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Solubility of Chrysin in Ethanol and Water Mixtures

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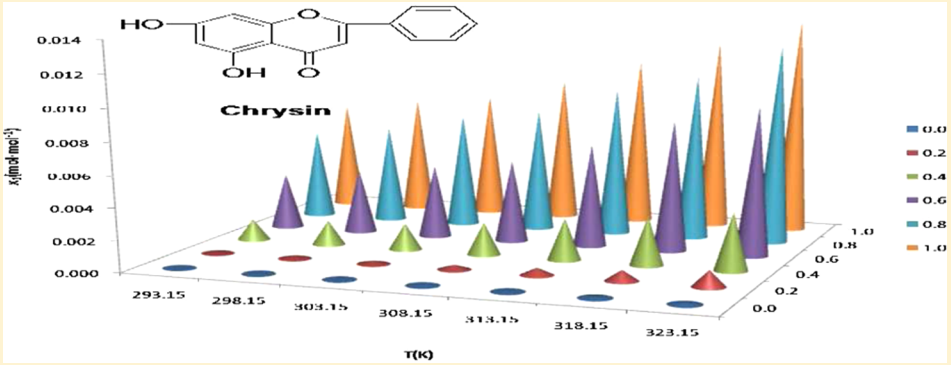

ABSTRACT: Solubility data of chrysin in different solvents are crucial for theoretical research and industrial application. In this work, the solubility of chrysin in the aqueous mixtures conformed by ethanol and water was assayed over the temperature range of (293.15 to 323.15) K using the ultraviolet spectrophotometric method. The experimental data indicated that the solubility of chrysin in ethanol and water mixtures increases with an enrichment in ethanol concentration and an increase in temperature. The modified Apelblat equation and λh equation calculated solubility of chrysin exhibited a great fit with the experimental data. The activity coefficients, thermodynamic functions, dissolution enthalpy, and entropy were acquired from the experimental values. The dissolution process of chrysin within studied temperature range was endothermic, and the driving mechanism is the entropy.

INTRODUCTION

The solubility of chemicals in the solution plays an essential part on determining appropriate solvents and developing industrial production processes.[1] To alter the solubility of chemicals in many processing applications, solvent mixing, one of the most common and feasible methods, should be employed. Lots of solubility data for a given component could be obtained using different ratios of the solvents. Another variable frequently utilized in the industrial production process is the temperature of the system. Finding an optimal solvent composition and the proper temperature in solubility investigations is usually acquired by trial and error; however, it is both time-consuming and costly.[2] Therefore, this research would discuss the solvent and temperature effect on the solubility of the compound.

Chrysin, with the formula $C_{15}H_{10}O_4$ (CAS 480-40-0, 5,7-dihydroxyflavone,[3] shown in Figure 1), is a naturally occurring flavonoid which exhibits a variety of bioactivities.[4,5] It shows anti-inflammatory, antioxidant, and anticancer effects. Furthermore, a recent clinical test has verified chrysin may lead to body weight reduction and lower postprandial blood-glucose.[6] However, chrysin is limited to use in the pharmaceutical field on account of its poor aqueous solubility and scarce absorption.[7] Chakraborty and Basu have investigated that the solubility of chrysin could be improved by encapsulating within β-CD (beta-cyclodextrin) or its derivatives,[8] but the solubility data of chrysin are rarely mentioned in the literature. Moreover, chrysin is usually extracted from the seeds of *Oroxylum indicum* by pure water, ethanol, or mixtures of both.[9] Thus, in this work, the solubility of chrysin in aqueous mixing solvents conformed by ethanol and water was assayed from (293.15 to 323.15) K using a UV spectrophotometric method. The obtained data were correlated with the modified Apelblat equation and λh equation. The solubility values correlated by the models show good agreement with the experimental data. Meanwhile, the activity coefficients and thermodynamic functions, dissolution enthalpy, and entropy calculated from the measured solubility data enable investigators to look at the mathematical respects of solubility.

