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Optimization of Soybean Oil by Modified Supercritical Carbon Dioxide

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***Abstract*—**The content of omega-3 in soybean oil is important in the development of infants and is an alternative for the omega-3 in fish oils. The investigation of extraction of soybean oil is needed to obtain the bioactive compound in the extract. Supercritical carbon dioxide extraction is modern and green technology to extract herbs and plants to obtain high quality extract due to high diffusivity and solubility of the solvent. The aim of this study was to obtain the optimum condition of soybean oil extraction by modified supercritical carbon dioxide. The soybean oil was extracted by using modified supercritical carbon dioxide (SC-CO_2) under the temperatures of 40, 60, 80 °C, pressures of 150, 250, 350 Bar, and constant flow-rate of 10 g/min as the parameters of extraction processes. An experimental design was performed in order to optimize three important parameters of SC-CO_2 extraction which are pressure (X_1), temperature (X_2) to achieve optimum yields of soybean oil. Box Behnken Design was applied for experimental design. From the optimization process, the optimum condition of extraction of soybean oil was obtained at pressure 338 Bar and temperature 80 °C with oil yield of 2.713 g. Effect of pressure is significant on the extraction of soybean oil by modified supercritical carbon dioxide. Increasing of pressure will increase the oil yield of soybean oil.

***Keywords*—**Soybean oil, SC-CO_2 extraction, yield, optimization.

I. Introduction

THERE are many extraction processes that can be used to extract and carry out oil and bioactive compounds inside of solutes. However, supercritical carbon dioxide extraction is a green alternative technology compared to conventional processes such as accelerated Soxhlet extraction and hydrodistillation [1]-[3]. Moreover, the benefits of using supercritical carbon dioxide as solvent are inexpensive and non-toxic solvents that can be easily separated from the extracts. From the previous research, the supercritical carbon dioxide extraction process has been successfully used to extract bioactive compounds from herbaceous plants. Other applications include the extraction of oils from avocado, rosehip, *Pithecellobium jiringan* (Jack), Prain seeds, peanut skin and ginger [4]-[8]. Additionally, the supercritical CO_2 extraction process can be applied in investigating the matrices of plants and herbs [9]-[11].

Soybean or its scientific name *Glycine Max* is classified as an oilseed. Soybean has history in all parts of the world as a major crop and is widely consumed as an alternative source of protein. Some examples of soy related products are soy vegetable oil, soy milk, and others. Previous studies showed that conventional extraction processes have been already applied on the extraction of soy bean such as Soxhlet extraction and ultrasound-assisted extraction [12], [13]. Extraction time, amount of solvent, and toxicity of solvent are the problems in the extraction of *Orthosiphon stamineus.* Therefore, supercritical carbon dioxide extraction is one of the solutions for extraction of soybean because supercritical carbon dioxide extraction has short extraction time, low consumption of solvents, and non-toxic solvent [14]-[17]. However, due to its limited polarity, supercritical CO_2 can only extract non-polar bioactive compounds from plants and herbs matrices [18], [19]. To overcome this problem, modification of the supercritical CO_2 process is needed to enhance the polarity of solvents used for the extraction of bioactive compounds. The modification process involves the addition of ethanol as a modifier during extraction [4], [20], [21]. The choice of ethanol as a modifier is due to its low toxicity compared to methanol and ethylene glycol [18], [19]. Therefore, the objective of this study is to investigate and optimize the effect of process parameters on supercritical carbon dioxide extraction with oil yield of soybean as a response.

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II. Materials and Methods

A. Plant Material

The sun-dried soybean was powdered using dry-mill grinder with the aim of increasing the samples' surface area [22], [23]. The dried powdered plants were then sieved by using sieve trays with particle size of 300 µm. The chosen size was made based on the preliminary result which showed that particle size of 300 µm was the optimum size for extraction process. At the end of this stage, the material's powder was put inside the sealed plastic bag and stored in a refrigerator at temperature −20 °C to maintain the freshness of samples before being used for extraction [24].

B. Chemicals

Liquid carbon dioxide (99% purity) was used in the supercritical extraction apparatus purchased from Kras Instrument, Johor Bahru, Malaysia. An analytical grade of ethanol (99.86%) was purchased from Permula Sdn Bhd, Johor Bahru, Malaysia.

