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# **THERMODYNAMIC FUNCTIONS, SOLUBILITY AND DENSITY OF HYDROQUINONE IN PURE WATER, METHANOL AND THEIR BINARY SOLVENT MIXTURES AT VARIOUS TEMPERATURES WITH FT-IR**

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## **ABSTRACT**

Solubility of hydroquinone in water, methanol and in water+methanol binary mixtures have been experimentally measured using a gravimetric method at temperatures (293.15, 295.15, 298.15, 300.15, 303.15, 305.15, 308.15, 310.15 & 313.15 K). Hydroquinone solubility values are correlated with temperature by using the Apelblat equation. The combined nearly ideal binary solvent (NIBS)-Redlich-Kister equation is used to fit experimental solubility data in mixed solvents at constant temperature. Thermodynamic functions including *ΔH°soln, ΔG°soln*, and *ΔS°soln* of hydroquinone in different solvents are obtained from the modified van't Hoff equation. FTIR study also done for some mole fraction of hydroquinone solution.

**Keywords:** Hydroquinone, Solubility, Density, Apelblat equation, FTIR.

# **1. INTRODUCTION**

Hydroquinone is the major benzene metabolite used for production dyes, paper, pesticides, polymeric material, pharmaceutical, petrochemical product etc. It can be used as a developing agent in photography, dye intermediate, stabilizer in paints, varnishes oils and motor fuels. In addition, hydroquinone has been used as an antioxidant in the rubber and food industry. From 1950s to 2001 hydroquinone was applied in the commercially available cosmetic skin lightening formulations in European Union countries and since 1960s it was commercially available as a medical product. It is also present in cosmetic formulations of products for coating finger nails and hair dyes [1, 2]. The antimicrobial properties of arbutin as the main compound and hydroquinone as the active metabolite were determined and compared with the antimicrobial properties of *A. unedo leaf* extracts so as to test the extent to which arbutin is responsible for antimicrobial activity [3]. There has increase industrial wastewater containing phenolic compound, which are toxic to aquatic life and human bring [4].Thus it is one of the base compound used in chemical synthesis, pharmaceutical and cosmetics.

Solubility data is required for selection of proper solvent and design an optimized crystallization process, in this paper the systematic study of solubility and density of hydroquinone in water + methanol binary solvents over

the entire composition range from 0 to 1 mole fraction, at temperatures (293.15 to 313.15)K is reported. The thermodynamic functions for saturated hydroquinone solution are calculated using modified van't Hoff equation.

# **2. MATERIAL AND METHODS**

## **2.1. Material**

Triple distilled water was used in all experiments. Hydroquinone was supplied by Sigma-Aldrich co. (99%, Reagent Grade) while Methanol was procured from Merck, Darmstadt, Germany (≥99.8%. G.R.)

# **2.2. Apparatus and Procedure**

The solubility of hydroquinone was measured using an apparatus similar to that described in the literature [5, 6]. In this work, an excess amount of hydroquinone is added to the binary solvents mixtures prepared by weight (Shimadzu, Auxzzo) with an uncertainty of  $\pm 0.1$  mg, in a specially designed 100 ml double jacketed flask. Water was circulated at constant temperature in jacket between the outer and inner walls of the flask. The temperature of the circulating water was controlled by auto temperature control thermostat within  $\pm 0.1$  K. The solution was continuously stirred using a magnetic stirrer for about 1 hour so that equilibrium was assured and no further solute dissolved, and the temperature of solution

was same as that of circulating water. The stirrer was switched off and the solution was allowed to stand for 1 hour. Then a fixed quantity of the supernatant liquid was withdrawn from the flask in a weighing bottle with the help of pipette which was hotter than the solution. The weight of this sample was taken and kept in an oven at 343 K until the whole solvent was evaporated and the residue was completely dry. This was confirmed by weighing two or three times until a constant weight was obtained after keeping the sample in an oven for another 30 min every time. The solubility has been calculated using weight of solute and weight of solution. Each experimental value of solubility is an average of at least three different measurements and the standard uncertainty of the experimental mole fraction solubility  $(x_B)$ , value is  $\pm$  0.003. The mole fraction solubility  $(x_B)$ , initial the mole fraction of methanol  $(x_C^0)$ , were calculated using usual equations [7]. The standard uncertainty for  $x_c^0$  is 0.0002. Densities are determined using a 15  $\text{cm}^3$  bicapillary pycnometer as described earlier. For calibration of pycnometer triply distilled and

degassed water with a density of 0.99705 *g*cm −3 at 298.15 K was used. The pycnometer filled with air bubble free experimental liquids was kept in a transparent walled thermostat (maintained at constant temperature  $\pm 0.1$  K) for (10 to 15) min to attain thermal equilibrium. The heights of the liquid levels in the two arms were measured with the help of a traveling microscope, which could read to 0.01 *mm*. The estimated standard uncertainty of the density measurements of the solvent and binary mixtures was 10  $kg·m<sup>-3</sup>$  [8-11].