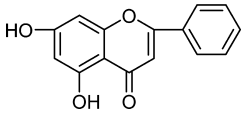

Figure 1. Chemical structure of chrysin.

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EXPERIMENTAL SECTION

Materials and Apparatus. Chrysin sample ($C_{15}H_{10}O_4$, CAS 480-40-0) was provided by Shaanxi Jiahe Phytochem Co., Ltd. (Shaanxi, China). Ethanol was of analytical grade, purchased from Tianjin Kermel Chemical Reagent Co., Ltd. (Tianjin, China), and the redistilled deionized water was employed through the study. Absorbance analysis was performed by UV−vis spectrophotometer (SP-752, Shanghai Spectrum Instrument Co., Ltd., Shanghai, China). A detailed description of chemicals used in the work is shown in Table 1.

Table 1. Purities and Sources of the Chemicals

name	purity (mass fraction)	source
chrysin	≥ 0.98	Shaanxi Jiahe Phytochem Co., Ltd.
ethanol	≥ 0.98	Tianjin Kermel Chemical Reagent Co., Ltd.

Sample Preparation and Analysis. A sealed 10 mL dual-wall flask was applied. Circulated water was filled between the outer and inner walls of the flask with the temperature controlled with a thermostat. First 5.0 mL solvent was put in the flask, and the excess amount of chrysin was added to the solvent. Then the suspension was kept stirring for 4 h. The equilibration time was verified from preliminary experiments performed in different solvent system. When it came to equilibrium state, the stirrer was turned off. After that 1 mL of the suspension was withdrawn and filtered. The known volume filtrate was diluted for ultraviolet measurement. The calibration curve was prepared for calculating chrysin amount with standard solutions in the proper concentration range. The maximum absorption wavelength was set at 275 nm.

Measurements were performed three times with mean values been accepted to estimate the mole fraction solubility of chrysin (x_1) by eq 1. The uncertainty of analysis values was no more than 1.0 %. The solvent composition x_2 could be determined by eq 2.

$$x_1 = \frac{m_1/M_1}{m_1/M_1 + m_2/M_2 + m_3/M_3} \quad (1)$$

$$x_2 = \frac{m_2/M_2}{m_2/M_2 + m_3/M_3} \quad (2)$$

where m_1, m_2, and m_3 denote the masses of chrysin, ethanol, and water. M_1, M_2, and M_3 represent the molecular weights of chrysin, ethanol, and water, respectively.

RESULTS AND DISCUSSION

Measured and Calculated Solubility. Table 2 exhibits solubility values of chrysin in the binary system at different temperatures. The measured data indicate the solubility of chrysin in aqueous mixtures conformed by ethanol and water increases with increasing ethanol concentration and increasing temperature in the range of (293.15 to 323.15) K. Since solid−liquid equilibrium data are usually not available, particularly for mixed solvents, correlation and prediction schemes are

Table 2. Solubilities of Chrysin from (293.15 to 323.15) K in Ethanol and Water Mixtures (P = 0.1 MPa)[a]