C. Modified Supercritical Carbon Dioxide Extraction

The procedure of SC-CO_2 extraction process is discussed in detail. To begin with, the collection vials were prepared, labelled and weighted. Meanwhile, the oven and chiller were turned on. The chiller and heater temperature were set at 5 °C and 50 °C, respectively. Next, 15 g of powdered soybean that has been processed through the sampling process was placed into the vessel. The extraction vessel was then fixed properly in the oven. The investigated values of pressure and temperature conducted in this experiment were: pressure of 150, 250 and 350 bar; temperature: 40, 60, and 80 °C and each set of extraction processes were run for 3 hours. Then, the back pressure regulator (Tescom 10000) had been controlled while the temperature was adjusted by setting the oven (France Etuves H555) temperature. Next, the CO_2 gas was pumped into the system continuously with supercritical pump (Separex Piston Pump P200GP50) at flow rate of 10 g/min while ethanol as modifier was pumped into the system at flow rate of 0.5 g/min. The extract was collected for every 15 minutes of the extraction process. The extract oil obtained was placed in the collection vials, sealed and stored in the chiller at 2.7 °C to prevent any possible degradation of the product.

D. Analysis of Oil Yield Percentage

The comparison of oil yield percentage of soy bean oil was expressed in terms of mass percentage of the samples:

$$\text{Percentage of Oil Yield} = (m_1/m_2) \times 100\% \tag{1}$$

where, m_1 is the mass of extracted oil (g) and m_2 is the mass of sample (g).

E. Experimental Design

An experimental design was performed in order to optimize three important parameters of SC-CO_2 extraction which are pressure (X_1), temperature (X_2) to achieve optimum yields of soybean oil. Box Behnken Design was applied for experimental design.

TABLE I
FACTORS AND LEVELS TESTED FOR THE EXPERIMENTAL DESIGN

Factors	**Low Level**	**Medium Level**	**High Level**
Pressure, (X_1, MPa)	-1	0	+1
Temperature, (X_2, °C)	-1	0	+1

Response surface methodology (RSM) was applied to optimize the operating parameters of SC-CO_2 for the extraction of soybean oil. RSM model or regression equation which includes linear and quadratic variables as well as interaction terms was used to fit the first and second-order polynomial equation based on the experimental data as follows:

$$Y = B_0 + \sum_{i=1}^{k} B_i X_i + \sum_{i=1}^{k} B_i X_i^2 + \sum_i \sum_j B_{ij} X_i X_j \tag{2}$$

where Y is the predicted response, B_0 is a constant, B_i, B_{ii}, B_{ij} are the interaction of effect, X_i and X_j are the coded value of factor. Finally, the response surfaces of the variables inside the experimental domain were analyzed by analysis of variance (ANOVA).

III. RESULTS AND DISCUSSION

Optimization of extraction process was conducted using three main parameters: the effect of pressure, temperature, and rate of co-solvent. extract yield and high antioxidant activity on peanut skin extract. The two variables were investigated by using second order polynomial statistical model. Preliminary studies were needed to determine fixed variables of the optimization. The quadratic effect of the treatments variables, their interactions, and coefficients on the response variables were obtained by ANOVA. Experimental design was made by using Box Bhenken design as shown in Table II.

TABLE II
VARIABLES AND RESPONSES OF EXTRACTION OF SOYBEAN OIL BY MODIFIED SUPERCRITICAL CARBON DIOXIDE

Run	Variables		Responses	
	Pressure, Bar (X_1)	Temperature, °C (X_2)	Oil Yield, gram (y_1)	Model, gram
1	60	350	1.929	2.423
2	80	250	2.145	2.181
3	40	350	2.463	2.142
4	80	350	2.874	2.704
5	60	150	0.582	0.817
6	40	250	2.084	2.426
7	60	250	2.381	2.304
8	40	150	1.534	1.342
9	80	150	0.334	0.293
10	60	250	2.607	2.304

From Table II, shows that the optimum condition to extract oil from soybean was at pressure 338 Bar and temperature 80°C with response oil yield 2.713 gram. Moreover, the maximum oil yield obtained was 2.874 gram at condition pressure 350 bar and temperature 80 °C. Furthermore, the minimum soybean oil yield was 0.334 gram at condition pressure 150 bar and temperature 80 °C

The ANOVA is presented in Table III where soybean oil yield is a response, and pressure and temperature are the variables. Based on the statistical analysis, a quadratic equation successfully optimized the extract yield of soybean. The analysis showed that both models were statistically significant (sig. <0.05) at 95% confidence. The coefficient of determination, R^2, of quadratic model was 0.893 and adjusted coefficient of determination was 0.803. From the coefficient of determination, the quadratic model has successfully fitted the experimental data.

