Solubility of Organic Compounds in Subcritical Water

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Contributor: Erdal Yabalak , Sema Akay , Berkant Kayan , A. Murat Gizir , Yu Yang

Data on the solubility and decomposition of organic compounds in subcritical water, a green solvent, are needed in environmental remediation, chemistry, chemical engineering, medicine, polymer, food, agriculture, and many other fields. The solubility of organics is significantly enhanced with increasing water temperature. Likewise, the percentage of organic decomposition also increases with higher temperature.

subcritical water (SBCW) solubility decomposition

1. Introduction

The use of subcritical water as a green solvent for extraction or reaction media has gained importance with advanced scientific studies in the last 20 years. Subcritical water has variable physical properties compared to water at ambient conditions such as the dielectric constant, which is typically used for measuring polarity and can easily be tuned by changing temperature and pressure. As the temperature rises above 373, 473 and 505 K, the dielectric constant of water reaches the normal values of dimethyl sulfoxide (DMSO) (46.68), acetonitrile (37.5), and methanol (32.7), respectively [1][2][3]. Therefore, recent studies have demonstrated that subcritical water is successfully used as the sole medium in both extraction and chromatography, thus completely removing organic solvents in these processes.

2. Solubility in Subcritical Water

A simple and reliable system for the determination of solubility and partitioning behavior of fuel components in subcritical water up to 523 K was developed by Yang et al. [4]. The solubility of toluene increased ~23 times by increasing the temperature from ambient temperature to 473 °C, but the pressure change (from 1 to 50 bar) did not affect the solubility values in the solubility studies performed at ambient temperature. The increases in the separation of benzene, toluene, ethylbenzene, xylenes, and naphthalene from gasoline to liquid water when the temperature is increased from ambient temperature to 473 °C range from 10 times for benzene to 60 times for naphthalene. Similarly, increases in the partitioning of polycyclic aromatic hydrocarbons from diesel fuel to liquid water when the temperature was increased from ambient temperature to 523 °C ranged from 130-fold for naphthalene to 470-fold for methylnaphthalene.

After this study in 1998, Miller et al. [5] studied that the solubility of anthracene, pyrene, chrysene, perylene, and carbazole were determined at temperatures ranging from 298 to 498 K and at pressures from 30 to 60 bar in subcritical water. They estimated the solubility equation based on simplifying assumptions and empirical correlations based on data presented in this work and previous reports. The calculation of solubility at desired temperature needs only knowledge of ambient temperature solubility. Equation (1) is given below:

$$\ln x_2(T) = \left(\frac{T_0}{T}\right) \ln[x_2(T_0)] + 15(\frac{T}{T_0} - 1)^3 \quad (1)$$

where $x_2(T_0)$ refers to the solubility of organic compounds at ambient temperature, and $x_2(T)$ refers to the solubility of organic compounds at a calculated temperature.

The solubilities of benzene, toluene, m-xylene, p-cymene, octane, 2,2,4-trimethylpentane (isooctane), tetrachloroethylene, 1,2-dichlorobenzene, and tetraethyltin were investigated at temperatures ranging from 298 to 473 K. Increasing the temperature by 175 K increased the solubilities by a factor of 10–250 [6].

2.1. Solubilities of Polycyclic Aromatic Hydrocarbons and Derivatives in Subcritical Water

The solubility of PAHs is important for many industrial plants. Furthermore, their aqueous solubility determines both their uptake by the roots of plants and their transfer to other parts of the plant and their mobility in the soil. The solubilities of three PAHs, namely acenaphthene, anthracene, and pyrene, in water were measured in temperature and pressure ranges of 323–573 K and 50–100 bar, respectively, by Andersson et al. [7]. The solubility values of the employed compounds below their melting point were determined to be consistent with literature values, and the solubility of pyrene and anthracene exponentially varies with temperature. The solubilities of acenaphthene, anthracene, and pyrene were calculated as mole fraction solubilities (x_2) and were determined as 1.25×10^{-3} at 300 K and 100 bar, within 1.02×10^{-7}–3.78×10^{-3} at a temperature range of 373–573 K and pressure of 50 bar and 6.87×10^{-8}–1.41×10^{-3} at a temperature range of 323–573 and pressure range of 50–100 bar, respectively.

