^o$), which have practical importance for industrial optimization processes.

Materials and Methods

Gallic acid ($C_7H_6O_5$, $M_W = 170.12$, > 98 % pure), vanillin ($C_8H_8O_3$, $M_W = 152.15$, > 98 % pure), syringic acid ($C_9H_{10}O_5$, $M_W = 198.17$, > 98 % pure), and protocatechuic acid ($C_7H_6O_4$, $M_W = 154.12$, > 97 % pure), as well as anhydrous sodium sulfate (> 99 % pure), were purchased from Sigma-Aldrich (Germany). The phenolic compounds were used without prior treatment but stored in a desiccator with P_2O_5 once the bottle has been opened. Distilled water (conductivity around 1.5 $\mu S \cdot cm^{-1}$) has been used.

Aqueous solutions of salts, from (0 to 1.5) $mol \cdot kg^{-1}$ in water, were prepared by weighing. Twenty-five grams of solution was introduced into a double jacketed reactor. An excess of the solid phenolic compound was added to the liquid phase, and the solution was continuously stirred with a magnetic stirrer. To check the equilibrium, a sample of the liquid phase was taken through a 0.25 μm filter and analyzed by high-performance liquid chromatography (HPLC). When the concentration of the liquid phase remained constant, it was assumed that the system was at equilibrium. Several equilibrium times have been tested (from (1 to 6) h), and it appears that 3 h is always sufficient to reach equilibrium. It has been verified that the presence of salts did not influence the relation between the HPLC equivalent peak area and the concentration. The reproducibility of the method was confirmed in our above-mentioned works.

The solubility of PhC was measured in pure water and in different concentrations of aqueous sodium sulfate solutions, at different temperature ranging from (293.15 to 313.15) K. This complementary study permits us to extend the solubility data basis of studied PhC using different salt counter-ions (chloride).[12,13]

The solubility experiments were used to calculate the mole fraction solubility x_{PhC} based on the following equation

$$x_{PhC} = \frac{\dfrac{m_{PhC}}{M_{PhC}}}{\dfrac{m_{PhC}}{M_{PhC}} + \dfrac{m_W}{M_W}} \qquad (1)$$

where m_{PhC} and m_w represent the masses of the solute and water, respectively, and M_{PhC} and M_W are the molecular weights of the solute and water, respectively.

Experimental Results and Discussion

The solubilities of the above-mentioned PhC in a series of concentrations of aqueous sulfate solutions are presented in

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Table 1. Experimental ($x_{\mathrm{PhC}}^{\mathrm{exptl}}$) and Calculated ($x_{\mathrm{PhC}}^{\mathrm{calcd}}$) Solubility of Phenolic Compounds (PhC) in Pure Water and in Aqueous Sodium Sulfate Solutions at Various Temperatures

molality	0.0000		0.5000		1.0000		1.5000	
T/K	$10^3 x_{\mathrm{PhC}}^{\mathrm{exptl}}$	$10^3 x_{\mathrm{PhC}}^{\mathrm{calcd}}$	$10^3 x_{\mathrm{PhC}}^{\mathrm{exptl}}$	$10^3 x_{\mathrm{PhC}}^{\mathrm{calcd}}$	$10^3 x_{\mathrm{PhC}}^{\mathrm{exptl}}$	$10^3 x_{\mathrm{PhC}}^{\mathrm{calcd}}$	$10^3 x_{\mathrm{PhC}}^{\mathrm{exptl}}$	$10^3 x_{\mathrm{PhC}}^{\mathrm{calcd}}$
Gallic Acid								
293.15	1.1668	1.1706	0.9602	0.9610	0.7807	0.7899	0.7072	0.7097
298.15	1.2225	1.2189	1.0170	1.0180	0.8504	0.8459	0.7664	0.7655
303.15	1.2835	1.2694	1.0818	1.0782	0.9175	0.9056	0.8274	0.8253
308.15	1.3087	1.3221	1.1457	1.1417	0.9753	0.9691	0.8956	0.8894
313.15	1.3805	1.3770	1.2067	1.2086	1.0310	1.0367	0.9586	0.9579
318.15	1.4307	1.4343	1.2747	1.2792	1.0996	1.1086	1.0235	1.0313

