Extraction of Oil from Canola Seeds With Supercritical Carbon Dioxide: Experimental and Modeling

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Abstract— In this work extraction oil from canola (Brassica Napus) seeds with supercritical CO_2 extraction at pressure of 1500 to 2750 Psi, temperature of 308 to 333 k, and particles size 0.08 to 0.2 mm in flow rate 5 Lit/hr was investigated in a bench scale apparatus. The extraction was modeled by the sovova extended lack's model. The fluid phase mass transfer coefficient (k_f), solid phase mass transfer coefficient (ks), and hardly accessible solute (x_k) were just able parameter of Models. The broken and intact cells model fit the experimental data, quite well, showing the applicability of the model to the supercritical extraction system studied here.

Keywords: **supercritical fluid extraction, canola oil, mathematical modeling, sovova model.**

I. INTRODUCTION

Supercritical carbon dioxide (SC-CO_2) extraction of oil from seed is an alternative process to solvent extraction, hydro distillation and steam distillation because of certain advantage of SC-CO_2 with low critical temperature. The extraction of vegetable oils using supercritical carbon dioxide has been studied as a potential alternative to the current industrial process of expeller pressing, prepress solvent extraction and straight liquid solvent extraction.

Hexane is widely used as a solvent in the processing of vegetables' oils; however, the limitation of this extraction method caused the development of alternative extraction procedures such as supercritical fluid extraction with carbon dioxide. This solvent has some advantages including non toxity; non explosive, non flammability, low cost, availability and ease of being removed from extract oils. Compared to the liquid extraction, the investment costs are higher yet due to simple solvent regeneration, the operation costs are low.

SFE of seed oil has been studied by several works (Brunner, 1985; Goodarznia, & Bikini, 1998).

Mathematical models used for extraction of solute from natural matrixes are classified as [1] empirical models (Esquivel, 1999; McHugh & Krakens, 1986), [2] models based on heat transfer analogue (Mgyesy, 1993; Nayyar, 1992), [3] models based

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On differential mass balance (Nolting, 1988; Papamichail et al., 2000; Reversion, 1993), the scale-up of the equipment and the evaluation of the cost of a process cannot be done without mass transfer rate data in a convenient form.

II. Material and methods

For taking the oil extract out of Canola seeds by supercritical fluid, a thermodynamic machine is required to extract the high pressure. The designed method of SCFE has been presented by different researchers such as Van Leer, Paulaititis, Kurnik, Hollow, Red Krukonis, Eckert and Johnson, and Praunits [10], [11], [12]. In this research, this pilot can be used for separation and extraction of oil out of canola seeds using SCFE laboratory pilot. As it can be seen in the Fig (1), this system can function in static and dynamic conditions. In this system two specially designed Transfer Vessels are used to provide system pressure using Nitrogen gas. The possibility of establishing of flow of Co_2 gas in the machine in two separate, different directions by fixing the existing valve in the machine, are the characteristics of the system. The extraction vessels are made of stainless steel like other parts of the system. It also resists 10000 Psi pressure.

The container has a side glass made of silicon material, and can withstand high pressures. Therefore, seeing the contents of internal compartment and the process of formation of fluids by machine is made possible. The ability of the designed pump for rotating the supercritical fluid within the system is another unique feature of this machine in comparison to other devices. The mechanical part this pump are designed and made manually and has the ability of two-phase fluid in thermal range up to 100 degrees Celsius and with Flow Rate between 2 liters to 8 liters regardless of creation of Cavitations in the system, can be tuned by operator. This device uses air bath system to provide temperature. The designed air bath is able to provide temperature of 100 degrees Celsius uniformly.

III. Mathematical and Modeling

The broken and intact cells model was employed for correlating the experimental data (Papamichail et al., 2000; Reversion, 1993). This model based on differential mass balance equation in a fixed bed. Assuming plug flow and negligible axial dispersion, the pressure and temperature and bed void fraction (ε) are constant during the extraction in the bed, the solute accumulation in the solvent is negligible. In this model the extraction process is divided into three periods.