Karásek et al. developed a semiempirical relationship to correlate the solubility of PAHs (naphthalene, anthracene, pyrene, chrysene, 1,2-benzanthracene, triphenylene, perylene, *p*-terphenyl) in pressurized hot water within the temperature range of 313–498 K, a pressure of 1–77 bars and equilibrium mole fraction (x_2) of 10^{-11}–10^{-3}. They used only pure-component properties such as cohesive energy density, internal pressure and dielectric constant of water and enthalpy of fusion, triple-point temperature, the molar volume of the solid compound and the molar volume of the subcooled liquid of PAHs [8]. The x_2 data were experimental values of the previously reported research studies. y_2 (Raoult's law activity coefficient of the solute) values of each PAH mentioned above were calculated using Equation (3), where fs02(the fugacity of the pure solid solute) and f102(the pure subcooled liquid solute) values were calculated by Equations (2) and (3).

$$x_2 = \frac{f_2^{s0}}{\gamma_2 f_2^{10}} \tag{2}$$

$$x_2 = \frac{f_2^{s0}}{\gamma_2 f_2^{10}} \tag{3}$$

The aqueous solubilities (x_2) of solid heterocyclic analogues of anthracene, phenanthrene and fluorene at a specific temperature range (313 K–the melting point of each compound) under 50 bar of pressure were reported by Karásek et al. [9]. They collected the solubility data of each compound via the dynamic saturation method based on pressurized hot water extraction. x_2 values for the employed compounds were found to be within the 3.17×10^{-9}–8.27×10^{-4} range and were widely changeable based on the applied temperature. It was also indicated that no appreciable degradation was observed for any compound in the temperature range studied based on the GC/MS results. Obtained solubilities were converted to activity coefficients of individual solutes in saturated aqueous solutions, and the relationship between temperature and type or number of heteroatoms was evaluated (Equation (4)).

$$\ln x_2 = b_1 + b_2\left(\frac{T_0}{T}\right) + b_3 \ln(\frac{T}{T_0}) \tag{4}$$

where T_0 and y_2 refer to 298.15 K and Raoult's law activity coefficient of the related compound, respectively. b_1, b_2, and b_3 denote the least-squares estimates of the coefficients, and T is the absolute temperature at the experimental conditions. Hence, the increase in the aqueous solubilities of solid heterocyclic analogues of anthracene, phenanthrene and fluorine was reported to strongly depend on the increasing temperature and variance with the heteroatoms.

The solubility values of PAHs in subcritical water were calculated using Equations (5) and (6) with the above-mentioned three UNIFAC-based thermodynamic models.

$$\ln x_2^{\mathrm{id}} = \ln\left(\frac{f_2^{\mathrm{s}}}{f_2^{\mathrm{o}}}\right) = -\frac{\Delta H_{\mathrm{m2}}}{RT_{\mathrm{m2}}}\left(\frac{T_{\mathrm{m2}}}{T} - 1\right) \tag{5}$$

$$\ln x_2 = \ln x_2^{\mathrm{id}} - \ln \gamma_2 \tag{6}$$

where f_2^{s} , f_2^{o} , γ_2 , x_2 , T_{m2} and ΔH_{m2} indicate the fugacity of the pure solid solute, the fugacity of the pure solute in liquid, the activity coefficient of the solute in the liquid state, molar fraction (solubility) of the related solid, the normal melting temperature of the solid and the enthalpy of the fusion, respectively.

The prediction of the solubility of 25 PAHs was investigated using the cubic-plus-association equation of state (CPA EoS, Equation (7)) by Oliveira et al. [10]. They collected the experimental data for all studied compounds in the specific temperature (313.15–498.15 K) and pressure range (40–65 bar) from the previously studied literature. In their study, vapor pressures and liquid densities were estimated with deviation values below 1.1% and 1.4%, respectively, using the CPA model. They also stated that the solubility values can be correlated within a 6% deviation when considering the dissolution between a self-assembled molecule and non-self-assembled molecule. Although the applied model provides a 20% global average deviation for solubilities of PAHs in pressurized hot water, the prediction capability of CPA EoS is quite good when used for PAHs without liquid density data.

$$x_s = \frac{\varphi_s^{L0}}{\varphi_s^{L}} \exp\left[-\frac{\Delta_{\mathrm{fus}} H}{R}\left(\frac{1}{T} - \frac{1}{T_m}\right)\right] \tag{7}$$

where $\Delta_{\mathrm{fus}}H$ indicates solute enthalpy of fusion, T indicates absolute temperature, T_m indicates melting temperature of a solute, R refers to gas constant, φ indicates the fugacity coefficient (“0” indicates a pure component), and x_s indicates mole fraction aqueous solubility.