molality	0.0000		0.5000		1.0000		1.5000	
T/K	$10^4 x_{\mathrm{PhC}}^{\mathrm{exptl}}$	$10^4 x_{\mathrm{PhC}}^{\mathrm{calcd}}$	$10^4 x_{\mathrm{PhC}}^{\mathrm{exptl}}$	$10^4 x_{\mathrm{PhC}}^{\mathrm{calcd}}$	$10^4 x_{\mathrm{PhC}}^{\mathrm{exptl}}$	$10^4 x_{\mathrm{PhC}}^{\mathrm{calcd}}$	$10^4 x_{\mathrm{PhC}}^{\mathrm{exptl}}$	$10^4 x_{\mathrm{PhC}}^{\mathrm{calcd}}$
Syringic Acid								
293.15	3.7606	4.0493	3.1490	3.4073	2.1657	2.3692	1.8357	2.0924
298.15	5.2155	4.9835	4.4563	4.3010	3.4473	3.2068	3.1132	2.8594
303.15	6.4596	6.1168	5.8340	5.4121	4.6553	4.3221	4.5160	3.8904
308.15	7.9358	7.4883	7.2164	6.7898	6.0615	5.8011	5.7588	5.2704
313.15	9.1536	9.1439	8.5768	8.49305	7.5759	7.7550	7.1844	7.1100
318.15	10.5010	11.1376	9.8812	10.5930	9.8961	10.3260	8.6218	9.5528

molality	0.0000		0.5000		1.0000		1.5000	
T/K	$10^3 x_{\mathrm{PhC}}^{\mathrm{exptl}}$	$10^3 x_{\mathrm{PhC}}^{\mathrm{calcd}}$	$10^3 x_{\mathrm{PhC}}^{\mathrm{exptl}}$	$10^3 x_{\mathrm{PhC}}^{\mathrm{calcd}}$	$10^3 x_{\mathrm{PhC}}^{\mathrm{exptl}}$	$10^3 x_{\mathrm{PhC}}^{\mathrm{calcd}}$	$10^3 x_{\mathrm{PhC}}^{\mathrm{exptl}}$	$10^3 x_{\mathrm{PhC}}^{\mathrm{calcd}}$
Protocatechuic Acid								
293.15	2.1230	2.13341	1.7806	1.7878	1.3322	1.3235	1.1237	1.1306
298.15	2.2270	2.2101	1.8571	1.8673	1.3930	1.3998	1.2133	1.2064
303.15	2.2861	2.2900	1.9778	1.9503	1.4792	1.4802	1.2997	1.2868
308.15	2.3829	2.3731	2.0567	2.0372	1.5725	1.5649	1.3680	1.3722
313.15	2.4420	2.4596	2.1123	2.1278	1.6408	1.6542	1.4594	1.4628
318.15	2.5566	2.5495	2.2092	2.2225	1.7549	1.7482	1.5545	1.5588

molality	0.0000		0.5000		1.0000		1.5000	
T/K	$10^3 x_{\mathrm{PhC}}^{\mathrm{exptl}}$	$10^3 x_{\mathrm{PhC}}^{\mathrm{calcd}}$	$10^3 x_{\mathrm{PhC}}^{\mathrm{exptl}}$	$10^3 x_{\mathrm{PhC}}^{\mathrm{calcd}}$	$10^3 x_{\mathrm{PhC}}^{\mathrm{exptl}}$	$10^3 x_{\mathrm{PhC}}^{\mathrm{calcd}}$	$10^3 x_{\mathrm{PhC}}^{\mathrm{exptl}}$	$10^3 x_{\mathrm{PhC}}^{\mathrm{calcd}}$
Vanillin								
293.15	0.9890	1.0030	0.68375	0.7341	0.4643	0.5056	0.3839	0.4356
298.15	1.1722	1.2078	0.93467	0.9232	0.744	0.6749	0.6515	0.59402
303.15	1.5097	1.4511	1.26416	1.1576	0.92087	0.8973	0.8968	0.8063
308.15	1.8182	1.7395	1.55458	1.4471	1.24323	1.1883	1.2189	1.0899
313.15	2.0962	2.0805	1.79497	1.8038	1.57252	1.5677	1.4610	1.4671
318.15	2.3901	2.4830	2.10516	2.2422	1.95401	2.0605	1.7895	1.9668