	$10^4\ x_1$	$(x_1 - x_1^{cal,Apel})$	$(x_1 - x_1^{cal,\lambda h})$	$10^3\ x_1$	$(x_1 - x_1^{cal,Apel})$	$(x_1 - x_1^{cal,\lambda h})$
T/K	x_2 = 0	x_1/100	x_1/100	x_2 = 0.2	x_1/100	x_1/100
293.15	0.126	−0.7138	15.2713	0.081	0.4272	4.3279
298.15	0.186	3.6130	14.4981	0.130	3.9076	5.2334
303.15	0.238	−5.8140	1.9339	0.185	−4.5233	−4.9085
308.15	0.353	−0.1078	3.9016	0.297	−0.3247	−1.6654
313.15	0.461	−7.2621	−5.7913	0.474	3.8662	2.3764
318.15	0.650	−5.7482	−6.3477	0.691	0.0643	−0.9835
323.15	1.002	4.7570	3.0305	1.043	0.0395	0.2710
	$10^3\ x_1$	$(x_1 - x_1^{cal,Apel})$	$(x_1 - x_1^{cal,\lambda h})$	$10^3\ x_1$	$(x_1 - x_1^{cal,Apel})$	$(x_1 - x_1^{cal,\lambda h})$
T/K	x_2 = 0.4	x_1/100	x_1/100	x_2 = 0.6	x_1/100	x_1/100
293.15	1.321	4.6202	6.9123	3.547	6.3471	8.3649
298.15	1.552	2.7092	3.9805	4.075	2.7225	3.3216
303.15	1.683	−7.3197	−6.9654	4.654	−1.5998	−1.8379
308.15	2.068	−4.3219	−4.9393	5.276	−6.8407	−7.6513
313.15	2.648	2.8376	2.2010	6.567	−2.2609	−3.0931
318.15	3.082	0.5546	0.0627	8.317	3.8888	3.5734
323.15	3.655	0.2767	0.4214	9.466	−0.4420	0.1724
	$10^2\ x_1$	$(x_1 - x_1^{cal,Apel})$	$(x_1 - x_1^{cal,\lambda h})$	$10^2\ x_1$	$(x_1 - x_1^{cal,Apel})$	$(x_1 - x_1^{cal,\lambda h})$
T/K	x_2 = 0.8	x_1/100	x_1/100	x_2 = 1.0	x_1/100	x_1/100
293.15	0.569	4.3339	5.7839	0.689	4.6468	5.8966
298.15	0.623	−0.1304	0.2124	0.749	1.0212	1.3605
303.15	0.728	1.6130	1.1702	0.780	−4.7508	−5.1673
308.15	0.783	−4.7876	−5.5733	0.928	−1.9809	−2.6906
313.15	0.943	0.2162	−0.3950	1.076	0.6131	−0.0031
318.15	1.052	−2.5742	−2.7549	1.213	0.3887	0.2509
323.15	1.266	2.3177	2.9401	1.372	0.5080	1.1124

[a] x_1 stands for the experimentally mole fraction solubility; $x_1^{calc,Apel}$ and $x_1^{calc,\lambda h}$ are the calculated solubilities according to eq 3 and the λh equation, respectively; x_2 is the solvent composition. The relative standard uncertainty for pressure is 0.05, for temperature is 0.1 K, for x_1 is 0.02, and for x_2 is 0.005.

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Table 3. Parameters of eq 3 for the Solubility of Chrysin in Ethanol and Water Mixtures

x_2	0	0.2	0.4	0.6	0.8	1.0
A	−170.7465	−167.0647	−105.4596	−122.1300	−107.2340	−100.7260
B	1906.4690	373.0405	1684.9551	2518.9810	2475.6870	2423.7250
C	26.9274	27.5252	16.3775	18.9818	16.4725	15.3912
R^2	0.9856	0.9935	0.9885	0.9835	0.9849	0.9886
10^5 rmsd	0.27	0.79	7.04	21.62	23.15	20.63

Table 4. Parameters of λh Equation for the Solubility of Chrysin in Ethanol and Water Mixtures

x_2	0	0.2	0.4	0.6	0.8	1.0
λ	0.8655	46.7455	0.3112	0.7570	0.3474	0.2518
h	8064.9632	175.8655	10990.4600	4447.6630	7433.4000	9022.1450
R^2	0.9903	0.9942	0.9892	0.9833	0.9829	0.9867
10^5 rmsd	0.26	0.70	7.56	24.10	27.52	24.86

frequently utilized. The temperature dependence of chrysin solubility in ethanol and water mixtures could be defined by empirical eq 3.[10,11]

$$\ln(x_1) = A + \frac{B}{T} + C\ln(T) \tag{3}$$

where x_1 is the molar fraction solubility of chrysin, A, B, and C are the parameters, and T is the absolute temperature. The discrepancies between measured data and eq 3 calculated ones presented in Table 2 are provided by $(x_1 - x_1^{cal,Apel})/(x_1/100)$. Table 3 lists the values of A, B, and C together with determination coefficient R^2.

