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Fluid Phase Equilibria 340 (2013) 7–10

3Contents lists available at SciVerse ScienceDirect Fluid Phase Equilibria journal homepage: www.elsevier.com/locate/fluid Measurement and modeling of epicatechin solubility in supercritical carbon dioxide

fluid Felycia Edi-Soetaredjoa, Suryadi Ismadjib, Yi-Hsu Jua,∗ a

5Department of Chemical Engineering, National Taiwan University of Science and Technology, 43, Sec. 4. Keelung Rd., Taipei, Taiwan b Department of Chemical Engineering, Widya Mandala Surabaya Catholic University, Kalijudan 37, Surabaya 60114, Indonesia

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Density based model Epicatechin Solubility Supercritical carbon dioxide abstract The experimental equilibrium solubility of epicatechin in supercritical carbon dioxide was measured at several

9temperatures (313.15, 323.15, 333.15 and 343.15 K) and pressure in the range of

12–26 MPa. The solubilities of epicatechin in supercritical CO2 increased with pressure and temperature. The effect of increasing temperature on the solubility is more significant at high pressure than low pressure. The experimental data were fitted very well by

2three density-based models of Chrastil, del Valle and Aguilera and the model of Méndez-Santiago and Teja.

Reasonable fitting parameters of the models were obtained. ©

1 1. Introduction

12Epicatechin ((−)-cis-3,3′,4′,5,7-pentahydroxyflavane (2R,3R)-2- (3,4-dihydroxyphenyl)-3,4-dihydro-1(2H)-benzopyran-3,5,7-

tri- ol) is a flavonol belonging to the group of flavonoids and widely present in fruits and vegetables. High quantities of epicatechin can be found in cocoa [1], tea [2–4] and grapes [5–7]. Epicatechin is also found in staple plant such as sago (Metroxylon sago) [8]. Epicatechin exerts beneficial health-related effects since it acts as free radical scavengers and inhibitors of eicosainoid biosyn- thesis [9,10]. Epicatechin also reduces low-density lipoprotein in oxidation, risk of stroke, heart failure, cancer and diabetes [1,9,11,12]. The initial step in the recovery and purification of flavonoids from plant materials is extraction. Soxhlet extractions using organic–aqueous solvents are commonly used; however this method is time consuming and uses a lot of organic solvents. Recently, microwave-assisted extraction [13], accelerated solvent extraction [14,15] and supercritical fluid extraction were employed to obtain better recoveries and to reduce extraction time [16,17]. Among these extraction methods, supercritical fluid extraction (SFE) offers an environmental friendly choice in terms of using less amount of organic solvent. SFE works in the absence of light and air that cause degradation of flavonoids. Among many super- critical fluids, carbon dioxide is extremely attractive because of it ∗

7Corresponding author. Tel.: +886 2 2737 6611; fax: +886 2 2737 6644. E-mail address: yhju@mail .ntust.edu.tw (Y.-H. Ju). 0378-3812/$ – see front matter © 2012 Elsevier B.V. All rights reserved. http://dx .doi.

org/10.1016/j.fluid.2012.12.005 has relatively low critical temperature (304.15 K), is less possible to cause degradation of thermally labile compounds and is easily separable from the extracted solutes. Other benefits in using super- critical carbon dioxide (SC CO2) are it is nontoxic, nonflammables and inexpensive. Process design based on SFE and the determination of opti- mum operating conditions require knowledge of phase equilibria and compound solubility in a supercritical fluid. In the last two decades, many attempts have been reported on the solubility of flavonoid compounds. Song et al. [18] determined the solubil- ity of epigallocatechin gallate in SC CO2 with ethanol cosolvent

6(0.044 and 0.084 mole fraction) at a temperature ranging from 313 to 333 K and pressure from 15 to 35 MPa. Their solubility

data were correlated using a

6thermodynamic model, a modified Chrastil model and the Méndez-Santiago and Teja [19] model.