# **3. RESULTS AND DISCUSSION 3.1. Solubility**

Table 1 show the experimental and calculated (using Apelblat equation) values of solubility  $(x_B)$  of hydroquinone at 293.15 to 313.15 K in water, methanol and water+methanol respectively. The density of each saturated solution is also reported. Variation of solubility with  $x_c^0$  is visually shown in Fig. 1.

Table 1: Experimental  $\mathbf{x}_{B(exp)}$  and Calculated  $\mathbf{x}_{B(cal)}$ Values of Mole Fraction Solubility and Density  $(\rho)$ of Hydroquinone for Various Initial Mole Fractions,  $(x_c^0)$ , of Methanol at Temperatures (293.15 to **313.15) K and Pressure 101.32** *kPa<sup>a</sup>* **.** 

T(K)	$x_c^0$	$x_{B(exp.)}$	$x_{B(cal.)}$	RD	$\rho \cdot 10^{-3} / kg \cdot m^{-3}$
	0.0000	0.0102	0.0103	0.0098	1.0090
	0.1001	0.0181	0.0181	0.0000	0.9911
	0.2002	0.0299	0.0297	0.0067	0.9805
	0.3001	0.0480	0.0478	0.0042	0.9731
	0.4000	0.0660	0.0657	0.0045	0.9672
293.15	0.5000	0.0828	0.0824	0.0048	0.9589
	0.6020	0.0965	0.0965	0.0000	0.9507
	0.7002	0.1082	0.1081	0.0009	0.9406
	0.7998	0.1167	0.1168	0.0009	0.9304
	0.9000	0.1245	0.1242	0.0024	0.9195
	1.0000	0.1331	0.1331	0.0000	0.9098
	0.0000	0.0112	0.0112	0.0000	1.0097
	0.1001	0.0199	0.0199	0.0000	0.9913
	0.2002	0.0327	0.0327	0.0000	0.9815
	0.3001	0.0511	0.0515	0.0078	0.9746
	0.4000	0.0693	0.0697	0.0058	0.9680
295.65	0.5000	0.0858	0.0862	0.0047	0.9605
	0.6020	0.1003	0.1002	0.0010	0.9510
	0.7002	0.1115	0.1117	0.0018	0.9409
	0.7998	0.1207	0.1207	0.0000	0.9308
	0.9000	0.1277	0.1278	0.0008	0.9201
	1.0000	0.1359	0.1360	0.0007	0.9103
298.15	0.0000	0.0122	0.0122	0.0000	1.0102