The λh equation proposed initially by Buchowski et al.[12,13] is also an optional way to describe solution behavior. The λh equation has only two adjustable parameters, λ and h, which give it versatility in correlating the experimental values in many solvents. The equation can be described as follows:

$$\ln\left[1 + \frac{\lambda(1 - x_1)}{x_1}\right] = \lambda h\left(\frac{1}{T} - \frac{1}{T_m}\right) \tag{4}$$

where T stands for the absolute temperature and T_m represents the normal melting temperature, for chrysin T_m = 558.15 K.[14] Table 4 lists the values of λ and h, together with the determination coefficient R^2. The values of λ and h are different in different solvent systems, and the large difference of values of λ and h present in 0.2 molar fraction of ethanol probably indicate the variation of dissolution enthalpy and entropy. In addition, Table 2 summarizes the discrepancies provided by $(x_1 - x_1^{cal,\lambda h})/(x_1/100)$ between the measured solubility and the λh equation calculated data.

The root-mean-square deviation is defined as

$$\text{rmsd} = \sqrt{\frac{\sum_{i=1}^{N}(x_i^{calc} - x_i)^2}{N}} \tag{5}$$

where N is the number of measured points, x_i^{calc} stands for the calculated solubility, and x_i stands for the experimental solubility values. For eq 3 and the λh equation, the values of rmsd are presented in Table 3 and Table 4, respectively. Figures 2 to 4 and Figures 5 to 7 show the measured solubility of chrysin in the investigated temperature range together with calculated values for eq 3 and the λh equation, respectively.

From the comparison of eq 3 and λh equation model, both of them have good agreement for the chrysin solubility values. The λh equation model was widely accepted previously to fit the solid−liquid equilibria experimental data because of its

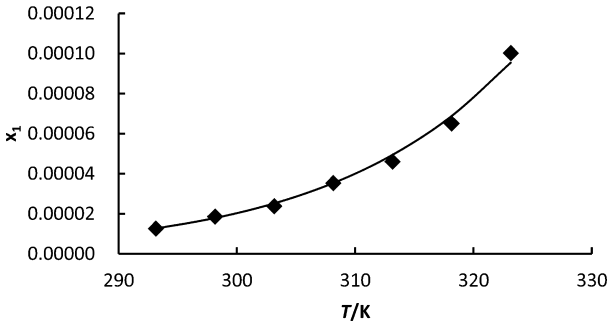

Figure 2. Solubility of chrysin (x_1) in solvent composition x_2 = 0 correlated by eq 3.

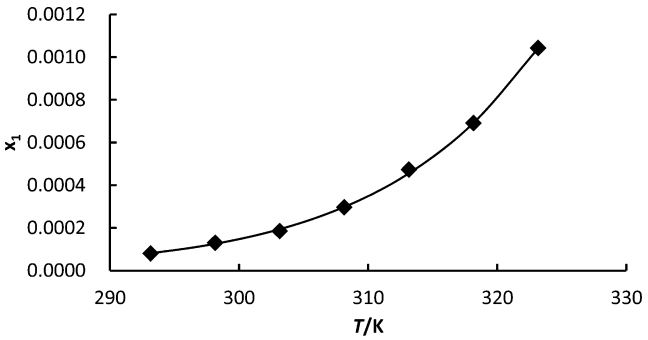

Figure 3. Solubility of chrysin (x_1) in solvent composition x_2 = 0.2 correlated by eq 3.

