

Solubility of Cis, Cis-Muconic Acid in Various Polar Solvents from 298.15 K to 348.15 K

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ABSTRACT: *The present work concerns with an investigation on the solubility of cis,cis-muconic acid dissolution in different polar solvents by characterizing and modeling the dissolution as a function of temperature. Water, ethanol, 2-propanol and acetic acid have been investigated as solvents in the range temperatures from a 298.15 to 348.15 K. Owing to the absence of cis,cis-muconic acid solubility data, the molar fraction and temperature were correlated using the modified Apelblat equation model, which is applied for the mathematic fitting of experimental data. A total relative average deviation of 3.54% was obtained for the experimental results and the solubility data obtained with the model, thus attesting the adequacy for this study. The use of modified Apelblat equation also allowed to determine the molar enthalpy and molar entropy of dissolution. The dissolution of cis,cis-muconic acid in water, ethanol, 2-propanol and acetic acid, over temperatures ranging from 298.15 to 348.15 K, has been shown to be exothermic.*

KEYWORDS: *Cis; Cis-Muconic Acid; Solubility; Modified apelblat equation; Dissolution enthalpy; Dissolution entropy.*

INTRODUCTION

Cis,Cis-Muconic Acid (CCMA) is a diunsaturated dicarboxylic acid with six carbon atoms. This compound presents characteristics that could make it useful as a raw material for new functional resins, pharmaceuticals and agrichemicals [1]. Unfortunately, its application has been limited because there are no safe or reliable methods to synthesize large-scale amounts of CCMA.

Nevertheless, in the last few years, attempts to obtain CCMA by means of microbial approaches have been reported. One of the most interesting of these attempts concerns the batch fermentation of D-glucose, through which CCMA has been obtained with a 22% molar yield after culturing modified *Escherichia coli* for 48 h [2].

Another way of obtaining CCMA is to produce CCMA from toluene using a mutant strain of *Pseudomonas putida*, without CCMA degrading enzymes. The mutant strain has been shown to accumulate 5 g/l of the product after 3 h of activity [3]. Other substrates have also been investigated: the production of CCMA from benzoic acid, using a mutant strain of *Corynebacterium glutamicum* or *Pseudomonas*, has been reported [4-6]. In the first case, the productivity and yield of CCMA were rather low, while in the second one, an almost 100% yield was reached. Furthermore, two studies have been conducted on the production of CCMA from catechol, even though it is very toxic and much more expensive than the other

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listed substrates [7, 8]. CCMA can also be obtained from biorefinery process intermediates and from the selective conversion of lignin, in which unsaturated oxygenated compounds, such as quinones, are converted to CCMA [9]. CCMA production, through, for instance, glucose fermentation, is carried out in an aqueous phase, and CCMA is generated as muconate. In order to recover CCMA from fermentation broth, after previous purifications, it has to be protonated; the CCMA crystals can then be filtered [2]. Generally, crystallization represents one of the preferred methods for the recovery of products after transformation; therefore, the solubility of a compound in a liquid medium plays a primary role in the recovery of materials [10, 11].

CCMA is currently of interest because it represents a precursor for adipic acid production. In fact, CCMA can easily be hydrogenated into adipic acid under relatively mild conditions [2]. The reaction is carried out using noble metal supported catalysts, using water or ethanol as solvents [2, 12, 13]. Recently, also a Ni-based catalyst was tested for the CCMA hydrogenation to adipic in aqueous mean [14]. On the other hand, all the reactions were carried out at higher temperatures than ambient, so, in order to predict the solubility of CCMA in the reactive mixture, a CCMA solubility evaluation becomes really interesting. Adipic acid is today one of the most important chemicals, and the 2.6×10^6 tons annually produced are mainly used for the manufacturing of nylon 66 [15]. Adipic acid is also used for the production of gelatins, jams, polyamides, polyurethanes and lubricants [16].