Fig. 1 shows that the experimental data vs. predicted quadratic model of extraction soybean oil by modified supercritical carbon dioxide. Fig. 1 indicates that quadratic model has successfully fitted the experimental data due to the R^2 and adj R^2. The quadratic model of extraction soybean oil by modified supercritical carbon dioxide is shown in (3).

$$Y = -0.594 - 0.0564\, X_2 + 0.0302\, X_1 + 0.002014\, X_1 X_2 - 0.0000684 X_1^2 \tag{3}$$

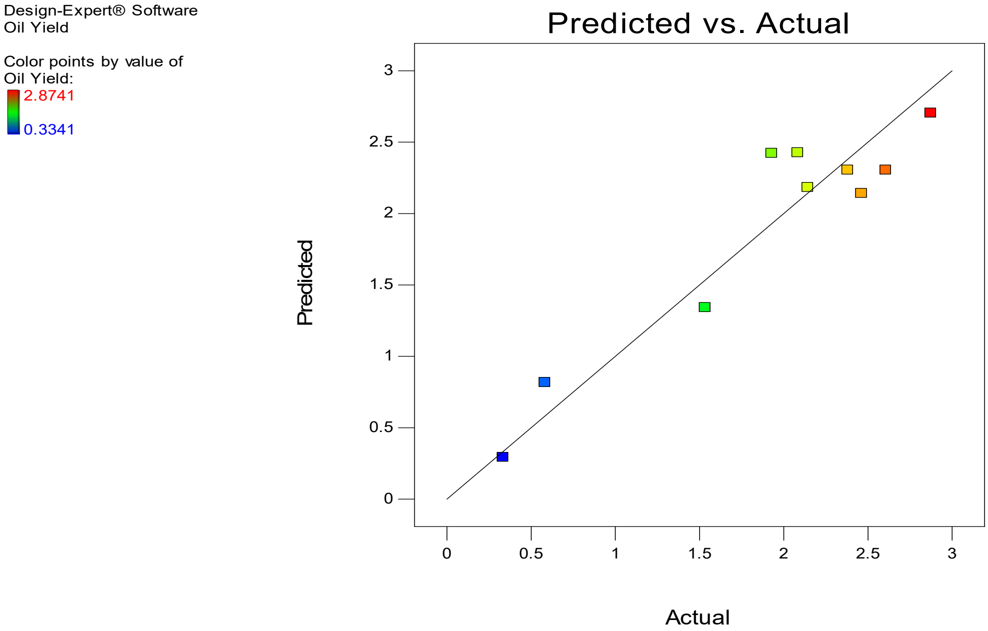

Fig. 1 The experimental data vs predicted quadratic model of extraction soybean oil from modified supercritical carbon dioxide

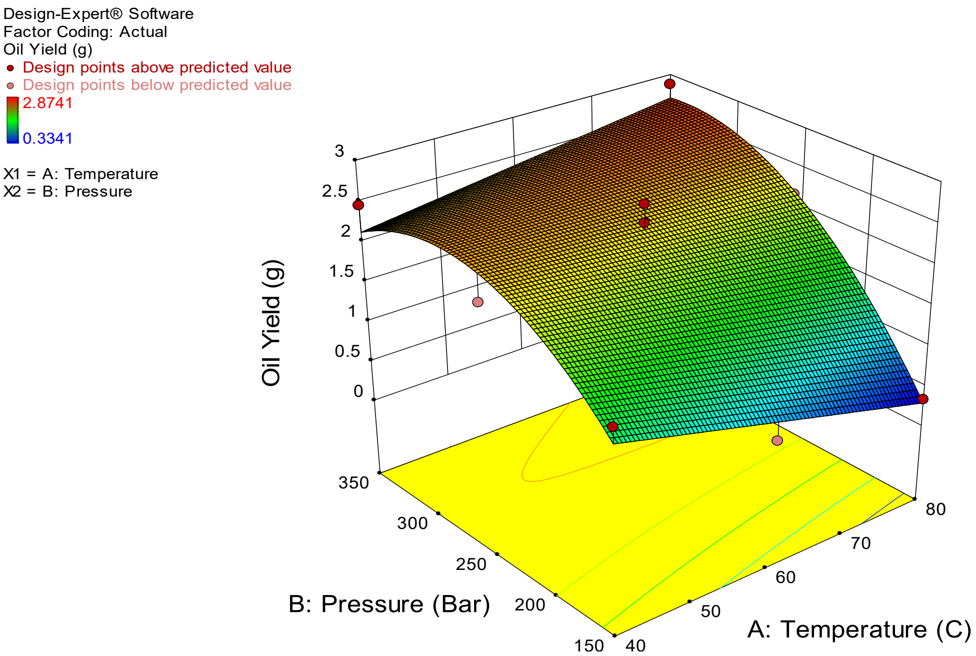

Fig. 2 3-D contour plot of the experimental data extraction soybean oil from modified supercritical carbon dioxide

