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SUPERCRITICAL CARBON DIOXIDE EXTRACTION OF RED PITAYA (*Hylocereus polyrhizus*) SEEDS: RESPONSE SURFACE OPTIMIZATION, FATTY ACID COMPOSITION AND PHYSICOCHEMICAL PROPERTIES

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ABSTRACT

A new technology called supercritical fluid extraction (SFE) was performed to obtain oil from red pitaya (*Hylocereus polyrhizus*) seeds. The extraction process was optimised by response surface methodology (RSM) and the effects of extracting variables namely temperature (35-65ºC) and pressure (1500-5000 psi) were evaluated. A model was developed using central composite design (CCD) for the determination of optimum condition that gave highest oil yield. The highest oil yield was predicted to be about 6.93 wt%, under optimal conditions temperature of 47ºC and pressure of 4750 psi. At optimum conditions obtained, triplicate extractions were performed and found that average experimental extraction yield of oil was 6.88 ± 0.06% and in a good agreement with the predicted value. Gas chromatography mass spectrometry (GC-MS) analysis was carried out to identify the chemical composition of the oil and compared with *n*-hexane extracted oil. GC-MS analysis revealed negligible differences in fatty acid composition of oil extracted from both methods with linoleic acid as the major component. In the studies of oil quality, the important physicochemical properties of the extracted oil were also analyzed.

Key words: Red pitaya, *Hylocereus polyrhizus*, supercritical fluid extraction, optimization, response surface methodology

INTRODUCTION

Pitaya or dragon fruit is a beautiful exotic fruit of the cactus species from the genus of *Hylocereus*. One of the pitaya varieties is *Hylocereus polyrhizus* or also known as red pitaya. It is red-skinned pitaya fruit with red flesh. Previous studies disclosed that oil extracted from pitaya seed rich in essential fatty acids (EFAs). They were of unsaturated fatty acids (UFA), namely linoleic acid and linolenic acid (Ariffin *et al*., 2009; Chemah *et al*., 2010). EFAs have been proved to show beneficial effects especially when applied on the skin. For example, the presence of certain EFAs enhanced skin permeation of co-administered molecules, balance the metabolism of the skin by controlling the oils flow and nourish collagen that is supporting structure beneath the skin. In addition, deficiencies

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of EFAs may contributed to many other health issues such as abnormal lipid barrier structure and function, epidermal hyper-proliferation, altered production of anti-inflammatory compounds where this resulted in a plethora of skin problems such as psoriasis, atopic eczema and acne (Simopoulos, 1991). However, both linoleic acid and linolenic acid cannot be synthesized in the body. Since the EFAs are necessary for proper skin function and offer many other health benefits, therefore, they must be present in the diet (Gani *et al*., 2015).

There are various extraction processes described in the literatures, supercritical fluid extraction (SFE) is a new technology that may be utilized as an alternative to classical techniques including physical pressure and organic solvent extraction using soxhlet. SFE with carbon dioxide (CO_2) as a solvent offers considerable advantages over traditional extraction processes including its desirable properties such as non-toxic, non-flammable, cost efficient (high purity of CO_2 solvent available at low cost and has major advantage of lack of solvent residue in the product), non-explosive, higher extraction rate (shorter extraction time) and enhance selectivity (Costa *et al*., 2015; Mukhopadhyay, 2000; Rai *et al*., 2016; Shao *et al*., 2014). Its non-toxic organic solvent may avoid the environment and sample contamination. Moreover, supercritical CO_2 has suitable ability to extract the lipophilic substances such as essential oils from seeds due to its non-polar properties (Uquiche *et al*., 2015).