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	0.1001	0.0217	0.0220	0.0138	0.9927
	0.2002	0.0355	0.0358	0.0085	0.9831
	0.3001	0.0555	0.0555	0.0000	0.9763
	0.4000	0.0732	0.0739	0.0096	0.9693
	0.5000	0.0897	0.0902	0.0056	0.9619
	0.6020	0.1039	0.1043	0.0038	0.9521
	0.7002	0.1156	0.1156	0.0000	0.9415
	0.7998	0.1247	0.1247	0.0000	0.9316
	0.9000	0.1312	0.1315	0.0023	0.9203
	1.0000	0.1392	0.1392	0.0000	0.9108
	0.0000	0.0134	0.0134	0.0000	1.0108
	0.1001	0.0245	0.0243	0.0082	0.9940
	0.2002	0.0388	0.0393	0.0129	0.9844
	0.3001	0.0597	0.0597	0.0000	0.9780
	0.4000	0.0790	0.0782	0.0101	0.9708
	0.5000	0.0945	0.0945	0.0000	0.9628
300.65	0.6020	0.1086	0.1086	0.0000	0.9534
	0.7002	0.1197	0.1196	0.0008	0.9430
	0.7998	0.1290	0.1289	0.0008	0.9324
	0.9000	0.1352	0.1355	0.0022	0.9215
	1.0000	0.1434	0.1427	0.0049	0.9115
	0.0000	0.0147	0.0147	0.0000	1.0113
	0.1001	0.0273	0.0269	0.0147	0.9953
	0.2002	0.0439	0.0431	0.0182	0.9869
	0.3001	0.0645	0.0643	0.0031	0.9802
	0.4000	0.0836	0.0828	0.0096	0.9729
303.15	0.5000	0.0996	0.0991	0.0050	0.9652
	0.6020	0.1134	0.1131	0.0026	0.9545
	0.7002	0.1238	0.1239	0.0008	0.9441
	0.7998	0.1334	0.1332	0.0015	0.9334
	0.9000	0.1398	0.1397	0.0007	0.9223
	1.0000	0.1458	0.1465	0.0048	0.9120
	0.0000	0.0161	0.0162	0.0062	1.0119
	0.1001	0.0300	0.0298	0.0067	0.9969
	0.2002	0.0473	0.0471	0.0042	0.9887
	0.3001	0.0698	0.0693	0.0072	0.9818
	0.4000	0.0869	0.0876	0.0081	0.9742
305.65	0.5000	0.1042	0.1039	0.0029	0.9661
	0.6020	0.1180	0.1179	0.0008	0.9558
	0.7002	0.1290	0.1285	0.0039	0.9456
	0.7998	0.1379	0.1377	0.0015	0.9344
	0.9000	0.1445	0.1440	0.0035	0.9230
	1.0000	0.1504	0.1507	0.0020	0.9127
	0.0000	0.0180	0.0180	0.0000	1.0127
	0.1001	0.0320	0.0330	0.0313	0.9987
	0.2002	0.0518	0.0516	0.0039	0.9906
	0.3001	0.0740	0.0746	0.0081	0.9835
	0.4000	0.0925	0.0925	0.0000	0.9760
308.15	0.5000	0.1090	0.1090	0.0000	0.9674
	0.6020	0.1231	0.1230	0.0008	0.9569
	0.7002	0.1333	0.1334	0.0008	0.9466
	0.7998	0.1419	0.1424	0.0035	0.9355
	0.9000	0.1486	0.1486	0.0000	0.9247

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"Standard uncertainties in u are u(T) = 0.1 K, u( $x_c^0$ ) = 0.0002, u( $x_b$ ) = 0.003, and u( $\rho$ ) = 10 kg·m<sup>-3</sup>. The relative uncertainty in pressure ur(p) *= 0.05.* 



**Fig.1: Mole Fraction Solubility of Hydroquinone ( ) Variation with Initial Mole**  Fraction  $(x_C^0)$  of Methanol at Various **Temperatures** (◆T=293.15 K, ■T=295.65 K;  $\triangle$ T=298.15 K;  $\times$ T=300.65 K;  $\circ$ T=303.15 K; ●T=305.65 K; +T=308.15 K; -T=310.65 K &  $-T=313.15 K$ ).

The solubility of hydroquinone in all solvents increases with temperature. At the same temperature, the solubility trend in solvent is methanol  $>$  water  $+$ 

methanol >water. This trend implies that solubility of hydroquinone increases with increasing with mole fraction of methanol, it is prefer to dissolve more in methanol than water. The solubility of hydroquinone in water-methanol mixture with  $x_c^0$  increases with increases in  $x_B$  up  $x_C^0=1$ . This implies that there is strong dipole-dipole interaction between solute and solvent molecules. As temperature increases density goes on decreasing. But here increase of temperature and mole fraction of methanol density goes on increases; this is because of increase of solubility with temperature and mole fraction of methanol.

#### **3.2. Apelblat Model**

Between the different methods, the modified semiempirical Apelblat model (eq 1) is a suitable way to correlate solubility data against temperature [12, 13]. The equation is based on solid-liquid equilibrium theory provide excellent agreement between experimental and calculated values of solubility [14].

$$
ln x_{B} = A + \frac{B}{T} + C ln T \quad \ldots \ldots \ldots \ldots \ldots \tag{1}
$$

*A, B,* and *C* are the model parameters and T is temperature in Kelvin. *A* and *B* represent the nonidealities of the solutions in terms of the variation of activity coefficients, *C* reflects to the effect of temperature on the enthalpy of fusion [15]. *A, B,* and *C* parameters are determined using non-linear least square fitting [16]. Solubility values of hydroquinone in water, methanol and their mixtures are calculated by eq 1. Relative deviation (*RD)*[17] is calculated using eq 2.

$$
RD = \frac{x_B^{\exp} - x_B^{\text{cal}}}{x_B^{\exp}} \qquad \qquad (2)
$$

The data of experimental mole fraction solubility, calculated solubility and *RD* in monosolvent (water, methanol) and water-methanol mixtures are listed in Table 1. The values of parameters *A, B, C* along with

corelation coefficient  $(R^2)$  are listed in Table 2.