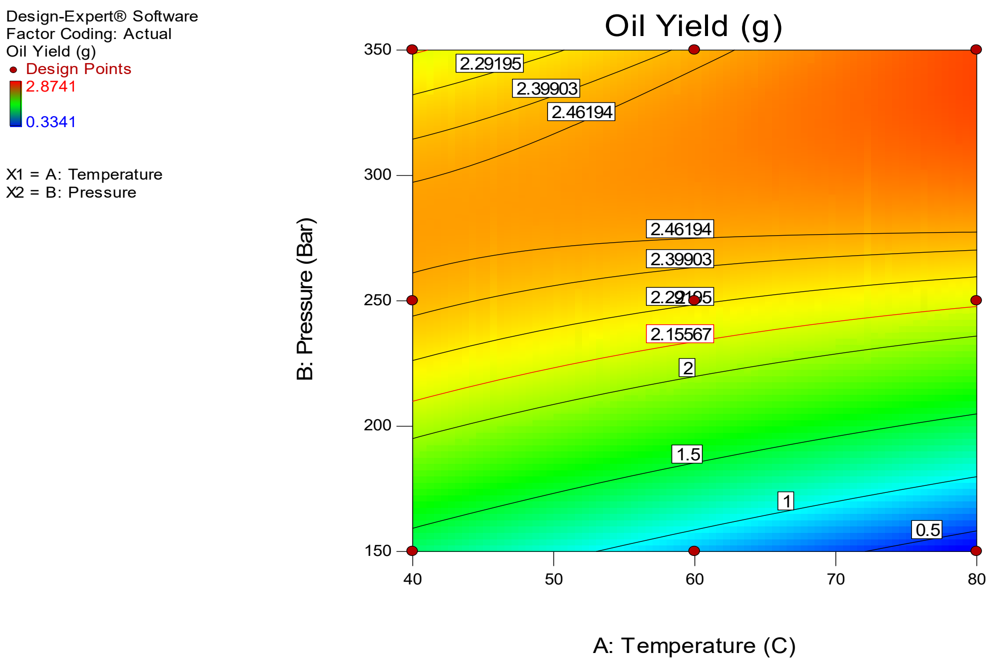

Fig. 3 Contour plot of the experimental data extraction soybean oil from modified supercritical carbon dioxide

TABLE III
ANOVA TABLE OF EXTRACTION OF SOYBEAN OIL BY MODIFIED SUPERCRITICAL CARBON DIOXIDE

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	5.78	5	1.16	7.30	0.0385	significant
X_1-Temperature	0.089	1	0.089	0.56	0.4958	
X_2-Pressure	3.87	1	3.87	24.41	0.0078	
$X_1 X_2$	0.65	1	0.65	4.10	0.1128	
X_1^2	0.052	1	0.052	0.33	0.5984	
X_2^2	1.17	1	1.17	7.42	0.0528	
Residual	0.63	4	0.16			
Lack of Fit	0.61	3	0.20		0.2533	not significant
Pure Error	0.025	1	0.025	7.98		
Cor Total	6.41	9				

From the coefficient of quadratic model on (3) shows that temperature gives a big influence on the extraction of soybean oil due to the highest coefficient model followed by pressure (0.564 > 0.0301).

From Figs. 2 and 3, it can be summarised that increasing pressure (150 bar- 350 bar) and temperatures (40–60 °C) increased the soybean oil yield. Increasing pressure will increase the density of supercritical CO_2. This is because the increase in interaction between CO_2 molecules and soybean as solute enhance the dissolution of oil into the solute [24]. Furthermore, the increase in density increases the solubility and diffusivity of extraction process [25]. This is similar to the result for extraction of prain seeds by modified supercritical carbon dioxide which reported that increasing of pressure will increase the prain seeds oil yield [6]. Increasing of pressure will increase diffusivity and solubility of solvent to extract soybean oil from the solute [24]. Furthermore, increasing of temperature enhances the soybean oil yield at a constant highest pressure (350 Bar). However, the increasing temperature decreases the soybean oil yield at a lowest constant pressure. At the highest pressure condition, the effect of vapour solute condition is significant during modified supercritical CO_2 [6]. Furthermore, at lowest constant pressure, increasing of temperature decreases the soybean oil extract. This is because increasing of temperature at a constant pressure will degrade the quality of extract and vaporize the extract [26]. This is similar to the result of extraction peanut skin oil by modified supercritical carbon dioxide where increasing of temperature will decrease the oil yield of peanut skin oil [24]. Increasing of temperature will decrease the solubility power of solvent to extract the oil from the solute [27].

IV. CONCLUSION

In this study, the quadratic model was successfully fitted to experimental data of extraction of soybean oil by supercritical carbon dioxide. The optimum recovery of soybean oil yield was 2.713 g at conditions 338 bar, 80 °C. However, the optimum temperature for extraction was not deduced. Nevertheless, the increase in pressure enhanced the yield of soybean oil at constant temperature condition.

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