The development of the solution theory necessarily requires the study of substance solubility [17, 18]. Unfortunately, there is a lack of published data on the solubility of organic compounds at different temperatures and pressure, and this shortage of data refers to CCMA [19-23]. Because of the nature of the compound, its solubility is not predictable. The most reliable way of obtaining solubility data is through experiments, even though they require appropriate apparatus and method [24].

In order to correlate the solubility data of certain substances, pertaining to the molar enthalpy and molar entropy of dissolution, the modified Apelblat equation model is usually applied [25-27]. This equation is generally called modified *Apelblat* equation because it was firstly adopted by *Apelblat* in several publications [28]. In fact, *Sun et al.* used the *Apelblat* equation model

for the calculation of the adipic acid solubility in various polar solvents at the temperature ranging from 303.0 to 403.0 K. *Sunsandee et al.*, instead, determined 4-acetylbenzoic acid solubility and thermodynamic data of solubility in different solvents from 303.15 to 473.15 K while *Zhang et al.*, studied the thermodynamics of the solubility of sulfamethazine in methanol, ethanol, 1-propanol, acetone, and chloroform from 293.15 to 333.15 K. Also the solubility of several organic acids were evaluated from 278.15 to 338.15 K in a water mean [19].

In this study, the solubility of CCMA was calculated in various solvents, such as water, ethanol, 2-propanol and acetic acid, over a temperature range from 298.15 to 348.15 K. The modified *Apelblat* equation model was employed to mathematically correlate the CCMA solubility data. The model was checked by matching the experimental results with the solubility data obtained from the model. The modified *Apelblat* equation model was claimed as a fast and easy method for obtaining acceptable solubility data as a function of temperature; furthermore, it provides simplified thermodynamic equations for the calculation of the enthalpy and entropy of dissolution.

EXPERIMENTAL SECTION

All the chemicals, that are, CCMA ($C_6H_6O_4$, MW 142,11 g/mol, melting point 467.15 – 468.15 K, HPLC assay $\geq 97\%$ [29], water (CHROMASOLV® Plus), ethanol (ACS reagent, $\geq 99.8\%$), 2-propanol (ACS reagent, $\geq 99.5\%$), acetic acid (Glacial, ReagentPlus®, $\geq 99\%$), were purchased from Sigma-Aldrich.

An excess of solid CCMA was introduced into a 250 mL glass flash tank, and 25 mL of solvent was added. A Teflon® sampler, able to hold a volume of 0.5 mL, was introduced inside the chamber and the tank was sealed. The temperatures were set by means of a thermostated bath of silicon oil. The temperature was controlled using a thermocouple with an uncertainty of ± 0.1 °C. As described by *S. Suren et al.* [30, 31], once the temperature was reached, the mixture was stirred, by means of magnetic agitation, for 90 minutes, then the system was maintained in a static state for 180 minutes in order to allow the deposition of suspended particles and to obtain a homogeneous solution. After 180 minutes, the Teflon® sampler was introduced into the liquid and the 0.5 mL of solution was trapped in the hole of the sampler.

Table 1: Molar fraction of CCMA solubility (x_2) in solvents at temperatures (T) ranging from 25.0 to 75.0 °C (298.15 – 348.15 K)^a.

T (°C)	T (K)	Water	Ethanol	2-propanol	Acetic Acid
25.0	298.15	0.000	0.008	0.010	0.003
30.0	303.15	0.000	0.010	0.011	0.003
35.0	308.15	0.001	0.016	0.012	0.004
40.0	313.15	0.001	0.018	0.014	0.004
45.0	318.15	0.001	0.021	0.016	0.005
50.0	323.15	0.002	0.027	0.017	0.006
55.0	328.15	0.003	0.034	0.020	0.007
60.0	333.15	0.006	0.048	0.024	0.008
65.0	338.15	0.007	0.068	0.027	0.009
70.0	343.15	0.009	0.101	0.030	0.010
75.0	348.15	0.015	0.168	0.034	0.013

^aStandard uncertainties u are $u(T) = 0.1$ °C, $u_r(x) = 0.01$

The solute amount was evaluated by means of gravimetric measurement after having calculated the solvent evaporation and the molar fraction according to the molecular weight. Washing procedures were carried out in order to ensure that only the solute, trapped inside the Teflon® sampler hole, was weighed [30]. All the experiments were replicated three times.