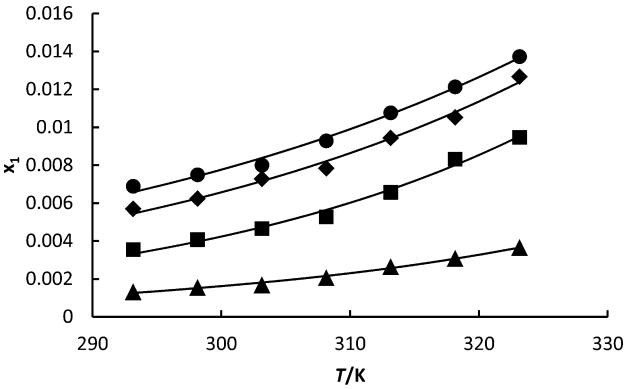

Figure 4. Solubility of chrysin (x_1) in solvent composition x_2 = ▲, 0.4; ■, 0.6; ◆, 0.8; ●, 1.0, correlated by eq 3.

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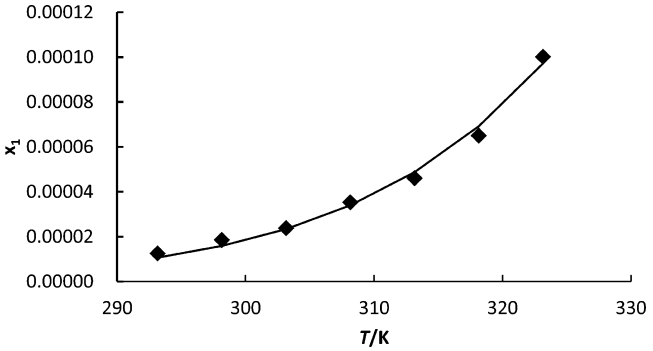

Figure 5. Solubility of chrysin (x_1) in solvent composition x_2 = 0 correlated by the λh equation model (eq 4).

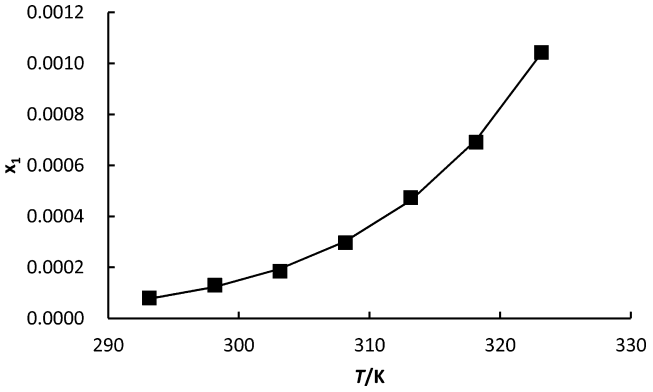

Figure 6. Solubility of chrysin (x_1) in solvent composition x_2 = 0.2 correlated by the λh equation model (eq 4).

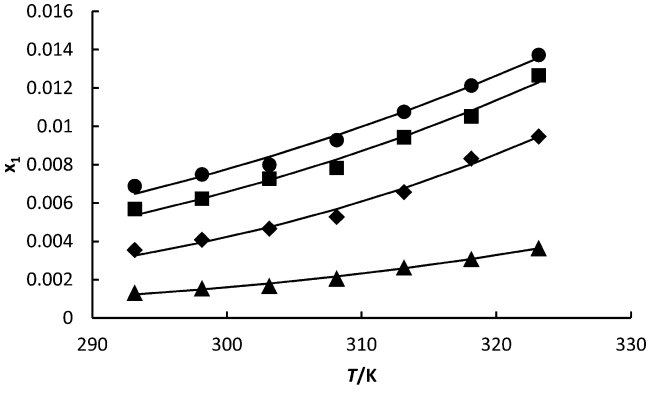

Figure 7. Solubility of chrysin (x_1) in solvent composition x_2 = ▲, 0.4; ◆, 0.6; ■, 0.8; ●, 1.0, correlated by the λh equation model (eq 4).

capability of handling strong polarity systems, which suggest strong interaction between molecules.[15] Thus, the regression result of λh equation model was more accurate in water-rich solvents. As the ethanol concentration decreases, the modified Apelblat eq 3 model also indicates a greater fit for the chrysin experimental solubility data. These small discrepancies could be found by comparing the values of R^2 and rmsd of both equation models.