## **3.3. NIBS-Redlich-Kister Model:**

The solubility data at constant temperature is fitted into combined NIBS-Redlich-Kister model [18-21].

 $ln x_B = x_C^0 ln x_1 + x_A^0 ln x_2 + x_C^0 x_A^0 \sum_{i=0}^3 M_i (x_C^0 - x_A^0)^i$ ---------------(3) Where  $x_A^0$  is initial mole fraction of water and  $x_1, x_2$ are solubilities of hydroquinone in pure methanol and water respectively. *M<sup>i</sup>* is curve fit parameters (four parameter equation). All values of  $M_i$  along with  $R^2$ value are listed in Table 3. The values of  $R^2$  are close to unity shows that NIBS-Redlich-Kister model is very well applicable for this solubility data.



#### **Table 2: Model Parameters and Correlation Coefficient of the Apelblat Equation**

## **Table 3: NIBS-Redlich-Kister model parameters**



### **3.4. Thermodynamics Functions of Dissolution**

According to the van't Hoff equation, the standard molar enthalpy change of solution *ΔH°soln* is generally obtained from the slope of the  $ln \mathbf{x}_B$  vs  $1/T$  plot. Average temperature *Tmean* is introduced to obtain a single value of *ΔG°soln* and *ΔS°soln* in the temperature range studied.

$$
T_{mean} \frac{n}{\sum_{i=1}^{n} \left(\frac{1}{T}\right)}
$$
 (4)

Where *n* is the number of experimental points. In the present work,  $T_{mean} = 302.98$  K and the temperature range is (293.15 to 313.15) K in both pure solvents and binary solvent mixtures. Heat capacity of the solution can be assumed as constant. Hence values of *ΔH°soln* are derived using eq 5.

$$
\Delta H_{sol}^0 = -R \left( \frac{\partial \ln x_B}{\partial 1/T} \right) - R \left[ \frac{\partial \ln x_B}{\partial \left( \frac{1}{T} - \frac{1}{T_{mean}} \right)} \right] \qquad \dots \dots \dots \dots \tag{5}
$$

The  $ln x_B$  Vs 10000 ( $1/T - 1/T$  mean) plot of different solutions including pure solvents and binary solvent mixtures are displayed in Figures 2.



Fig. 2: Plot of  $ln x_B$  vs.  $(1/T-1/T_{hm})$  for **Hydroquinone+Water+Methanol System at various** Mole fractions. ( $\blacklozenge x_c^0$ =0.0000; ■  $x_c^0$ =0.1001;  $\blacktriangle$   $x_c^0$ =0.2002;  $\times$   $x_c^0$ =0.3001;  $\alpha_{\mathcal{C}}^0 = 0.4000; \quad \bullet \quad x_{\mathcal{C}}^0 = 0.5000; \quad + \quad x_{\mathcal{C}}^0 = 0.6020;$  $x_c^0$ =0.7002;  $-x_c^0$ =0.7998 and  $\diamondsuit x_c^0$ =0.9000;  $\Box$  $x_C^0$ =1.0000).

From these figures, it can be seen that a trend of increasing solubility with temperature is observed. The slope and the intercept for each solvent are listed in Table 4. Thus the modified van't Hoff equation can be thought to be fit to calculate the enthalpy change of solution. The standard molar Gibbs energy change for the solution process *ΔG°soln,* can be calculated in the way similar to Krug et al [22] as

$$
\Delta G_{sol}^0 = -RT \times intercept \qquad \qquad (6)
$$

In which the intercept used is that obtained in plots of l*nx<sup>B</sup>* as a function of (*1/T-1/Tmean*). The standard molar entropy change  $\varDelta S_{\mathit{soln}}^{\mathit{0}}$  is obtained from

$$
\Delta S_{Sol}^0 = \frac{\Delta H_{Sol}^0 - \Delta H_{Sol}^0}{T_{mean}} \qquad \qquad \dots \dots \dots \tag{7}
$$

Both *ΔG°soln* and *ΔS°soln* pertain to the mean temperature  $T_{mean} = 302.92$  K.