Cháfer et al. [20] reported experimental measurements and thermodynamic modeling of the solubility of quercetin with ethanol as the cosol- vent (5–30%) at 313.15 K and pressure ranging from 8 to 12 MPa. The same operating conditions were used by Berna et al. [21] for catechin and Cháfer et al. [22] for epicatechin and the data were modeled using equations of state (EOS): Peng–Robinson and Soave–Redlich–Kwong models. Other solubility data of hydroxy- cinnamic acids in SC CO2 were reported for

144-hydroxycinnamic acid (p-coumaric acid), 3,4-dihydroxycinnamic acid (caffeic acid), 4 -hydroxy-3-methoxycinnamic acid (ferulic acid)

[23–25]. The existing solubility data for epicatechin in SC CO2 + ethanol [22] was limited at one fixed temperature (313

17K) and a narrow pressure range of 8– 12 MPa.

Wider ranges of temperature and pressure are essential in order to determine optimum operating 8 F. Edi-Soetaredjo et al. / Fluid Phase Equilibria 340 (2013) 7–10 conditions for separation of epicatechin from plant materials using SC CO2. Epicatechin is almost insoluble in SC CO2 due to its strong polarity; therefore ethanol is the best choice as cosolvent especially for food and pharmaceutical applications. In their report, Cháfer et al. [22] used ethanol as cosolvent in the range of 5–30%. The pres- ence of high amount of ethanol in their system may have changed the supercritical condition of CO2 into subcritical [26–29]. Joung et al. [29] found that the maximum concentration of ethanol in the system should be 6.49 mol% at 8.15 MPa and 313.15 K in order to obtain supercritical condition. Under subcritical condition, most epicatechin dissolved in ethanol rather than in SC CO2. Further sep- aration of epicatechin from ethanol is needed in order to obtain pure epicatechin. Considering the importance of solubility data of solids in super- critical fluids for the developing of the extraction process and the dependency of solubility on density, temperature and pressure of supercritical fluids; extensive amounts of data are needed. More- over, the present of cosolvent in SC CO2 extraction produces impure extract with the cosolvent as an impurity. Therefore, in order to obtain pure extract, separation of extract and cosolvent is needed. The objective of this paper is to measure the solubilities of epi- catechin in SC CO2 without cosolvent at

9different temperatures (313.15, 323.15, 333.15, and 343.15 K)

and pressures (12–26 MPa). The experimental data were then correlated using

2**density-based** models **(Chrastil** [30], **del Valle and Aguilera** [31] **and Méndez-Santiago and Teja**

[19] models).

20**To the best of knowledge, there is no** literature available **on the solubility of** epicatechin **in SC CO2 in**

16**the range of pressures** and temperatures **studied in this work.**

112**. Experimental 2.1. Materials** HPLC grade (−)-epicatechin **with a purity of**

98 wt.%

3**was obtained from Sigma–Aldrich** (Singapore) **and was used without** any **further purification.**

Analytical grade ethanol was supplied by Merck (Darmstadt, Germany) and was used as a solvent to collect extract for further analysis. Food grade carbon dioxide (99% purity) was used as the supercritical solvent and supplied as liquid CO2 by Aneka Gas Pty Ltd (Indonesia) with a purity of 99.9%. 2.2. Experimental procedure The experiments of epicatechin solubility in supercritical CO2 were carried out in a static system consists of a 50 ml long equil- ibration column (Swagelok, USA), a high pressure pump (Eldex AA-100-S-2-CE, USA) and a pressure transducer (Druck PTX 611, USA) with a digital process indicator (Druck DPI 280, USA) which gives a pressure measurement uncertainty of ±0.01 MPa. The sys- tem temperature was controlled by a heating chamber (Oven Memmert, Germany). The uncertainty of temperature measure- ment in the heating chamber is ±1 K. All fitting and tubing used in this system were made of