An analytical balance (Radwag, AS220.R2) was used for the gravimetric measurements and mass values were generated with an uncertainty of ± 0.1 mg. The liquid was analyzed by means of high performance liquid chromatography (HPLC) analysis. The equipment (Shimadzu Instruments) was constituted by a pump and an auto-sampler, which injected a volume of sample equal to 10 μ l into the column; the sample was made up of REZEX-Organic Acid ROH⁺ column (Phenomenex Inc.). A sulfuric acid solution (5 mmol/L in water) was used as the mobile phase, and a flow of 0.6 mL/min was used to elute the compounds in 30 minutes. The column temperature was 343.15 K and the pressure was around 43 bars. A photodiode array was used as a detector at a wavelength of 260 nm and an external standard calibration was generated to quantify the CCMA.

RESULTS AND DISCUSSION

Solubility of CCMA in the studied solvents

The mole fractions of CCMA, calculated over a range of temperatures from 298.15 to 348.15 K, increased with

the temperature for all the solvents (Table 1). So, the solubility of CCMA depended directly on the temperature, in a specific manner with respect the solvent used. Generally, the solubility of a compound depends by the net contribution of the physical properties of a solvent. It has been demonstrated how, for instance, polarity or molecular weight of solvents could be cause of difference in solubility [32-34]. Anyways, the net contribution of all the physical properties determines resulting behavior of a solid dissolved in a liquid. Nevertheless, to discern the contribution of singular property it could be arduous [35].

From Table 1, it is clear how CCMA was more soluble in the alcoholic solvents. Actually, due to the presence of two carboxylic groups, greater affinity with water or acetic acid was expected, but higher molar fractions of solubility were instead obtained using 2-propanol and ethanol; even more, the latter was the best solvent for CCMA dissolution. Thus, it can be seen from the results that the CCMA solubility is not predictable among the employed solvents. Neither correlations between solubility and molecular weight or solvent polarity were found: according to [36], the polarities for the selected solvents are listed as follows: 2-propanol < ethanol < acetic acid < water. So, this polarity order does not reflect the effective solubility order (ethanol > 2-propanol > acetic acid > water).

It can be seen, from the experiments that CCMA suffered from interaction with polar solvent when heated

Solvents	A	B	C	RAD (%)
Water	-81.77	-3924.92	15.18	3.19
Ethanol	-83.20	-15.17	13.80	4.66
2-propanol	-53.40	24.87	8.54	2.41
Acetic Acid	-57.14	-167.86	9.09	3.90
Total relative average deviation (%)				3.54

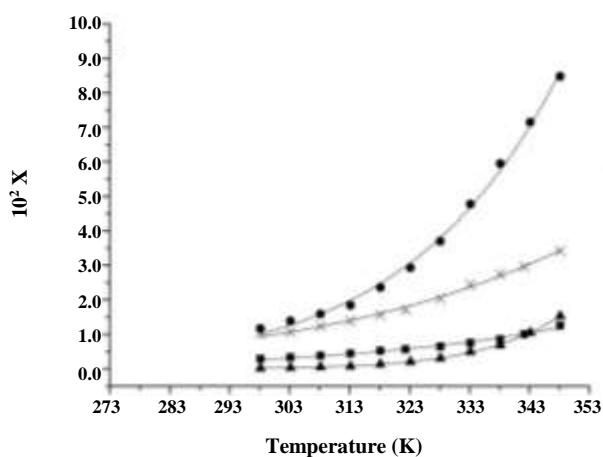


Fig. 1: Experimental data of CCMA solubility (x_i) at different temperatures fitted with the modified Apelblat equation model using (\blacktriangle) water, (\bullet) ethanol, (\times) 2-propanol and (\blacksquare) acetic acid. The table reports the resulting parameters (A, B and C) of the determined modified Apelblat equation model of CCMA solubility and the Relative Average Deviation (RAD) calculated for the model validation.