During the first period extraction precedes a constant rate determined by the oils solubility in sc-co_2. The second period is transition period, in these periods easily accessible solute is completely depleted at the extractor's entrance the diffusion mechanism starts. In the last period mass transfer occurs only by the diffusion in the bed and inside the solid substratum particles. Sovova (1994) obtained analytically the equation for the overall extraction curve (Eq. (1)).

$$e = m_{extr.} = \begin{cases} q y_r [1 - \exp(-z)] & q \langle q_m \quad (1) \\ y_r [q - q_m \exp(z_w - z)] & q_m \le q \langle q_n \quad (2) \\ \left[\left[x_0 - \frac{y_r}{w} \ln\left\{1 + \left[\exp\left(\frac{w x_0}{y_r}\right) - 1\right] \exp[w(q_m - q)] \frac{x_k}{x_0}\right\}\right]\right] & q \ge q_n \quad (3) \end{cases}$$

Where:

$$q_m = \frac{(x_0 - x_k)}{y_r z} \quad (4)$$

Parameter z and w are directly proportional to the fluid phase and to the solid-phase mass transfer coefficients respectively and are given below:

$$z = k_f a_0 \rho / [q(1 - \varepsilon)\rho_s] \quad (5)$$

$$w = k_s a_0 / [q(1 - \varepsilon)] \quad (6)$$

In the present work, the parameters, k_f , k_s , x_k , and y_r were adjustable and determined by minimizing the errors:

$$q_n = q_m + \frac{1}{w} Ln \frac{x_k + (x_0 - x_k)\exp\left(\frac{w x_0}{y_r}\right)}{x_0} \quad (7)$$

$$\frac{z_w}{z} = \frac{y_r}{w x_0} Ln \frac{x_0 \exp[w(q - q_m)] - x_k}{(x_0 - x_k)} \quad (8)$$

$$\text{AAD}(\%) = \frac{1}{n}\sum_{i=1}^{n} \frac{|y_{\exp} - y_{cal}|}{y_{\exp}} \times 100 \quad (9)$$

IV. Results and discussion

The operational conditions for each experiment are given in Table (1). The measured yield of oil extraction (Y) is defined by the following equation:

$$\text{Yield of oil extraction (\%)} = \frac{\text{Mass of oil extracted}}{\text{Mass of oil in seeds}} \times 100 \quad (10)$$

V. Effect of pressure

Fig (2) shows the effect of pressure on yield at temperature of 328 K and particle size 0.12 mm. with pressure increase, Co_2 density increases significantly, due to the increase of the solubility of the oil components. A faster extraction rate at low extraction times was observed when pressure increases.

The volume mass transfer coefficient in the fluid phase, $k_f a$ decreased with pressure increased. Consequently, the mass transfer resistance increases contrast the mass transfer coefficient in the solid phase $k_s a$ increases as pressure increases and the resistance in the solid decreases. [Table (2)].

VI. Effect of temperature

As it is shown in Fig (3), as temperature increases, the extraction rate decreases, the solubility of oil directly affects the extraction rate and it is controlled by a balance between the SC-Co_2 density and the oil vapor pressure. The overall mass transfer coefficient in the fluid phase increased with increase of temperature. In contrast the overall mass transfer, coefficient in the solid phase increases with temperature at 2250 Psi. (Table 2).

VII. Effect of particle size

At 2250 Psi pressure and 328 K, the yield increases with decreasing particle size Fig (4), grinding of canola before extraction not only increase the interfacial area but also releases oil from the broken cells, results indicated that extraction rate is high if the oil is released on the surface of particle, and it is comparable very slow if it is embedded in the kernel particles.

Moreover, after milling, the diffusion paths in the solid matrix become shorter and cause the resistance of smaller intra particles to diffusion. But production of very small particles could produce bed caking with formation of channels along the bed in which supercritical fluid can preferentially flow, thus reducing the extraction efficiency in the fluid phase, $k_f a$, and solid phase, $k_s a$, increased [Table (2)].

VIII. Conclusion

The results indicated that the extraction curves in a plot of extraction yield versus time are significantly affected by the extraction pressure and particle size. But temperature has a slight effect on the extraction curves. Model of sovova represented canola oil extraction well. A theoretical model based on the evidence that part of the oil is freely available to the solvent after pre-treatment of the seeds was successfully used to fit the experimental extraction curves.

Nomenclature

- a specific interfacial area (m^2/m^3)
- e Mass of extract (k_g)
- k_f a fluid-phase mass transfer coefficient (s^{-1})
- k_sa solid-phase mass transfer coefficient (s^{-1})
- n number of data point
- q Mass of solvent (k_g)
- q_m q value at start of the extraction of difficult Accessible oil (k_g/k_g)
- q_n q value at end of the extraction of easily Accessible (k_g/k_g)
- $q^{\cdot}$ specific flow rate (k_g/s)

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W parameter of solid-phase mass transfer
Ps density of solid phase
ρ density of solvent (k_g/m^3)
ε bed voidage
Z parameter of fluid phase mass transfer
y_r oil solubility in the supercritical fluid
x_k hardly accessible solute (g/g)
x_0 initial oil concentration in the solid (g/g)

