Investigation of Thermodynamic Equilibria of Vitamin C in Various Solvents Via Experimental Determination and Model Correlation

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ABSTRACT

Vitamin C plays an essential role in pharmaceutical industry. In general, the solubility of this vitamin in different environments is an important factor for drug development processes. Indeed, solid-liquid equilibrium (SLE) determination of the interested compound in various solvents needs high-precision determination which, however, frequently consumes a lot of time and labor work. In this study, we have developed a simple approach that allows detecting quickly solubility and other important thermodynamic data of dissolution processes. In fact, this approach bases on a combination of the polythermal SLE experimental-determination and the modified Apelblat model. Besides water as a single solvent, different alcohols will be used in this work. For all studied media, dissolution processes are found as endothermic with positive values of enthalpy of dissolution. Furthermore, different alkyl groups have shown influence on the solubility of this compound. Indeed, vitamin C tends to dissolve in water stronger than in ethanol and 2-propanol that relates to polarity properties and hydrogen bonding formation. The results in this contribution gain a good agreement comparing to the reported values from the literature.

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KEYWORDS

SLE; Apelblat model; Vitamin C; Polythermal method; Water and alcohols.

1. Introduction

Crystallization is considered as one of the most important units in chemical and biotechnology [1,2]. In these particulate processes, solid formation from liquid phases will be occurred when the system reaches to supersaturated states. The interface between super-saturated and under-saturated states is known as so called solid-liquid equilibrium (SLE) which is actually corresponding to the saturated solution. For particulate processes such as crystallization, SLE plays an essential role that allows operating and optimizing the designed processes by controlling temperature and/or concentration profiles. Furthermore, SLE is also important for drug formulation since it supplies information about solubility of the medicine in different media such as water or the environment inside human body [3,4]. There are plenty of needed data which are required for process monitoring and controlling, e.g. nucleation, particle growth, agglomeration, breakage, etc. [5,6]. Above all, SLE data are always needed as fundamental inputs, for those, sustainable procedures for high-precision SLE determination are highly required. In practice, experimental SLE determination is a time consuming task which could be accelerated with assistant of the computational science. Nowadays, SLE correlation models such as NRTL, UNIQUAC, COSMO-SAC, etc. become more popular [7,8] and the modeling approach significantly helps to reduce experiment efforts.

Vitamin C is selected in this work as a case study. This compound has been frequently used in many applications, particularly in pharmaceuticals, food industry, etc. [9,10]. Vitamin C has been commercialized in various forms including tablet, capsule, solution, etc. According to many reports, vitamin C shows strong effects in different fields relating to antioxidant agents, cosmetic, improving immune system [9-12]. Thus, vitamin C is a multiple function compound, however, its thermodynamic equilibrium data are relatively limited. Among of them, Neto et al. published several solubility data, nevertheless, these reported values were measured just in a narrow temperature range (i.e. from 25 to 35
and exploited a costly method (i.e. isothermal solubility measurements) [13]. Therefore, an alternative approach is suggested and a wider temperature range is also highly interested. In general, there are two approaches to determine solubility, which are briefly introduced as follows.

At first, the isothermal method is a well-known technique [14]. This method is applied by storing a solid-liquid suspension sample at a fixed temperature under ideal stirring conditions for a sufficient period in order to get the equilibrium state. The basic concept of this method is described in Figure 1a. Isothermal solubility measurements have advantages in terms of not only using simple apparatus but also able to determine simultaneously the compositions of solid and liquid phases at the equilibrium. However, this classical method is sometimes disfavored due to its intrinsic limitations. Practically, these measurements are very time consuming that always requires many sub-steps. For instance, after establishing equilibrium, solid-liquid phase separation must be carried out quickly to avoid side effects such as evaporation, or recrystallization (especially for determination of SLE at high temperature and volatile solvents). Furthermore, absolute drying step is also required to remove the solvent completely out of the solid sample to measure its dry weight, etc. Normally, applying the isothermal method, the time requirement for each experiment is about 2-5 days to obtain a single experimental value depending on different systems. Obviously, such a complex process contains high-potential risk which could lead to large systematic errors.