22**stainless steel** 316 **(Swagelok,** USA). **The maximum working pressure and temperature of the** supercritical extraction **system** were 40 **MPa and** 373.15 K, **respectively.**

Epicatechin solubility data were obtained by adding 100 mg of (−)-epicatechin in a sample holder in the equilibration column. The system was heated to a desired temperature (313.15, 323.15, 333.15, or 343.15 K). Subsequently, liquid

11**CO2 was** pumped **into the system using the high-pressure pump until a** desired **pressure was reached**

(12–26 MPa). After equilibrium condition was achieved in 4 h (insignificant increase of epicatechin concentration in CO2 was observed after 3 h), the output valve was released, and the sample flowed into a collector containing a known amount of ethanol to separate epicatechin and CO2. At least three replications of trials were carried out for every set of temperature and pressure, and the equilibrium composition was represented by average of the three replications. The uncertainty of each measurement was within ±2%. The determination of epicatechin concentration in ethanol was based on the method of Zuo et al. [32] by using a HPLC (Jasco HPLC PU-2089 plus) with a UV–vis detector (UV-2077 plus).

163**. Results and discussion The experimental** equilibrium **solubility data of**

epicatechin and the result of calculated densities of supercritical SC CO2 at vari- ous pressures and temperatures using the Stryjek and Vera [33] modification of the Peng–Robinson equation of state are reported in Table 1. The solubility of epicatechin in SC CO2

4**increases with increasing pressure** and temperature. **At constant temperature,**

increasing pressure raises SC CO2 density and increases its ability to dissolve solute. At constant pressure, increasing temperature raises solute vapor pressure as well as the diffusivities of both solvent and solute. The correlation of experimental solubility data was investigated using several semi-empirical models. Chrastil [30] proposed the first model for density-based correlation. It

10**is based on the** hypoth- esis **that** one **molecule of a solute** A **associates with k molecules of** a **solvent** B **to form** one molecule of **solvato-complex** ABk **in** equi- librium **with the system. The**

definition of the equilibrium constant through thermodynamic consideration resulted in the following model for the solubility: c1= kexp T +b a (1) wherec1istheconcentrationofthesoluteinthegas(gl−1), isthe ( ) density of the gas (g l−1), k is an association number, a

15**is a function of the enthalpy of solvation and enthalpy of vaporization** (K−1), **and** b **is** a function of **association number** and molecular weights **of the**

solute and supercritical fluids. Various modifications on Chrastil model were proposed such as modified Chrastil by Garlapati and Madras [34,35], modified Chrastil by Wang [36], and modified Chrastil by del Valle and Aguil- era [31]. Among these modified Chrastil models, del Valle and Aguilera [31] claimed that their model fitted well for tempera- tures

8**from 293 to 353 K and** pressure **between** 15 **and** 88 MPa **and adequately predicted** solubility **under 100 g l−** 1 **within the region suggested for commercial supercritical fluid extraction of food components**

(Eq. (2)). c1 = k′ exp b′ + a ′ d T + r2 (2) The physical(meaning of t)he parameters k′, b′, and a′ are similar to Chrastil model. The parameter d′ is introduced to compensate the variation of enthalpy of vaporization (? Hvap) with temperature. Multivariable non-linear regression analysis of all experimental data was performed to estimate the constants in Chrastil model and modified Christil model by del Valle and Aguilera. The quality of all data correlations is quantified by the sum of squared errors (SSE), defined as follows: SSE = c 1(exp) − c1(cal) N 2 1/2 (3) ((∑)) where c1(exp) is the actual solubility of epicatechin in SC CO2, c1(cal) is the calculated solubility, and N is the number of experimen- tal data. Multivariable non-linear regression technique employed an iterative curve fitting procedure. An initial estimation for each parameter was provided, and then calculation of a point-by-point sum of squares (Eq. (3)) for each iteration was conducted until convergence criteria were fulfilled. Table 1 Solubility of epicatechin in SC CO2 and the SC CO2 density. P (MPa)