Appendix

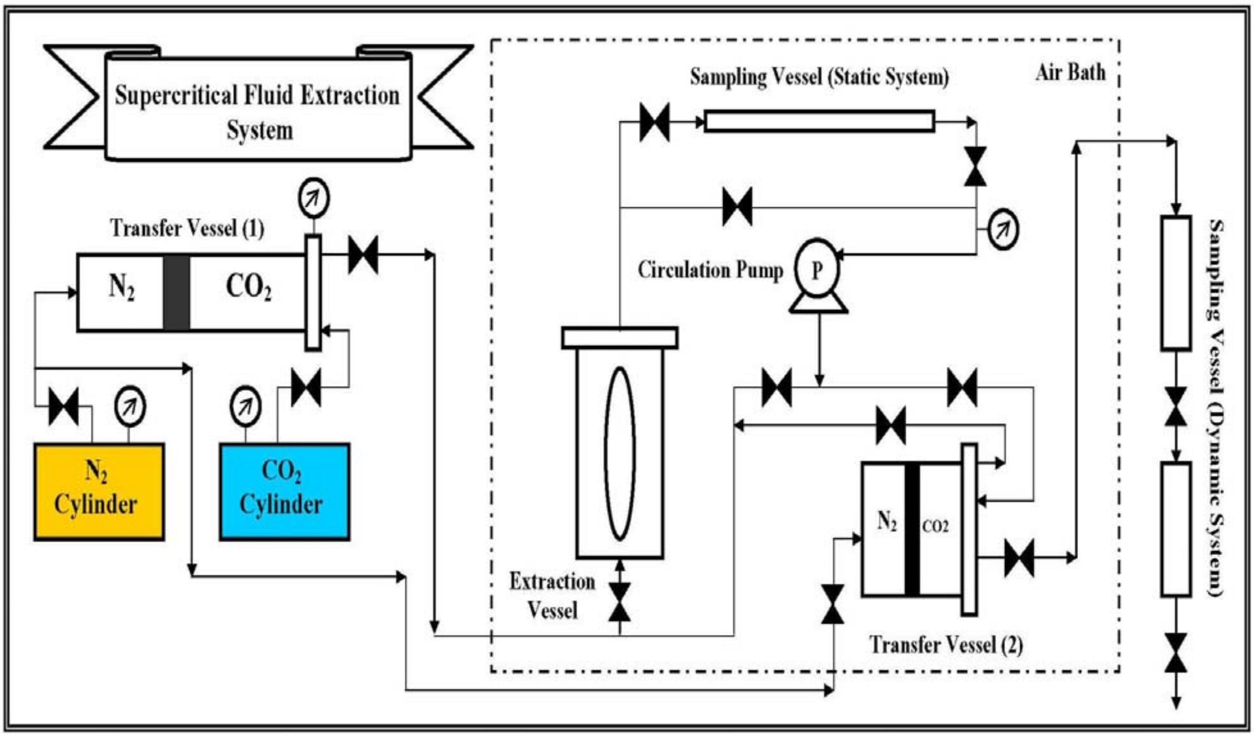

Fig (1). Schematic diagram of the Supercritical Fluid Extraction System (SCFE) for Extraction of oil from canola seed with supercritical carbon dioxide