The results are shown in Table 5, together with %*ζH* and %*ζTS*. It is worthy to note that relative contribution of enthalpy %*ζH* and %*ζTS* which are defined as

$$
\% \zeta_H = \frac{\Delta H_{Sol}^0}{|\Delta H_{Sol}^0| + |\tau \Delta S_{Sol}^0|} X 100 \quad \dots \dots \dots \dots \tag{8}
$$

$$
\% \zeta_{TS} = \frac{|\tau \Delta S_{Sol}^{\circ}|}{|\Delta H_{Sol}^{\circ}| + |\tau \Delta S_{Sol}^{\circ}|} X 100 \qquad \qquad (9)
$$

can be simply used to calculate the main contributors of enthalpy or entropy to *ΔG°soln* [23].



Water + Methanol+Hydroquinone			
$\overline{X_c}^{\bullet}$	m	C	$R^2$
0.0000	$-3584$	$-4.201$	0.995
0.1001	$-3732$	$-3.607$	0.996
0.2002	$-3343$	$-3.142$	0.998
0.3001	$-2723$	$-2.739$	0.999
0.4000	$-2067$	$-2.490$	0.998
0.5000	$-1729$	$-2.307$	0.998
0.6020	$-1517$	$-2.174$	0.998
0.7002	$-1316$	$-2.083$	0.997
0.7998	$-1213$	$-2.013$	0.999
0.9000	$-1120$	$-1.964$	0.997
1.0000	$-999.2$	$-1.913$	0.991

**Table 5: Thermodynamic Functions Relative to Solution Process of Hydroquinone at** *Tmean* **=302.928K** 



The values of *ΔH°*and *ΔS°* for all solutions are positive indicating the solution process as endothermic. The contribution of enthalpy to positive molar Gibbs energy is more as compared to entropy for all solutions.

Density values are used to calculate excess molar functions. [24]

#### **3.5. FTIR Spectra**

In ternary system, υ-OH decreases with increase with mole fraction of alcohol because hydroquinone interaction with binary solvent increase with increase of mole fraction of alcohol s observed in Table 6. The υ-OH decreases as amount of alcohol in mixture increases this is because frequency is after interaction of -OH of alcohol and -OH of water. The υ-OH of alcohol is less than that of water therefore as amount of alcohol increases in mixture υ-OH decreases as shown in Fig. 3, 4, 5, 6 and 7. Solubility of hydroquinone increases

with increasing amount of alcohol because hydroquinone is more soluble in alcohol than in water. This is due to solvent-solvent and solute-solvent interaction in terms of hydrogen bonding. More is the hydrogen bonding, lower the υ-OH and more is the solubility of hydroquinone.

**Table 6: Experimental υ-OH for 0, 0.2, 0.5, 0.7**  and 1 mole fraction of alcohol  $(x_C^0)$  in ternary **solutions.** 

Methanol $(x_c^0)$	Experimental v-OH Hydro- quinone + water + alcohol $cm^{-1}$
	3387
02	3379.29
0.5	3375.43
07	3363.86
	3332.99



**Fig. 3: FTIR spectra for Hydroquinone+Water** 



**Fig. 4: FTIR spectra for solution of Hydroquinone in 0.2 mole fraction of Methanol** 



**Fig. 5: FTIR spectra for solution of Hydroquinone in 0.5 mole fraction of Methanol** 



**Fig. 6: FTIR spectra for solution of Hydroquinone in 0.7 mole fraction of Methanol** 



**Fig. 7: FTIR spectra for Hydroquinone+Methanol.** 

## **4. CONCLUSION**

Solubility data and Thermodynamic functions including *ΔH°soln, ΔG°soln*, and *ΔS°soln* of hydroquinone in water,

methanol and their mixture are more useful in field of physical chemistry and chemical engineering calculations involving fluid flow, heat and mass transfer, pharmaceutical industry, agriculture, biology, medicine. Solubility data is required for selection of proper solvent and design an optimized crystallization process.

The solubility of hydroquinone is more in methanol than in water and increases with increase in mole fraction of methanol. *ΔH°soln* values are higher in water than water +methanol mixture and lowest in methanol indicates the solubility trend in various solvents. Density of solution is depends on solubility and solvent system both. Increase of density with temperature and mole fraction of methanol indicates higher solubility. Also lower values of υ-OH shows increase of molecular interaction and hence the solubility of hydroquinone.

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