19**T = 313.15 K T = 323.15 K T = 333.15 K T = 343.15 K**

(g

21**l−3) 103** c1 **(g l−3)** 106 ya **(g l−**

3) 103 c1 (g l−3) 106 ya (g

21**l−3) 103** c1 **(g l−3)** 106 ya **(g l−**

3) 103 c1 (g l−3) 106 ya 12 669 13 701 14 727 15 750 16 769 17 787 18 803 19 818 20 831 21 844 22 856 23 867 24 877 25 887 26 896 2.90 ± 0.06 0.66 ± 0.014 3.56 ± 0.10 0.77 ± 0.022 3.97 ± 0.02 0.83 ± 0.004 4.40 ± 0.12 0.89 ± 0.024 5.15 ± 0.12 1.02 ± 0.024 5.65 ± 0.10 1.09 ± 0.019 6.02 ± 0.18 1.14 ± 0.034 6.53 ± 0.02 1.21 ± 0.004 6.97 ± 0.03 1.27 ± 0.006 7.49 ± 0.06 1.34 ± 0.011 8.01 ± 0.03 1.42 ± 0.005 8.32 ± 0.22 1.46 ± 0.038 8.87 ± 0.09 1.53 ± 0.016 9.20 ± 0.12 1.57 ± 0.021 9.68 ± 0.08 1.64 ± 0.014 537 3.28 ± 0.077 588 4.51 ± 0.136 627 6.48 ± 0.162 659 7.83 ± 0.196 685 9.19 ± 0.259 709 10.70 ± 0.195 729 11.50 ± 0.129 748 13.36 ± 0.338 765 14.10 ± 0.229 780 15.60 ± 0.409 794 17.08 ± 0.482 808 18.10 ± 0.275 820 19.50 ± 0.316 832 20.73 ± 0.273 843 21.82 ± 0.203 0.93 ± 0.022 1.16 ± 0.035 1.57 ± 0.039 1.80 ± 0.045 2.03 ± 0.057 2.290 ± 0.042 2.39 ± 0.027 2.72 ± 0.068 2.79 ± 0.045 3.03 ± 0.079 3.26 ± 0.092 3.40 ± 0.052 3.60 ± 0.058 3.77 ± 0.050 3.92 ± 0.036 417 2.95 ± 0.088 474 5.07 ± 0.136 523 7.64 ± 0.222 563 9.85 ± 0.207 598 14.25 ± 0.322 627 17.89 ± 0.292 653 20.10 ± 0.308 675 22.50 ± 0.215 696 25.70 ± 0.402 714 29.51 ± 0.507 732 32.01 ± 0.465 747 36.01 ± 0.343 762 38.05 ± 0.474 775 41.14 ± 0.328 788 44.92 ± 0.263 1.09 ± 0.032 1.65 ± 0.044 2.27 ± 0.064 2.88 ± 0.056 3.50 ± 0.082 4.09 ± 0.071 4.67 ± 0.071 5.19 ± 0.048 5.73 ± 0.088 6.22 ± 0.108 6.73 ± 0.096 7.18 ± 0.070 7.66 ± 0.094 8.10 ± 0.064 8.54 ± 0.051 342 390 436 478 515 549 578 604 628 649 669 687 704 719 734 3.25 ± 0.053 5.64 ± 0.152 9.07 ± 0.165 13.90 ± 0.160 19.91 ± 0.358 24.20 ± 0.544 30.01 ± 0.430 36.21 ± 0.927 44.78 ± 0.961 51.38 ± 0.904 57.96 ± 0.569 62.06 ± 0.673 70.15 ± 0.635 77.72 ± 0.694 85.86 ± 0.673 1.49 ± 0.024 2.27 ± 0.059 3.26 ± 0.057 4.38 ± 0.051 5.56 ± 0.011 6.85 ± 0.015 8.10 ± 0.011 9.34 ± 0.023 10.57 ± 0.023 11.77 ± 0.021 12.98 ± 0.13 14.14 ± 0.015 15.31 ± 0.014 16.38 ± 0.015 17.53 ± 0.014 a y is mole fraction. The standard uncertainty is estimated using standard deviation of the mean: 1 n 1/2 u(xi) = n(n − 1) (Xi,k − $\bar{X}$l)2 k=1 . (∑) data (?). Fig. 2. del Valle and Aguilera model fittings (wire mesh) and experimental solubility not the same as that in Eq. (2). The effect of enthalpy of solvation and Aguilera as shown in Eq. (2), the parameter b in Eq. (1) is an extra parameter d in the modified Chrastil model by del Valle Therefore, the value should be similar. However, since there is number and molecular weight of solute and supercritical fluid. model and in del Valle and Aguilera is a function of association one molecule of a solvato complex AB4. The parameter b in Chrastil echin (A) is associated with four molecules of gas CO2 (B) to form both models are similar. This indicates