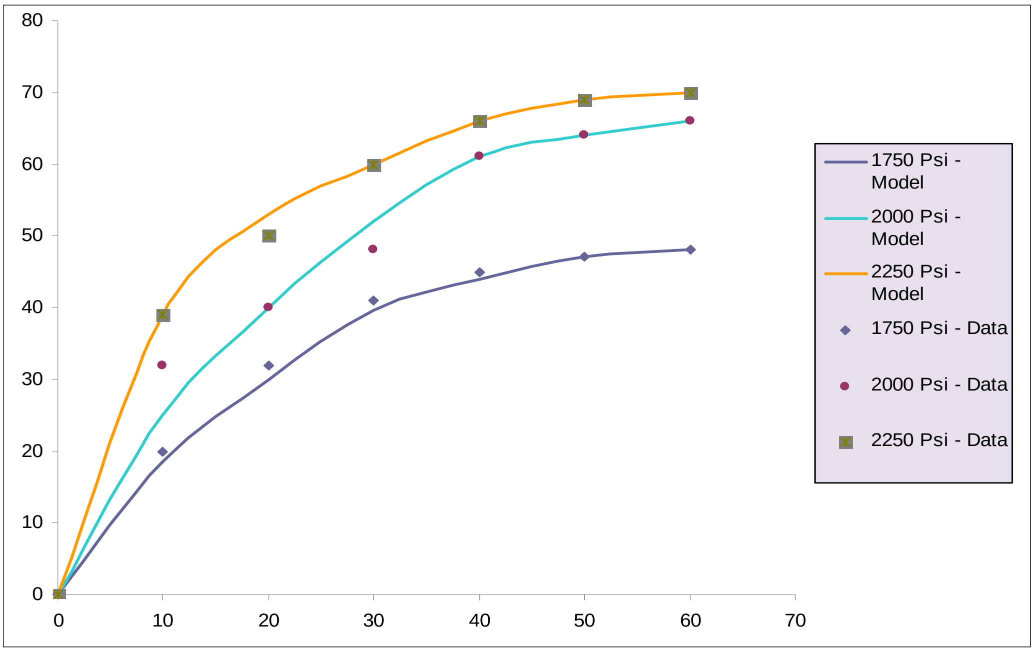

Fig (2): Effect of pressure on the extraction yield of oil at T= 328 K and dp = 0.12 mm

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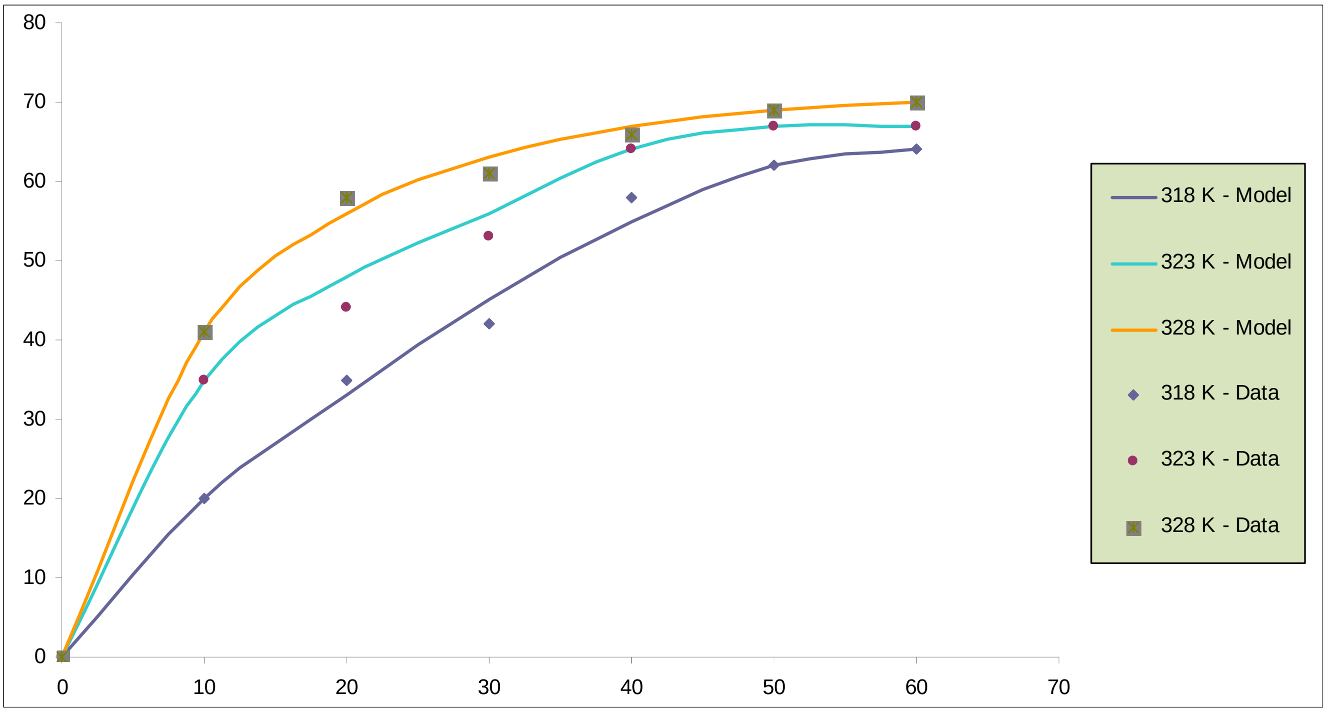

Fig (3): Effect of temperature on the extraction yield of oil at P= 2250 Psi and dp= 0.12 mm