At second, in contrast to the isothermal method, the polythermal method bases on another concept. For a given solid-liquid composition, this suspension is gradually heated to dissolve slowly all solid particles. The target is determination of the final temperature when the solid phase completely dissolves even the smallest particles [14]. In practice, we determine the light transmittance of the suspension when a laser beam is continuously shone through the studied sample under well-stirred and slowly-heated conditions. The intensity of the transmittance is digitized by the optical sensor and the results are collected continuously every 1 second interval. On the one hand, this procedure has the advantage of rapidly determining SLE, which usually takes only a few hours for a measurement. On the other hand, this method has disadvantages such as being only suitable for systems possessing a dissolution rate faster than the heating rate applied for the system. Furthermore, determining the solid phase composition at equilibrium is not an easy task. Therefore, in many cases, independent experiments are required to obtain additional information from the solid phase regarding phase-transition between different polymorphs and/or solvates, etc. Theoretically, Figure 1b presents the basic concepts of the mentioned method.

In this work, we will develop a simple approach for vitamin C’s SLE determination which not only allows reducing the cost of experiments but also able to be used as a quick tool supporting quantities important thermodynamic data. Firstly, this approach bases on the polythermal concept in order to overcome the disadvantages of the conventional isothermal measurements. Secondly, this approach allows assessing thermodynamic properties including enthalpy and entropy of dissolution processes using the modified Apelblat model. Throughout these analyses, the influence of solvent types on the dissolution of this compound as well as model compatibility will be discussed.

2. Materials and Methods
2.1 Experimental part
2.1.1 Chemicals
The following chemicals were used without any further purification steps: Vitamin C was purchased from Merck (99% purity). Solvents were included ethanol and 2-propanol from Xilong Scientific Co. (Analytical Reagent) and distilled water.

2.1.2 Apparatus

Turbidity change was monitored using a laser source (LD-P5L Hangzhou, 5mW, 680nm) in combination with a digital light intensity module (GY-30 BH1750). Mass of solute and solvent were measured using a Mettler Toledo ME203 (electronic balance). Homogeneity of the suspensions was remained using a magnetic stirrer (IKA CMAG HS7).

2.2 Methodology

In this work, the polythermal measurements were conducted in a system visualized as Figure 2 which is similarly described in the literature [15]. Herein, the major steps are summarized as the following: At first, solid is pre-treated by a fine grinding step in which the initial powder was continuously grinded in 30 min using agate mortar and pestle. Defined amounts of the solid and solvents (prepared according to different designed concentrations) are placed in a 5 mL glass vial to obtain a suspension with a known-composition. This solid-liquid system (5) is placed in a container (1) that is stirred with magnetic fish (6) and placed in a Teflon heating chamber (2). The magnetic stirrer (7) is set to 300 rpm to create a well-mixed suspension and also avoid air bubbles. The temperature is raised slowly with a heating rate of 0.5 K.min⁻¹ thanks to the temperature control system (3). The laser beam (4) has a wavelength of 680 nm and a power of 5 mW, shining through the glass vial containing the studied suspension. Then, the beam is focused on the laser sensor module (10). The laser intensity obtained from the sensor module is then transferred to a computer (9). The internal temperature of the suspension is recorded using an Omron temperature sensor (8) which is connected to the computer as well.

![Figure 2. Setup of polythermal solubility determination [15].](image)

2.3 Model correlation

2.3.1 The modified Apelblat model

Nowadays, thanks to the development of the computational science, various thermodynamic models e.g. NRTL, Wilson, UNIQUAC, and UNIFAC have been frequently applied for correlation of the SLE [7,8]. However, their computational complexities are usually challenges for coding and explaining physical parameters inside these models. Hence, an alternative empirical model i.e. the modified Apelblat [16] is introduced as Equation (1) which calculates SLE based on only experimental data without using any thermodynamic parameters. In this work, this model is recommended for the vitamin C system.