to over 348.15K in all the investigated organic solvents or in water. So, solubility measurements of CCMA at higher temperature of 348.15K were no possible to perform.

Thermodynamic parameters

The modified Apelblat equation is an effective method to forecast the concentration of an organic compound in saturated solutions. In order to employ the modified Apelblat equation, it is necessary to assume that the enthalpy of the solution depends directly on the temperature [37, 38]. The modified Apelblat equation is shown below:

$$\ln x_i = A + B/T + C \times \ln(T) \quad (1)$$

where x_i is the mole fraction of CCMA in the solution, the A, B and C values are extracted by curve fitting of the experimental data with a non-linear optimization method [30] and T is the temperature in K. The experimental data of CCMA solubility in

the explored solvents are represented in Table 1, while the A, B and C parameters, determined as described above, are reported in Fig. 1.

Other works have validated the modified Apelblat equation using the Relative Average Deviation (RAD), calculated as in Eq. (2) [25, 31]:

$$\text{RAD} = \frac{1}{N} \sum_{i=1}^N \left| \frac{x_{\text{exp}} - x_{\text{cal}}}{x_{\text{exp}}} \right| \times 100\% \quad (2)$$

Where N is the number of experimental data; x_{exp} the value of the CCMA molar fraction calculated experimentally; x_{cal} the molar fraction obtained from the fitted curve.

Fig. 1 shows the experimental points and curve fitting using the modified Apelblat equation of CCMA molar fractions in water, ethanol, 2-propanol and acetic acid at temperatures ranging from 298.15 K to 348.15 K. On the basis of the RAD results and from Fig. 1, it seems clear that Eq. (1) fitted the experimental data well. Furthermore, the RAD was between a maximum value of 4.66% and a minimum one of 2.41%, thus demonstrating the reliability of the model. The modified Apelblat equation can therefore be considered a reliable model to predict the solubility of CCMA for the investigated polar solvents.

The molar enthalpy and molar entropy of dissolution ($\Delta_{\text{sol}}H$, $\Delta_{\text{sol}}S$) of the solid solutes were also estimated with the Apelblat equation [25]. They were calculated after determining the equilibrium constant (K_i); K_i was obtained from the relationship between the dissolution activities, conforming the process of dissolution to a chemical reaction [39]. Eq. (3) shows the mathematical equation of the equilibrium constant:

$$K_i = \frac{a_i}{a_s a_w} \quad (3)$$

Where a_i is the CCMA activity in the solution, while a_s and a_w are the pure solid and pure liquid activities, respectively. Since the standard solid and liquid states, a_s and a_w , were considered constant, and K_i described as below [25]:

$$K_i = \frac{\gamma_i x_i}{a_s a_w} \quad (4)$$

Where γ_i is the activity coefficient of the CCMA in the solution.

Table 2: Molar enthalpy of dissolution ($\Delta_{sol}H$) and molar entropy of dissolution ($\Delta_{sol}S$) of CCMA in the investigated solvents.