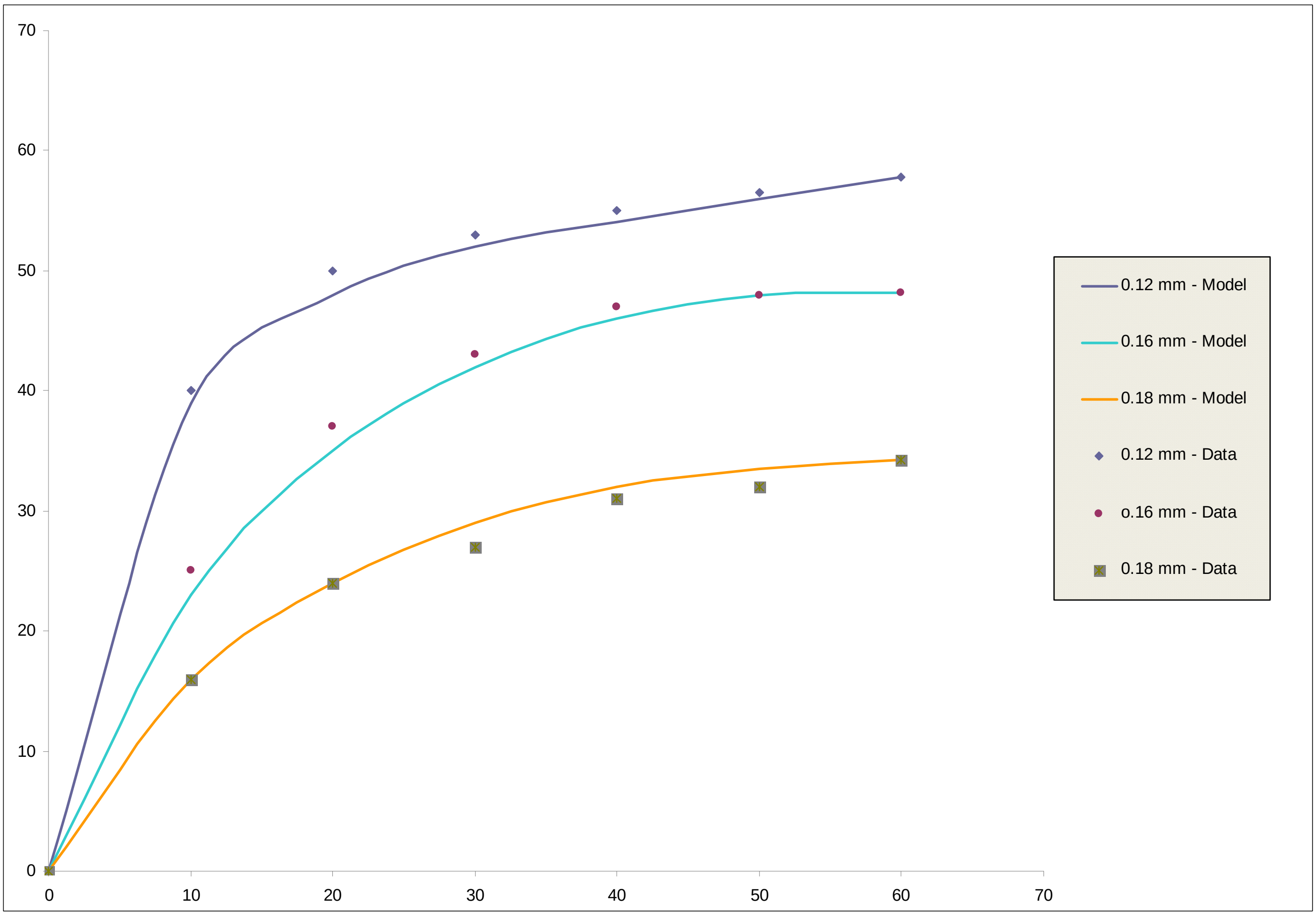

Fig (4): Effect of particle size on the extraction yield of oil at P= 2250 Psi and T= 328 K

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Table (1): Yield of oil extracted under different extraction process conditions

Run	Pressure (Psi)	Temperature (K)	particle size (mm)	Yield (gr/gr)
1	1500	308	0.2	20.14
2	1750	318	0.18	34.32
3	2000	323	0.16	48.25
4	2250	308	0.14	55.09
5	2250	328	0.12	58.45
6	2500	318	0.1	58.25
7	2750	323	0.08	57.81

Table (2): Calculated values of parameters and AAD (%) in BIC model.

Run	X_0	y_r	X_k	$K_f a$	$K_s a * 10^{+5}$	% AAD
1	0.54	0.00974	0.463	0.0079	3.01	5.03
2	0.54	0.00583	0.462	0.008	0.104	7.1
3	0.54	0.003	0.5	0.0155	1.157	5.43
4	0.54	0.00967	0.47	0.0056	1.58	3.85
5	0.54	0.00948	o.472	0.00486	1.356	4.01
6	0.54	0.00953	0.4805	0.00756	1.597	5.25
7	0.54	0.00298	0.52	0.0159	1.52	4.87

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