The modified Apelblat model is presented as follows.

\[
\ln x = A + \frac{B}{T, K} + C \ln T, K
\]
\[ x_i = \frac{w_{i,j}}{\sum_{j=1}^{n} w_{i,j} / M_j} \]  

(2)

Where: \( x \), \( w_i \) and \( M \) are mole fraction, mass fraction and molar mass of the constituent components, respectively; \( T \) is the absolute temperature.

Herein, the model parameters \( A, B \) and \( C \) are estimated from solubility data \( (x) \). \( A \) and \( B \) values relate to the variation activity coefficient in solution while \( C \) presents the effect of temperature and relates to the deviation of heat capacity \( \Delta C_p \).

### 2.3.2 Enthalpy andentropy of dissolution

According to the variation of the Gibbs free energy for solution formation, combining Equations (3, 4) allow deriving Equations (5, 6). The Equation (6) shares the similarity to the Apelblat model as introduced in Equation (1). Entropy of an isocratic process is simplified from Equation (7) using isocratic heat capacity change \( \Delta C_p \) which is also assumed to be a constant in a narrow temperature range. From solubility data, parameters \( A, B \) and \( C \) in Equation (1) will be estimated that allow calculating the enthalpy and entropy of dissolution processes in different solvents using Equations (7-9). In these equations, \( R \) is the universal gas constant.

\[ \Delta G_{\text{diss}} = \Delta H_{\text{diss}} - T \Delta S_{\text{diss}} \]  

(3)

\[ \Delta G_{\text{diss}} = -RT \ln K_{\text{eq}} \]  

(4)

\[ \ln K_{\text{eq}} = -\frac{\Delta H_{\text{diss}}}{R \cdot T} + \frac{\Delta S_{\text{diss}}}{R} \]  

(5)

\[ \ln x = -\frac{\Delta H_{\text{diss}}}{R \cdot T} + \frac{\Delta S_{\text{diss}}}{R} + \text{const} \]  

(6)

\[ \Delta S_{\text{diss}} = \int \frac{dQ}{T} = \int \frac{d\Delta H}{T} = \int \frac{\Delta C_p dT}{T} = \Delta C_p \ln T \]  

(7)

\[ \Delta H_{\text{diss}} = -R \cdot B \]  

(8)

\[ \Delta C_p = R \cdot C \]  

(9)

In another way, the enthalpy of dissolution in various solvents can be also determined from the experimental solubility data via the van’t Hoff Equation (10).

\[ \ln x = -\frac{\Delta H_{\text{diss}}}{R \cdot T} + \text{const}. \]  

(10)

### 2.3.3 Parameter estimation

The Matlab was used to determine parameters for the given problem. Parameters were estimated using a nonlinear optimization procedure via lsqnonlin function (Levenberg-Marquardt). Three parameters \( A, B, \) and \( C \) were obtained by minimizing the objective function \( OF \) which was defined as Equation (11). This equation computes the differences between the calculated \( (x_{\text{sim}}^{(i)}) \) and experimental \( (x_{\text{exp}}^{(i)}) \) results of the solute concentration.

\[ OF = \sum_{i=1}^{n} \left( x_{\text{sim}}^{(i)} - x_{\text{exp}}^{(i)} \right) \]  

(11)

### 3. Results and Discussion

Table 1 summarizes measurements of solubility of vitamin C in three single solvents including water, ethanol and 2-propanol according to the polythermal method. Each measurement was repeated at least 3 times to take an average value. The error was found relatively small (less than 2.3%) that proved good applied measurement procedure. This is comparable for another case when polythermal method was applied for L-lactide system as reported in the literature [15] which showed a deviation at a value of 1.5% between the isothermal and polythermal measurements. For visualization and comparison purposes, these data are plotted in Figure 3. Herein, distinguished behaviors of this compound in the investigated solvents were observed.
Table 1. Solubility of vitamin C (x - mole fraction, [-]) determined via the polythermal approach.

