녹차로부터 초임계 CO_2을 이용한 카페인 추출

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Caffeine extraction from green tea using Supercritical CO_2

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Introduction

Tea is among the most popular beverages all over the world. In numerous epidemiological and pharmacological studies, It says that green tea extracts possess strong antioxidant effects [1] and antimutagenic activity. There are caffeine(1-5%), catechin(5-27%) and other composition in the extracts. Caffeine typically is used as a stimulant which could affect central nervous system and mental alertness. Prevents sleepiness and speeds recovery from fatigue. The conventional production methods of caffeine such as solvent extraction and soxhlet, although effective for extraction, can lead to degradation of heat sensitive compounds as well as leave traces of toxic solvents in the solute. Supercritical fluid technology may be a viable alternative to solvent extraction methods. CO_2 is generally the most desirable solvent for supercritical fluid extraction (SFE). It is an inert, non-flammable, non-explosive, inexpensive, non-toxic, colourless and accept as a harmless ingredient in pharmaceuticals and food. In addition, CO_2 has a low surface tension and viscosity and high diffusivity which make it attractive as a supercritical solvent. In this experimental work, we investigated the characteristics of caffeine extraction from green tea using supercritical carbon dioxide and the operating conditions extractor of such as CO_2 flow rate, CO_2 pressure and temperature. Also, we studied of the correlation between a theoretical models and experimental results.

Theory

The general equations for the process of supercritical fluid extraction are similar to those of mass transport operations involving solids and fluids such as leaching and adsorption/desorption processes [3]. Those models contain two differential solute mass balances in fluid and solid phase, in addition to a local equilibrium adsorption that describes the elation between solute and solid. In this work, the experimental data were correlated a theoretical model proposed by Goto. et al.[4]. The following assumption are first made: axial dispersion is negligible, because of small column diameter, radial dispersion is also neglected, isothermal process, the packed column is isobaric, no interaction among solutes in the fluid phase or solid phase, local equilibrium adsorption between solute and solid in pore of green tea, assumed differential bed is gradientless bed in solid and fluid phase, physical properties of the supercritical fluid are constant.

The cumulative fraction of solute extracted up dimensionless time Θ is given by Eq.(1)

$$F(\theta)=\frac{1}{(1-\alpha)}\int_0^{\theta} x d\theta=[\frac{A}{1-\alpha}]\times\{[\frac{\exp(a_1\theta)-1}{a_1}]-[\frac{\exp(a_2\theta)-1}{a_2}]\} \quad (1)$$

Where, $x=C/C_0$, $x_s=C_s/C_0$, $\theta=t/\tau$, $\phi=K_p a_p \tau$

$$a_1=\frac{(-b+(b^2-4c)^{1/2})}{2} \ , \ a_2=\frac{(-b-(b^2-4c)^{1/2})}{2} \ , \ b=\frac{\phi}{[\beta+(1-\beta)K]}+\frac{1}{\alpha}+\frac{\phi(1-\alpha)}{\alpha}$$

$$c=\frac{\phi}{[\beta+(1-\beta)K\alpha]} \ , \ A=\frac{(1-\alpha)\phi}{[\beta+(1-\beta)K]\alpha(a_1-a_2)}$$

The goodness of fit, R^2 was calculated as follows ;

$$R^2=1-\frac{\sum_{i=1}^{n}(F_i-F_i^{'})^2}{\sum_{i=1}^{n}(F_i-\overline{F_i})^2} \quad (2)$$

Experiment

The SFE apparatus shown in Fig. 1 was used to perform the experiments. 50g of green tea powder was placed in the extractor and allowed to equilibrate to a extraction temperature. CO_2 was then charged into the CO_2 storage, the high-pressure pump further compressed the CO_2 to the desired pressure. When the valve between the pump and the extractor was opened supercritical CO_2 was flowed into the extractor containing the green tea powder. The caffeine dissolved in the $SCCO_2$ was separated from the CO_2 by pressure reduction and collected in the separator. The caffeine-free CO_2 was cooled and recycled to the CO_2 recycle storage. Caffeine concentration was analyzed using an HPLC.