Table 1 and visually in Figures 1 and 2. Results show a decrease in the solubility of PhC in the presence of an increasing amount of sodium sulfate (salting-out effect). The conceptual model for this salting-out effect relates to the strong tendency of ionic solutes to form shells of tightly bound water (hydration shells). In fact, as the concentration of the sodium sulfate increases, more and more water is bound up in hydration shells, and therefore the solubility decrease.

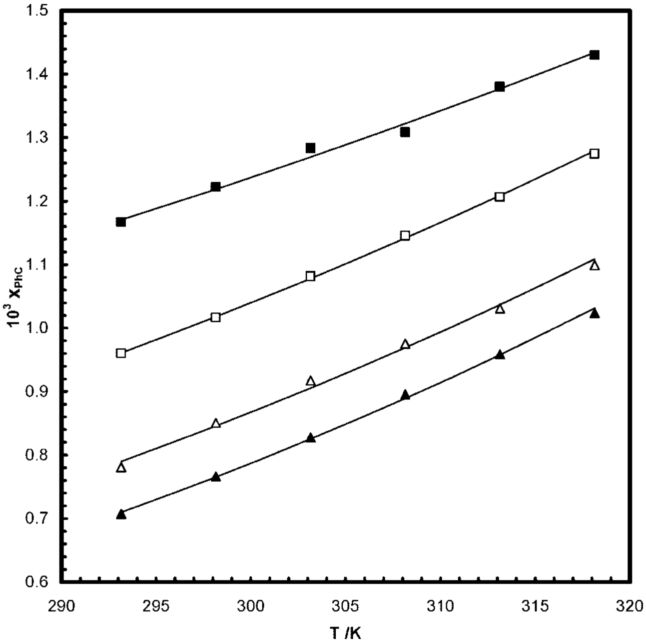

Figure 1. Experimental and calculated solubility of gallic acid in aqueous sulfate solutions and in pure water. Symbols are for experimental points, and lines are for calculated values. ■, pure water; □, 0.5 mol·kg^{-1} Na_2SO_4; Δ, 1 mol·kg^{-1} Na_2SO_4; ▲, 1.5 mol·kg^{-1} Na_2SO_4.

The solubility−temperature dependence of studied PhC in aqueous solutions was fitted to the empirical equation proposed by Heidman et al.[14]

$$\ln x_{\mathrm{PhC}} = A + \frac{B}{T/\mathrm{K}} + C\ln(T/\mathrm{K}) \qquad (2)$$

where x_{PhC} is the mole fraction solubility of PhC in water; T is the absolute temperature (K); and A, B, and C are the empirical parameters.

Experimental ($x_{\mathrm{PhC}}^{\mathrm{exptl}}$) and calculated ($x_{\mathrm{PhC}}^{\mathrm{calcd}}$) solubilities of PhC in pure water and in sulfate aqueous solutions are presented in Table 1 and visually in Figures 1 and 2. Values of the empirical parameters A, B, and C are given in Table 2 together with the corresponding root-mean-square relative deviations (rms) calculated according to the formula

$$\mathrm{rms} = \sum_{i=1}^{N}\left(\frac{x_i^{\mathrm{calcd}} - x_i^{\mathrm{exptl}}}{x_i^{\mathrm{exptl}}}\right)^2 \qquad (3)$$

where N is the number of experimental points and x_i^{calcd} and x_i^{exptl} represent the solubility calculated from eq 1 and the experimental solubility value, respectively.