The Raoult’s law activity coefficient of the solute in the saturated solution (γ_2^{sat}) can also be obtained from Equation (9), where f_2^{s0} and f_2^{l0} indicate the fugacity of the pure solid solute and the pure subcooled liquid solute, respectively.

$$\ln x_2 = a_1 + a_2\left(\frac{T_0}{T}\right) + a_3 \ln(\frac{T}{T_0}) \tag{8}$$

$$\ln x_2 = a_1 + a_2\left(\frac{T_0}{T}\right) + a_3 \ln(\frac{T}{T_0}) \tag{9}$$

Teoh et al. [11] investigated the solubility of anthracene and *p*-terphenyl in subcritical ethanol and water at two different pressures (50–150 bar) and between 393 and 473 K by using a static analytical equilibrium method. They indicated that the ethanol composition and temperature are effective on the solubility of polyaromatic hydrocarbons in the subcritical solvent mixture. The solubilities of PAHs in subcritical ethanol increased exponentially with temperature. A new empirical approach model has been proposed to correlate the ethanol mole fraction and temperature with the solubility of anthracene and *p*-terphenyl (Equation (10)).

$$\ln\ x_{\text{solute}}\ = pT + qf + r \qquad (10)$$

2.2. Alkyl and Chlorobenzene Solubilities in Subcritical Water

The solubility of chlorobenzenes, which are used as intermediates in industrial products such as dyestuffs, in water, as well as their distribution rates in water and other organic solvents, is necessary to determine their distribution rates in aquatic environments or whether they tend to accumulate. The solubility of ethylbenzene, m-xylene, and benzene in water was determined using a laboratory-made system at temperatures ranging from 298 to 473 K and a pressure of 50 bars by Mathis et al. [12]. The solubility of alkylbenzenes increased by at least one order of magnitude by increasing the temperature from 298 to 473 K. A simple and reliable approximation model was developed, Equation (11), to predict the solubility of liquid organics in subcritical water.

$$\ln x_2(T) = \left(\frac{T_0}{T}\right)\ln x_2(T_0) + 2\left(\frac{T - T_0}{T_0} - 1\right)^3 \qquad (11)$$

2.3. Organic Acid Solubilities in Subcritical Water

Organic acids are used in many biomedical applications as well as in various industrial productions. In addition to many physicochemical properties, their solubility value is also an important parameter in these processes. Kayan et al. [13] investigated the solubility of benzoic and salicylic acids at constant pressure and different temperatures. The mole fraction solubility of benzoic acid varied from 2.22×10^{-3} at 298 K to 1.36×10^{-2} at 473 K and that of salicylic acid varied from 4.69×10^{-5} at 298 K to 1.02×10^{-1} at 473 K. The solubility of both compounds increased two-fold with increasing the temperature from 298 to 473 K. However, it was determined that benzoic acid is as stable as 473 K, but salicylic acid is partially degraded. They proposed a new approximation model to estimate the solubility of both compounds as shown in Equation (12):

$$\ln x_2(T) = \left(1.85\frac{T_0}{T} - 1\right)\ln x_2(T_0) \qquad (12)$$

where the mole fraction solubility at any temperature T is $x_2(T)$, and the ambient mole fraction solubility is given by $x_2(T_0)$. This model provides a better approximation of the solubility of both acids than previous models.

The aqueous solubility of gallic acid hydrate was found to vary between 1.24 × 10^{-3} at 298.75 K and 2.33 × 10^{-1} at 415.85 K. The mole fraction solubility of protocatechuic acid at the same temperatures varied between 3.55 × 10^{-3} and 1.26 × 10^{-1}, respectively. The solubility of the selected phenolic compounds was measured as a function of temperature using a dynamic flow apparatus. It was found that the solubility of these compounds increased considerably with temperature. The obtained data are in agreement with the literature. Solubility data were fitted with other semi-empirical equations as well as with empirical equations such as the modified Apelblat equation to predict the water solubility of phenolic compounds for which a solubility value is known at room temperature conditions. Thermodynamic data were obtained from solubility data as a function of temperature. The obtained thermodynamic data showed that the dissolution process of phenolic compounds in water is endergonic, exothermic and entropy-driven. The solubility of phenolic compounds was approximated using the following Equation (13). The approximation model was derived as a function of temperature.

$$\ln x_2 = \left(\frac{T_0}{T}\right) \ln x_2\left(T_0\right) + 11\left(1 - \frac{T_0}{T}\right) \tag{13}$$

where $x_s(T)$ and $x_s(T_0)$ are the mole fraction solubilities of the phenolic compounds at temperature T and reference temperature T_0, respectively.