Activity Coefficients. From the activity coefficient (γ) values a rough estimate of the solute−solvent intermolecular interactions could be taken by eq 6, which is not general but based on the regular solution theory.

$$\ln \gamma = (e_{11} + e_{22} - 2e_{12})\frac{V_1\phi_2^2}{RT} \quad (6)$$

where e_{11}, e_{22}, and e_{12} represent the solute−solute, solvent−solvent, and solute−solvent interaction energies, respectively, V_1 is the mole volume of supercooled liquid solute, φ_2 is the volume fraction of the solvent, and R stands for gas constant. As a first approximation, for relatively low solubilities (x_1), the term $V_1\varphi_2^2/RT$ may be considered as constant: therefore, γ relies mainly on e_{11}, e_{22}, and e_{12} terms. The e_{11} and e_{22} terms are unfavorable for solubility; on the contrary the e_{12} term favors the solution process. The contribution of the e_{11} term could be considered as constant in all mixtures we used.[16]

The activity coefficients of chrysin in different solvents at the experimental temperatures are calculated based on eq 7.[17] The results are listed in Table 5.

$$\ln \frac{1}{x_1\gamma} = \frac{\Delta H_F}{RT_m}\left(\frac{T_m}{T} - 1\right) \quad (7)$$

Table 5. Chrysin Activity Coefficients in Ethanol and Water Mixtures at Investigated Temperatures

T/K	x_2 = 0	x_2 = 0.2	x_2 = 0.4	x_2 = 0.6	x_2 = 0.8	x_2 = 1.0
293.15	38.40	5.99	0.37	0.14	0.08	0.07
298.15	34.04	4.85	0.41	0.16	0.10	0.08
303.15	34.44	4.42	0.49	0.18	0.11	0.10
308.15	29.90	3.55	0.51	0.20	0.13	0.11
313.15	29.27	2.85	0.51	0.21	0.14	0.13
318.15	26.26	2.47	0.55	0.21	0.16	0.14
323.15	21.44	2.06	0.59	0.23	0.17	0.16

where γ stands for activity coefficient of chrysin; T_m and ΔH_F are the melting temperature and enthalpy of fusion, respectively, which could be obtained from the literature.[14] As a qualitative approach, the following analysis can be taken on the basis of the energetic quantities and magnitudes described in eq 6: the term e_{22} is higher in pure water and smaller in ethanol.[18] Neat water and water-rich mixtures which have higher γ values (even higher than 30) would imply high e_{22} and low e_{12} values. On the other hand, in ethanol-rich mixtures (having γ values between 0.07 and 0.16), the e_{22} values are relatively low, and the e_{12} values would be relatively high. Thus, the solubility of chrysin would be higher in ethanol-rich mixtures. Interestingly, we find that γ values are highly dependent on temperature. It is obvious that the activity coefficients have a diminishing trend as the temperature increases in water-rich mixtures (x_2 = 0, 0.2), and an incremental trend with the ethanol concentration (x_2 from 0.4 to 1.0) and temperature (from 293.15 to 323.15 K) increasing. Therefore, this solution processes become "more ideal".[16]

Thermodynamic Functions of Solution. The van't Hoff equation for real solutions is concerned with the logarithm of molar fraction of a solute as a linear function of the reciprocal of the absolute temperature:

$$\ln x_1 = -\frac{\Delta H_F}{RT} + \frac{\Delta S_F}{R} \quad (8)$$

where x_1 is the molar fraction solubility of the solute, ΔH_F represents the molar enthalpy of fusion of the solute, ΔS_F stands for the molar entropy of fusion of the solute, T

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represents absolute temperature, and R stands for the gas constant.