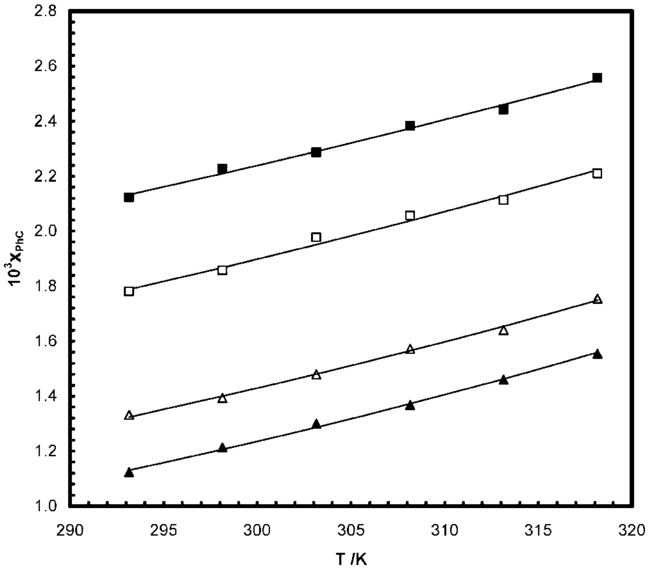

Figure 2. Experimental and calculated solubility of protocatechuic acid in aqueous sulfate solutions and in pure water. Symbols are for experimental points, and lines are for calculated values. ■, pure water; □, 0.5 mol·kg^{-1} Na_2SO_4; Δ, 1 mol·kg^{-1} Na_2SO_4; ▲, 1.5 mol·kg^{-1} Na_2SO_4.

Table 2. Parameters of Equation 2 for Phenolic Compounds in Pure Water and in Aqueous Sulfate Solution

molality	*A*	*B*	*C*	10^2 rms
		Protocatechuic Acid		
0.00	−36.73	827.31	4.88	0.062
0.50	−39.64	827.25	5.37	0.066
1.00	−44.15	827.15	6.11	0.056
1.50	−47.28	827.09	6.63	0.627
		Syringic Acid		
0.00	−96.23	825.94	15.06	3.95
0.50	−104.89	825.75	16.56	9.46
1.00	−128.70	825.25	20.69	8.75
1.50	−132.04	825.15	21.25	6.45
		Gallic Acid		
0.00	−39.06	827.24	5.19	0.043
0.50	−45.01	827.11	6.20	0.051
1.00	−48.88	827.03	6.85	0.059
1.50	−51.39	826.98	7.27	1.77
		Vanillin		
0.00	−88.016	826.17	13.78	4.39
0.50	−102.89	825.84	16.35	9.95
1.00	−123.28	825.39	19.87	7.92
1.50	−130.53	825.23	21.12	5.71

Calculated solubilities of phenolic compounds in aqueous solutions show good agreement with experimental values. Then, experimental solubility values and the correlation equation can be used as essential data and models in the practical purification process of phenolic compounds.

From Table 1 and Figures 1 and 2, we can accomplish the following conclusions: (1) the solubility of phenolic compounds in sulfate aqueous solutions increases with temperature; (2) the sulfate has a salting-out effect on the solubility of PhC; (3) with increasing concentrations of sulfate in water, the mass of PhC dissolved in water decreases.

Table 3. Thermodynamic Properties of the Dissolution of PhC in Pure Water and in Na_2SO_4 1 mol·kg^{-1} Aqueous Solutions at $\Delta_{sol}G°$ (kJ·mol^{-1}), $\Delta_{sol}H°$ (kJ·mol^{-1}), and $\Delta_{sol}S°$ (J·mol^{-1})