Terephthalic acid (TPA) has industrial importance because of its use as a raw material in polyesters. Takebayashi et al. [14] investigated the solubility of TPA in subcritical water at a constant pressure of 10 MPa and a temperature range of 349–547 K. The solubility of TPA was found to vary between 1.25 × 10^{-5} at 349 K and 2.99 × 10^{-2} at 547 K. This shows that there is an exponential increase with temperature. They expressed the temperature dependence of $\ln x_2$ as Equation (14):

$$\ln x_2 = a + b\left(\frac{T}{K}\right) \tag{14}$$

2.4. Pharmaceutical Compounds Solubilities in Subcritical Water

Most studies have been conducted on the pharmaceutical solubility of compounds in subcritical water. Srinivas et al. [15] applied the Hansen three-dimensional solubility parameter concept, a group contribution method, to test its prediction performance. They used the data from literature on the extraction of betulin (an antiviral agent), niacin (vitamin B3) and flavonoids (malvidin-3,O-glucoside, malvidin-3,5-diglucoside, malvidin-3,O-(6,O-p,acetyl) glucoside, malvidin-3,O-(6,O-p,coumaroyl)glucoside and catechin) from natural sources using subcritical fluid solvents such as water and ethanol [16]. They characterized and quantifies solute–subcritical solvent interactions and miscibility as a function of temperature by Hansen solubility spheres based on relative energy differences (RED). Equation (6) was used to predict and optimize the temperature and solvent conditions for extraction of the organic compounds from natural sources based on the RED values and R_a value; the term in Equation (15) is calculated by Equation (16).

$$RED = \frac{R_a}{R_o} \tag{15}$$

$$R_a^2 = 4(\delta_{D_1} - \delta_{D_2})^2 + (\delta_{P_1} - \delta_{P_2})^2 + (\delta_{H_1} - \delta_{H_2})^2 \tag{16}$$

where R_o , R_a , δ_D , δ_P and δ_H indicate the radius of the Hansen sphere, the distance between the solute or solvent and the mass center of the Hansen sphere, dispersion solubility parameter, polar solubility parameter and hydrogen bonding solubility parameter, respectively, each with MPa$^{1/2}$ unit.

Emire et al. [17] investigated the subcritical water solubility of paracetamol, known as an anti-inflammatory drug, at constant pressure in the temperature range of 293–403 K. The mole fraction solubility of paracetamol was found to vary depending on temperature. They proposed a new approach model, shown in Equation (17), to determine the subcritical water solubility of paracetamol.

$$\ln x_2 = \left(\frac{T_0}{T}\right) \ln x_2\,(T_0) + \frac{8}{5}\left(\frac{T - T_0}{T_0}\right) \tag{17}$$

Escitalopram (ESC) has very low water solubility and is used in the treatment of depressive disorders. Akay et al. [18] investigated the mole fraction solubility of ESC at constant pressure and a temperature range of 298–473 K. Based on experimental data, a new mathematical model was developed as shown in Equation (18):

$$\ln x_2 = \left(\frac{T_0}{T}\right) \ln x_2\,(T_0) + \left(\frac{\varepsilon_{(T_0)}}{\varepsilon_{(T)}}\right)^{1/2} \tag{18}$$

2.5. Carotenoids and Flavonoids Solubility in Subcritical Water

A pioneering study of degradation and solubility of fragrance and flavonoids was conducted by Yang et al., which is an investigation of the degradation of *α*-pinene, limonene, camphor, citronellol, and carvacrol, terpene member compounds, under subcritical water conditions (423 K–523 K) and their extraction from basil and oregano leaves [19] . They reported that the stability of terpenes is highly dependent on temperature and begins to decompose as the temperature increases. In total, 25–31% of *α*-pinene and limonene were found to be degraded after 30 min of heating at 373–423 K; moreover, increasing the temperature to 523 K resulted in a 64% degradation rate of these two compounds. Nevertheless, camphor, citronellol, and carvacrol showed better stability at these temperatures, as a below 10% of degradation rate was obtained at ≤273 K and 20–42% of degradation rates were obtained at 473–523 K for these compounds.