T (°C)	T (K)	$\Delta_{sol}H$ (kJ/mol)				$\Delta_{sol}S$ (J/mol K)			
		Water	Ethanol	2-propanol	Acetic Acid	Water	Ethanol	2-propanol	Acetic Acid
25.0	298.15	-70.26	-34.34	-20.96	-23.93	235.67	115.16	70.31	80.26
30.0	303.15	-70.90	-34.91	-21.32	-24.31	233.86	115.16	70.32	80.18
35.0	308.15	-71.53	-35.48	-21.67	-24.69	232.12	115.15	70.33	80.11
40.0	313.15	-72.16	-36.06	-22.03	-25.06	230.42	115.14	70.35	80.04
45.0	318.15	-72.79	-36.63	-22.38	-25.44	228.79	115.14	70.36	79.97
50.0	323.15	-73.42	-37.20	-22.74	-25.82	227.20	115.13	70.37	79.90
55.0	328.15	-74.05	-37.78	-23.09	-26.20	225.66	115.12	70.38	79.83
60.0	333.15	-74.68	-38.35	-23.45	-26.57	224.17	115.12	70.38	79.77
65.0	338.15	-75.31	-38.93	-23.80	-26.95	222.72	115.11	70.39	79.71
70.0	343.15	-75.94	-39.50	-24.16	-27.33	221.31	115.11	70.40	79.65
75.0	348.15	-76.57	-40.07	-24.51	-27.71	219.95	115.10	70.41	79.59

Assuming that γ_i is independent of the temperature and composition, Eq.(4) was/can be written in logarithmic form:

$$\ln K_i = \ln x_i + J \quad (5)$$

where $J = \ln \gamma_i - \ln a_{s,w}$ and J is also a constant that is independent of the temperature.

Using the modified Vant't Hoff method and the principle of the Gibbs equation, it was possible to determine the molar enthalpy of dissolution [40, 41]:

$$\Delta_{sol}H = -R \frac{d \ln K_i}{dT^{-1}} \quad (6)$$

Where R is the gas constant. Thus, the $\Delta_{sol}H$ expression was be calculated substituting Eq. (5) in Eq. (6):

$$\Delta_{sol}H = -R \frac{d \ln x_i}{dT^{-1}} \quad (7)$$

Finally, by substituting the derivative obtained with Eq. (1) in Eq. (7), it was possible to obtain Eq. (8):

$$\Delta_{sol}H = RT(C - B/T) \quad (8)$$

All the temperatures of the molar enthalpy of dissolution are reported in Table 2 for each solvent. From the results, it can be observed that the dissolution of

CCMA in water, ethanol, 2-propanol and acetic acid was exothermic. Thus, the energy of the component was higher than the energy of the blend.

The molar entropy of dissolution has also been calculated. Using the B and C values determined as previously described, and the fundamental thermodynamic relationship, the $\Delta_{sol}S$ of CCMA dissolution was calculated as [39]:

$$\Delta_{sol}S = R(C - B/T) \quad (9)$$

The resulting values of $\Delta_{sol}S$ are shown in Table 2; the entropy data are always below zero. According to the basis of the third law of thermodynamics, the CCMA dissolution in water, ethanol, 2-propanol and acetic acid that presented $\Delta_{sol}H < 0$ and $\Delta_{sol}S > 0$, are all favorable processes [36].

CONCLUSIONS

In this study, the solubility data of CCMA in polar solvents, such as water, ethanol, 2-propanol and acetic acid, have been calculated over a temperature range from 298.15 to 348.15 K. The modified Apelblat equation model has been applied to correlate the solubility data of the CCMA. Matching the experimental results with the solubility data obtained with the model, a total relative average deviation of 3.54% was obtained. The mole

fractions of CCMA, calculated over a range of temperatures from 298.15 to 348.15 K, increased with the temperature for all the solvents (Table 1). In all cases the solubility of CCMA increased with temperature and it can be seen from the solubility results that CCMA solubility order was ethanol > 2-propanol > acetic acid > water. Therefore, ethanol was the best solvent for CCMA dissolution. The molar enthalpy and molar entropy of dissolution of the solid solutes have also been estimated using the modified Apelblat equation. The dissolution of CCMA in the polar solvents has been exothermic and a favorable process for all the solvents, over a 298.15 to 348.15 K temperature range.

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