			Pure Water			
PhC	protocatechuic acid			syringic acid		
T/K	$\Delta_{sol}G°$	$\Delta_{sol}H°$	$\Delta_{sol}S°$	$\Delta_{sol}G°$	$\Delta_{sol}H°$	$\Delta_{sol}S°$
293.15	14.999	5.036	−33.988	19.053	29.880	36.935
298.15	15.168	5.239	−33.300	18.863	30.507	39.055
303.15	15.333	5.443	−32.624	18.662	31.134	41.140
308.15	15.494	5.646	−31.959	18.451	31.760	43.191
313.15	15.652	5.849	−31.304	18.230	32.387	45.209
318.15	15.808	6.052	−30.660	17.999	33.014	47.195
PhC	gallic acid			vanillin		
T/K	$\Delta_{sol}G°$	$\Delta_{sol}H°$	$\Delta_{sol}S°$	$\Delta_{sol}G°$	$\Delta_{sol}H°$	$\Delta_{sol}S°$
293.15	16.463	5.780	−36.442	16.840	26.741	33.773
298.15	16.644	5.996	−35.712	16.666	27.314	35.712
303.15	16.820	6.212	−34.993	16.483	27.887	37.620
308.15	16.994	6.428	−34.287	16.290	28.461	39.495
313.15	1.7163	6.644	−33.591	16.088	29.034	41.341
318.15	17.330	6.860	−32.907	15.877	29.608	43.157
			Na_2SO_4 1 mol·kg^{-1}			
PhC	protocatechuic acid			syringic acid		
T/K	$\Delta_{sol}G°$	$\Delta_{sol}H°$	$\Delta_{sol}S°$	$\Delta_{sol}G°$	$\Delta_{sol}H°$	$\Delta_{sol}S°$
293.15	16.164	8.020	−27.782	20.360	43.598	79.272
298.15	16.301	8.274	−26.922	19.956	44.459	82.184
303.15	16.433	8.528	−26.077	19.538	45.320	85.047
308.15	16.561	8.782	−25.245	19.106	46.181	87.863
313.15	16.686	9.036	−24.427	18.659	47.041	90.634
318.15	16.806	9.290	−23.622	18.199	47.902	93.361
PhC	gallic acid			vanillin		
T/K	$\Delta_{sol}G°$	$\Delta_{sol}H°$	$\Delta_{sol}S°$	$\Delta_{sol}G°$	$\Delta_{sol}H°$	$\Delta_{sol}S°$
293.15	17.423	9.828	−25.908	18.511	41.598	78.754
298.15	17.550	10.113	−24.944	18.110	42.424	81.550
303.15	17.672	10.398	−23.996	17.695	43.251	84.299
308.15	17.790	10.683	−23.063	17.267	44.077	87.004
313.15	17.903	10.968	−22.146	16.826	44.904	89.665
318.15	18.011	11.253	−21.243	16.371	45.731	92.284

From an energetic aspect, the dissolution of a PhC in a liquid is related to some thermodynamic changes, specifically the Gibbs energy ($\Delta_{sol}G^o$), molar enthalpy ($\Delta_{sol}H^o$), and molar entropy ($\Delta_{sol}S^o$) of dissolution. Those thermodynamic parameters can be calculated by using the experimental solubility data fitted to eq 2. These parameters reflect the modification of the solution properties due to the presence of the solute at its infinite dilution state at a given temperature.[15–17] Assuming that the activity coefficient of water in the aqueous phase is equal to 1 and from the Gibbs−Helmholtz equation, the following equation can be obtained[18]

$$\Delta_{sol}H^o = RT^2\left(\frac{d \ln x}{dT}\right)_P \tag{4}$$

where the molar enthalpy of dissolution $\Delta_{sol}H^o$ is the difference between the partial molar enthalpy of phenolic compound in solution, H_{PhC}, and the pure molar enthalpy, H^o_{PhC}, at temperature *T*

$$\Delta_{sol}H^o = H_{PhC} - H^o_{PhC} \tag{5}$$

The remaining parameters, $\Delta_{sol}G^o$ and $\Delta_{sol}S^o$, can be calculated as follows[16,19,20]

$$\Delta_{sol}G^o = -RT \ln(x)_P \tag{6}$$

$$\Delta_{sol}S^o = R\left(\frac{d\,(\ln x)}{d\,(\ln T)}\right)_P \tag{7}$$

The values of the thermodynamic functions, $\Delta_{sol}H^o$, $\Delta_{sol}G^o$, and $\Delta_{sol}S^o$, of the PhC dissolution in various sulfate aqueous solutions were calculated using eqs 4, 6, and 7, respectively, and are reported in Table 3.