| Water | Ethanol | 2-Propanol |
|-------|---------|------------|
| T, (K) | x,(-)   | T, (K) | x,(-) | T, (K) | x,(-) |
| 300.15 | 0.027 | 298.15 | 0.0024 | 297.15 | 0.0011 |
| 305.15 | 0.034 | 312.15 | 0.0067 | 301.15 | 0.0017 |
| 312.15 | 0.055 | 307.15 | 0.0050 | 305.15 | 0.0027 |
| 318.15 | 0.089 | 318.15 | 0.0113 | 314.15 | 0.0061 |
| 328.15 | 0.159 | 322.15 | 0.0132 | 322.15 | 0.0105 |
|       |        | 331.15 | 0.0266 | 326.15 | 0.0132 |
|       |        |         |         | 333.15 | 0.0257 |

Figure 3. Solubility (x-mole fraction) of vitamin C in water, ethanol and 2-propanol; In these solvents, open symbols present experiment data (“-poly.”) and the literature data are denoted by “-iso.”. Lines were plotted from the modified Apelblat models. The crosscheck experiments (blue symbols) were independently carried out for model validation.

As seen from Figure 3, vitamin C tends to strongly dissolve in water than in the alcohols. In water, the solubility of this compound were found not only significantly higher (comparing to the alcohols) but also more sensible with temperature change. In the studied temperature range, solubility in water follows a high order function versus temperature. Otherwise, these relationships in cases of alcohols were observed dissimilarly. To explain these phenomena, the structure of vitamin C is needed and presented in Figure 4. In the structure of vitamin C, there are many hydroxyl groups which can form hydrogen-bonds between the solute and solvents. On the one hand, in term of hydrogen-bond formation, alkyl groups in alcohols are less effective than case of water since they repulse electrons stronger than hydrogen does [17]. On the other hand, water molecule is small than ethanol and 2-propanol, thus, its equilibrium was achieved more easily. For those reasons, dissolution of vitamin C is accelerated in water more than in ethanol and 2-propanol.

Additionally, increasing alcohol size affects to dissolution processes as well. As presented in Figure 3, solubility of vitamin C in ethanol are higher than those in 2-propanol. The difference is relatively small but it indicates role of alkyl groups. This observation is also explained based on hydrogen-bonding formation ability of different alcohols. Further studies about the influence of alkyl groups are highly recommended in which other alcohols such as methanol, n-propanol, butanol, glycerol, etc. should be taken into account. It is believed that size and complexity of alcohols are important factors to vary solubility of vitamin C. This information could be exploited for a sustainable crystallization process design.

Figure 4. Molecular structure of vitamin C.
Furthermore, comparing the measured data in this work and the reported values from the literature [13], good agreements were obtained for all studied solvents including water, ethanol and 2-propanol. The highest error was found at 4.6% which is acceptable due to advantages of the applied polythermal method. Indeed, the described process in the literature [13] took about 3 days for each of measurements (applied isothermal procedure) while time cost in this work was significantly reduced to less than 2 hours for one single measurement. This achievement helps to shorten the early state during the development of drug formulation process (i.e. determination of solubility in various solvents – these completed tasks usually take years [18]).

From the solubility data, the van’t Hoff calculation (Figure 5) was applied to estimated enthalpy of dissolution which will be later compared to the values obtained from the modified Apelblat model in the next section. The results were shown in Figure 3. From Equation (10), the dissolution enthalpies were calculated and summarized in Table 2.

In another scenario, three A, B and C terms in the modified Apelblat model were also estimated using the above solubility data. The function lsqnonlin in Matlab minimizes the sum of difference between experimental and model solubility data. The best fitting of A, B and C values were listed in Table 2. From these terms, enthalpy and entropy of dissolution were calculated using Equations (7-9).