that one molecule of epicat- The parameter k is an association number, and its values from 2.279, −11,596 and 134,374, respectively, with a SSE of 0.06%. the experimental data and the values of k′, b′, a′, and d are 4.2313, is shown in Fig. 2. This modified Chrastil model also fits very well data of epicatechin in SC CO2 using del Valle and Aguilera model 1.067, respectively, with a SSE of 0.04%. The fitting of solubility from multivariable non-linear regression are 4.2311, −10,788, and solubility data. The parameters of Chrastil model, k, a, and b resulted using Chrastil model. Chrastil model fits well to the experimental Fig. 1 shows the fitting of solubility data of epicatechin in SC CO2 Fig. 1. Chrastil model fittings (wire mesh) and experimental solubility data (?). F. Edi- Soetaredjo et al. / Fluid Phase Equilibria 340 (2013) 7–10 9 10 F. Edi-Soetaredjo et al. / Fluid Phase Equilibria 340 (2013) 7–10 Fig. 3. Méndez-Santiago and Teja model fittings (wire mesh) and experimental solubility data (?). and enthalpy of vaporization in Chrastil model is presented in the parameter a (a = ?H/R). However, del Valle and Aguilera [31] modified this parameter to compensate the variation of enthalpy of vaporization with temperature (?H(T) = R(a′ + 2d/T)). The heat of solution (?H) for epicatechin and CO2 using Chrastil model is 89.7 kJ/mol, while using del Valle and Aguilera the heat of solution falls in a range of 87.3–89.9 kJ/mol. From the analyses of all parameters for both models, we conclude that

4**the solubility of** epicatechin **in SC CO2**

can be well fitted by both models. Another semi-empirical density based model was

4**proposed by Méndez-Santiago and Teja** [19] **based on the theory of dilute** solu- tions. **This model**

took into account Henry's law, infinite dilution partition coefficients near

4**the critical point of the solvent**

and

17**Clausius–Clapeyron-type expression for** the **sublimation pressure. The equation**

has three adjustable parameters (A′, B′ and C′), defined as follow: T ln(c1P) = A′ + B′ + C′T (4) Fig. 3 shows that the Méndez-Santiago and Teja model can repre- sent the experimental solubility data of epicatechin in SC CO2 well. Values of A′, B′ and C′ are −15,230, 3.008 and 4.108, respectively, and the SSE is 1.18%. The three constants (A′, B′ and C′) are the result of multivariable non-linear regression analysis, which is indepen- dent of temperature and pressure without any significant physical meaning, therefore, can be used to extrapolate solubility data to other temperature [19]. 4. Conclusion Solubility data of epicatechin in SC CO2, in wider ranges of temperature and pressure, were obtained in this study. The exper- imental data was fitted by using

2**density-based** models **(Chrastil, del Valle and Aguilera and Méndez-Santiago and Teja).**

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