The enthalpy of dissolution versus temperature, derived from experimental solubility data for each solution, is presented in

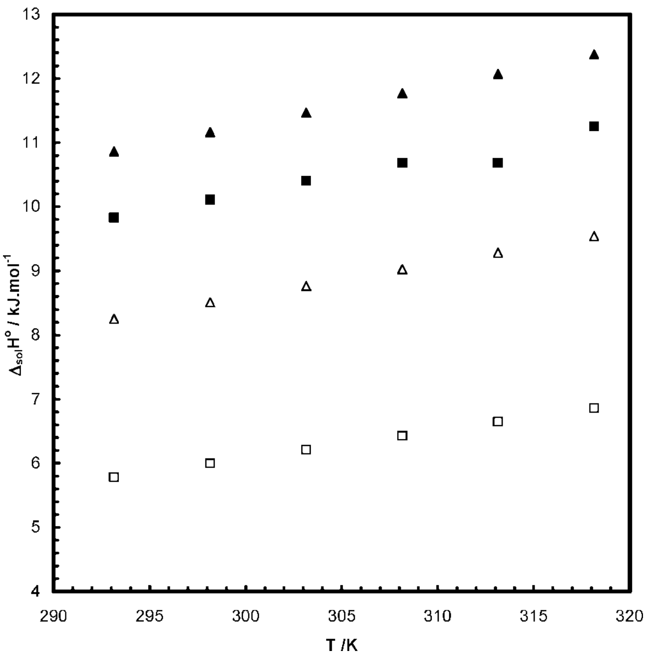

Figure 3. Enthalpy of solution of gallic acid: □, in pure water; Δ, in Na_2SO_4 0.5 mol·kg^{-1} aqueous solution; ■, in Na_2SO_4 1 mol·kg^{-1} aqueous solution; ▲, in Na_2SO_4 1.5 mol·kg^{-1} aqueous solution.

Figure 3. The estimated enthalpies of dissolution from solubility measurements of PhC in several aqueous solutions are comparable, with the major deviation for the more concentrated salt solution. For all the studied cases, it was found that the enthalpy of solution is a linear function of temperature, thus giving a constant heat capacity of solution.

The experiments showed that the whole process is endergonic ($\Delta_{sol}G^o > 0$), therefore nonspontaneous. Even though the $\Delta_{sol}S^o$ is positive in the case of vanillin and syringic acid, the $\Delta_{sol}H^o$ is sufficiently positive to provide positive $\Delta_{sol}G^o$ values. The dissolution of PhC is subsequently especially of enthalpic origin.

According to $\Delta_{sol}H^o$ values and in the same way of our previous work,[13] studied PhC can be divided into two groups having comparable molar enthalpy values. Gallic and protocatechuic acids compose the first group, while the second group is reserved for vanillin and syringic acid. This classification can be explained by the fact that the first family carries more than one hydroxyl group and is thus more soluble, whereas the second family bears only one hydroxyl group and one methoxy group at least and is therefore less soluble compared to the first family (Tables 1 and 3).

Conclusions

Original data for the solubility of some PhC in aqueous sulfate solutions, at temperatures ranging between (293.15 and 318.15) K and at atmospheric pressure, are presented. The presence of electrolytes decreases the phenolic compound's solubility in water. The salting-out effect was pointed out and found to increase with the increase of salt concentration, from pure water to salt solution. Solubility experimental data were fitted to the Heidman equation, and from the proper fits, the thermodynamic molar properties of solution were derived.

Note Added after ASAP Publication. This paper was published ASAP on June 12, 2008. Table 1 headings, Figure 1 caption, and Figure 2 caption were updated. The revised paper was reposted on June 19, 2008.

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