The mole fraction solubility of anhydrous quercetin changed from 2.05×10^{-7} at 298 K to 7.12×10^{-5} at 413 K and that of quercetin dihydrate changed from 1.38×10^{-7} at 298 K to 8.58×10^{-5} at 413 K. The solubility of both substances increased significantly with increasing temperature. In addition, they indicated that the aqueous solubility of both molecules showed similar solubility behavior up to 353 K, but at temperatures equal to or higher than 373 K, the quercetin dehydrate form dissolved 1.5–2 times more than the anhydrous form. The solubility of the anhydrate and dihydrate forms of quercetin at different temperatures was correlated with the Apelblat equation. The importance of optimizing the solvent flow rate at a given temperature to effectively dissolve a solute such as quercetin in water was emphasized.

2.6. Carbohydrates Solubilities in Subcritical Water

Due to the physicochemical properties of carbohydrates, they play many roles in the design and optimization of chemical engineering processes. The solubility of three different carbohydrates such as glucose, maltose and xylose was investigated by Zhang et al. [20] in the temperature range from 293 to 453 K. The aqueous solubility of glucose was found to vary between 4.45×10^{-2} at 298 K and 2.32×10^{-1} at 453 K. The mole fraction solubility of maltose at the same temperatures varied between 2.44×10^{-2} and 1.47×10^{-1}, and xylose varied between 5.08×10^{-2} and 2.07×10^{-1}. While the solubility of all three substances increased normally up to the boiling point of water, there was a five-fold increase in solubility for all molecules above the boiling point of water. Although the solubility values differ partially from the study of Yalkowsky and He [21], they are generally compatible with the literature. The solubility of the sugar was measured using the continuous flow technique, in which the sugar was saturated at various temperatures in a stream of flowing hot water. The resultant sugar solubility trends were modeled empirically or by use of a modified Apelblat equation or the modified UNIQUAC functional group activity coefficient (A-UNIFAC) model. The thermodynamic properties of the solution for the sugars and the free energies of the solution were found for all molecules to be positive and of similar magnitude.

2.7. Preservative Ingredient Solubilities in Subcritical Water

The solubility of parabens such as methyl, ethyl and butylparaben was measured using a homemade system in the temperature range of 273–473 K [22]. The mole fraction solubilities of methylparaben, ethyl and butyl were found to be 2.50×10^{-4}, 0.74×10^{-4} and 0.18×10^{-4} at 298 K and 1.50×10^{-3}, 0.91×10^{-3} and 0.41×10^{-3} at 473 K, respectively. The solubility of all parabens increases with increasing temperature, and the solubility increased between 6 and 19-fold by increasing the temperature from 298 to 373 K. However, severe degradation of all three parabens studied occurred at 473 K. They determined that because of the degradation of parabens at high temperature, the experimental solubility of parabens decreased.

2.8. Fatty Acids Solubilities in Subcritical Water

In a study on the solubility of saturated fatty acids (caprylic acid, capric acid, lauric acid, myristic acid, stearic acid and palmitic acid) with carbon numbers from 8 to 18, the effects of the temperature at 333–503 K and the pressure in the 5–15 MPa range on the solubility were investigated [23]. It was reported that the solubility of fatty acids was increased by increasing the temperature, while it did not change with the pressure. It was determined that the logarithm of the mole fraction of solubility at temperatures higher than 433 K was linearly related to the inverse of the absolute temperature for each fatty acid, and this phenomenon was associated with the formation of a regular solution of the water containing solubilized fatty acid molecules at higher temperatures.

2.9. Pesticide Solubilities in Subcritical Water

The solubility of three triazine pesticides, which are known as food contaminants, namely atrazine, cyanazine, and simazine, were measured in pure and modified subcritical water at a temperature range of 323–373 °C and a pressure of 50.6 bar using Equation (26) [24]. It was indicated that the temperature and the amount and type of the modifier (cosolvent) are major system variables that influence the solvent strength, as the dielectric constant of water is highly dependent on these variables. Therefore, ethanol and urea were used in a variable amount and variable temperature range to modify the water. Herein, it was determined that an increase in each 25 K in temperature provides a three-fold increase in the solubility of triazine.

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