![Figure 5. van’t Hoff calculation](image)

Table 2. Parameter estimation and thermodynamic properties of dissolution.

| Solvent     | Parameter | Apelblat model | van’t Hoff |
|-------------|-----------|----------------|------------|
|             | A         | B              | C         | δH\text{diss} (kJ.mol\(^{-1}\)) | δS\text{diss} (J.mol\(^{-1}\).K\(^{-1}\)) | δH\text{diss} (kJ.mol\(^{-1}\)) |
| Water       | -16.78    | -6172.72       | 5.87      | 51.32                                   | 280.41                                   | 52.82                                   |
| Ethanol     | -22.12    | -6640.61       | 6.68      | 55.21                                   | 319.52                                   | 55.85                                   |
| 2-Propanol  | -27.86    | -7258.84       | 7.95      | 60.35                                   | 380.21                                   | 62.49                                   |

Obviously, the enthalpy of dissolutions obtained from the modified Apelblat and van’t Hoff models are in a relatively good agreement. The deviation of these enthalpies is less than 3.5% which is reasonable due to different approaches. However, more valuable than the van’t Hoff method, results from the modified Apelblat model also allow further estimating other thermodynamic properties, especially the entropy of dissolution. As seen in Table 2, enthalpies of dissolution processes of vitamin C in the investigated solvents are endothermic. Among of them, water is the most favorite solvent due to its minimum energy requirement for solution formation comparing to those of ethanol and 2-propanol. This could be explained due to the fact that vitamin C (as a polar solute) tends to well dissolve in strong polar solvents. For the studied solvents, polarity decreases as water > ethanol > 2-propanol. Another reason relates to hydrogen bonding formation which also more significant in case of water than ethanol and 2-propanol. Furthermore, variation of the dissolution entropy in these solvents followed the order H\text{2}O < ethanol < 2-propanol which proved that as more complicated solvent molecules as higher.
entropies of dissolution are observed. Herein, the size of R- (R-OH, with R- are H-, CH3-, CH(CH3)2-) has influence on solubility of vitamin C. In short, both enthalpy and entropy values obtained from this calculation supported the experimental observation for dissolution processes of vitamin C in the investigated solvents.

Finally, as presented in Figure 3, the calculated solubility from the obtained Apelblat model matched well with experimental data at relatively low temperatures. For the temperature lower than 317.15K, the obtained Apelblat model can be used for solubility correlation. However, the deviation was clearly observed at elevated temperatures that indicated the insufficient mathematical model due to complexity of the system at high temperature. Indeed, such as at 328.15 K, the worst deviation between the model and experiments reached to a value of 22.4% in case of water as solvent which shows the limitation of the modified Apelblat model applicability. This observation is also confirmed comparing to the validated independent experiments carried out at 303.15 and 333.15K as presented in Figure 3. To handle this problem, advanced mathematical models such as NRTL or UNIQUAC/UNIFAC are needed that could describe better interaction between solute and solvent molecules in order to obtain a more precise description of SLE at high temperature regions.

4. Conclusions

This paper presented a practical approach for solid-liquid equilibrium (SLE) determination based on the polythermal method in combination with a simple mathematical description. Experiments proved that the polythermal measurements allow detecting SLE quicker than the isothermal method does. Moreover, the polythermal method still remains high precision comparing to the isothermal method. For systems possessing high dissolution rates such as vitamin C, the proposed procedure is highly suitable for SLE determination not only for single solvents as studied in this paper but also for complex mixed solvents (e.g. binary, ternary and quaternary mixtures, etc.) for future works. The system works robustly with the measurement error lower than 2.3 percentages. Obviously, the reduced complexity of SLE determination via the applied method will help to save time cost for drug development processes which are frequently known as decays. Furthermore, the achieved Apelblat model allows quantifying thermodynamic properties of solution formation of vitamin C in different solvents. These dissolution were seen as endothermic processes and interactions of the solute and solvents are distinguished between different solvents. The solubility of this vitamin is higher in water than those in alcohols. This is explainable due to stronger hydrogen-bond formation between hydroxyl groups of vitamin C and water in comparison to those in cases of alcohols. Furthermore, as first information, the solubility also decreases when the sizes of alcohols are getting larger but more details need to be studied. The obtained thermodynamic data strongly supported the experimental observation. As seen in this paper, the Apelblat model is relatively simple but it allows predicting well data at temperatures lower than 315K but it is hard to predict SLE at higher temperature. For future work, advanced models such as NRTL, UNIQUAC/UNIFAC will be implemented to enhance the calculated results from the mathematical models.

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