As a matter of fact, most real solutions present nonideal behavior. Thus, the solvent effect must be thought, which indicates that the enthalpy and entropy of the mixtures should be taken into consideration by replacing ΔH_F with ΔH_d (enthalpy of dissolution) and ΔS_F with ΔS_d (entropy of dissolution), following:

$$\ln x_1 = -\frac{\Delta H_d}{RT} + \frac{\Delta S_d}{R} \quad (9)$$

The van't Hoff plots present in Figure 8 can be made from the linear fit of ln x_1 against $1/T$. The dissolution enthalpy and

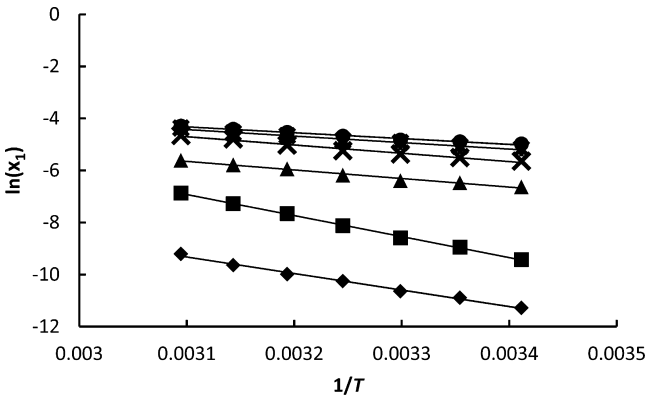

Figure 8. Van't Hoff plots of ln x_1 versus $1/T$ in different solvents x_2 = ◆, 0; ■, 0.2; ▲, 0.4; ×, 0.6; ∗, 0.8; ●, 1.0.

entropy of chrysin listed in Table 6 could be estimated from the slope and the interception of the lines, respectively. In general, our measurements exhibit dissolution enthalpy and entropy of chrysin decreases with an increase in ethanol molar fraction of the solvents, except the ones in the 0.2 mole fraction of ethanol solvent.

It can be seen that the dissolving process of chrysin in the aqueous mixtures conformed by ethanol and water was endothermic over the investigated temperature range (according to Gibbs–Helmholtz equation: $\Delta G = \Delta H - T\Delta S$, all $\Delta G > 0$). The values of ΔH_d and ΔS_d imply that dissolution of chrysin in the binary solvent of ethanol and water mixtures was an entropy-driving course. At certain temperatures, the decreased entropy increases solubility with an increase in ethanol concentration in the binary system.

CONCLUSIONS

The solubility data of chrysin in water and ethanol mixtures was assayed from (293.15 to 323.15) K by the UV spectrophotometric method. The measured data were correlated with eq 3 and the λh equation. Both of the models provide great representations of the experimental data. At the same time, the solubility activity coefficients of chrysin calculated from the measured data are highly dependent on temperature, and the values of ΔH_d and ΔS_d indicated dissolution of chrysin in the binary solvent of ethanol and water mixtures was an entropy-driving course. Overall, this study offered valuable information for the extraction or purification fields of chrysin in the industry and could be used for industrial design or theoretical studies.

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Notes

The authors declare no competing financial interest.

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Table 6. Dissolution Enthalpy and Entropy of Chrysin in Ethanol and Water Mixtures[a]

x_2	0	0.2	0.4	0.6	0.8	1.0
ΔH_d (kJ·mol^{-1})	52.793	67.26	27.39	26.46	20.80	18.67
ΔS_d (J·mol^{-1}·K^{-1})	86.05	150.90	37.55	42.93	27.67	21.95
r	0.997	0.999	0.992	0.991	0.992	0.991

[a] ΔH_d stands for the dissolution enthalpy, and ΔS_d represents the dissolution entropy.

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