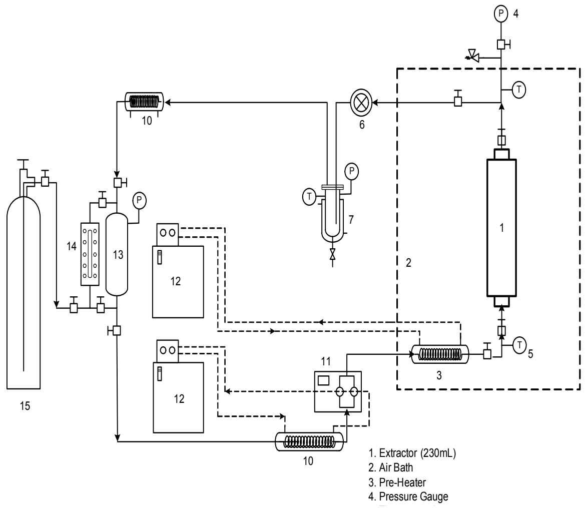

Fig.1. Schematic diagram of supercritical extraction apparatus.

Result and discussion

The experimental data of the caffeine extraction from green tea with supercritical carbon dioxide was compared with predictions from the model presented earlier. The first set of extraction experiments was carried out at a constant pressure of 40MPa, temperature of 50℃ and flow rate of 3.14 and 17.46/min. Fig. 2 show during a given interval of time more CO_2 passes through the sample when the flow rate is high thereby extracting more caffeine in a given time interval.

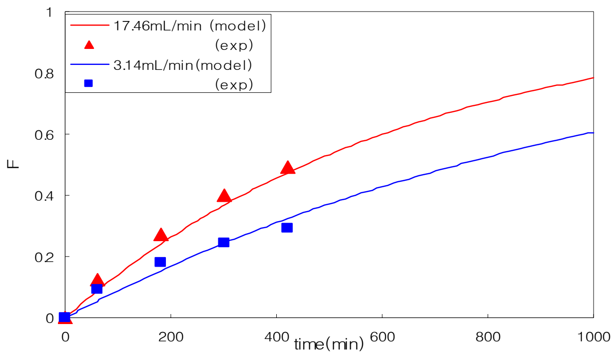

Fig. 2. Effect of solvent flow rate on extraction yield vs. dimensionless time at 50℃, 40MPa and 5% water, comparison of theatrical model and experimental model

The second set of SFE experiments was varied from 10MPa to 40MPa. The results, shown in Fig. 3, indicated that as the pressure increased the extraction rate also increased. The phenomenon is well known by the fact as the pressure increases the density of the supercritical fluid. The third set of SFE experiments was designed to investigate the effect of extraction temperature on the extraction rate. The results are presented in Fig. 4. In particular we found the optimum temperature was 50℃. Goto et al. and Stastova, Jez, Bartlova, and Sovova present experimental results that do not show any clear trend as far as temperature is concerned [4].

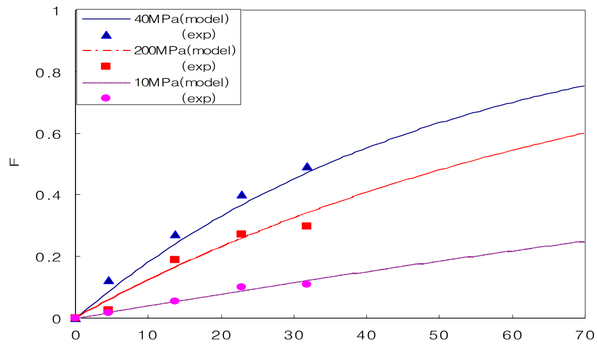

Fig.3. Effect of pressure on extraction yield vs. dimensionless time at 17.46㎖/min, 50℃ and 5% water, comparison of theatrical model and experimental model

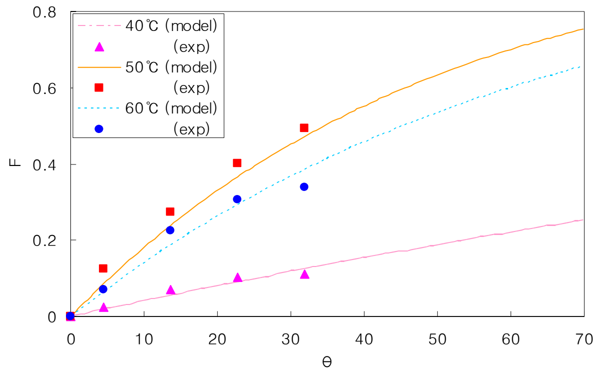

Fig. 4. Effect of temperature on extraction yield vs. dimensionless time at 17.46㎖/min, 40MPa and 5% water, comparison of theatrical model and experimental model

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