Generally, the SFE efficiency is affected by many variables including temperature, pressure, CO_2 flow rate, extraction time, particle size and co-solvent content. Among those variables, temperature and pressure are considered as the principal variables that affect the behavior of SFE. This is because the solvent power of the supercritical fluid is determined by these variables (Uquiche *et al*., 2015). In attempt to study the effect of temperature and pressure, the other variables are maintained to give constant value. This paper is focus on experimental study of supercritical carbon dioxide (SC-CO_2) extraction of red pitaya seed oil. Furthermore, to our knowledge, there are limited studies on the literature on this extraction process to evaluate the compound extraction profile from red pitaya seed oil. The aim of this study is to develop the statistical model to optimize the SC-CO_2 extraction conditions to obtain highest oil yield. Response surface methodology (RSM) was performed to interpret the oil yield obtained by suggested experiment runs. Gas chromatography mass spectrometry (GC-MS) was utilized to determine the fatty acid content of SC-CO_2 extracted oil and compared with the *n*-hexane extracted oil. At the end, the determination of physicochemical properties of the extracted oil was also carried out to facilitate the potential use.

MATERIALS AND METHODS

Materials

Red pitaya fruit was obtained locally from vicinity of Sepang, Malaysia. Red pitaya seeds were then manually separated from the red fleshed and pulp in the lab. The seeds were cleaned and washed under running water until all the flesh and pulp removed. Seeds were then dried, crushed into smaller particles in a mill and keep in a desiccator until further analysis. A pressurized deep tube cylinder containing liquefied CO_2 of purity 99.9% was supplied by Poly Gas Sdn. Bhd. The entire standard chemical such as *n*-hexane with purity of 99.99%, ethanol, methanol and sodium chloride were purchased from Merck (Darmstadt, Germany), respectively. Fatty acid methyl esters (FAMEs) FAME 37 Mix standard solution was obtained from Sigma (St. Louis, MO, USA). All other chemicals were of analytical grade.

Supercritical carbon dioxide (SC-CO_2) extraction

SC-CO_2 extraction was carried out in a 60 mL extraction vessel using SFE system (OV-SCF) supplied by Taiwan Supercritical Technology Co., Ltd. 20 g of dried, ground red pitaya seeds were placed into the extraction vessel (4.5 cm internal diameter and 14.5 cm in height). In order to keep CO_2 liquefied, CO_2 was fed from a gas cylinder equipped with a cooler circulator. The liquefied CO_2 was pressurized under the needed pressure using an air-booster pump and fed the CO_2 into the vessel. The percentage of oil yield was calculated using Eq. (1) and average experimental values were computed (Table 1).

$$\text{Oil yield (\%)} = \frac{\text{Amount of extracted oil (g)}}{\text{Amount of ground seeds (g)}} \times 100 \qquad \text{Eq. (1)}$$

Experimental design and statistical analysis

RSM was applied in the optimization process and evaluation of the effect of two different operating variables including temperature (35–65ºC) and pressure (1500–5000 psi) on SC-CO_2 extraction yield. The parameter of SC-CO_2 flow rate was kept constant. All other parameters were also maintained constant such as the extractions have been carried out for 60 min (dynamic extraction time) with 20 g of seed particle. A two-factor-five-level rotatable central composite design (RCCD) has been chosen in the extraction of red pitaya seed oil, which resulted in 13 experimental runs consisted

4 factorial points, 4 axial points and 5 central points. In order to minimize systematic errors, all experiments were carried out in triplicate and random order.

The experiment data was fitted into a multiple regression model (second order polynomial equation) and the general form of that equation is presented below:

$$Y = \beta_o + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i=1}^{k} \sum_{j>1}^{k} \beta_{ij} X_i X_j \quad \text{Eq. (2)}$$

where Y is the response (percentage oil yield); X_i and X_j are the independent variables (i and j range from 1 to k); β_o, is a contant (intercept coefficient), β_i, β_{ii} and β_{ij} are coefficients of variables for linear, quadratic and interaction terms, respectively; k is the number of independent variables (k = 2 in this study) (Maran & Priya, 2015; Sodeifian *et al*., 2016b). In order to evaluate whether the constructed models were adequate fitted to the experimental data, corresponding analysis of variance (*ANOVA*) was applied. Numerical optimization function of the software was used in the determination of the optimum conditions for the extraction of red pitaya seed oil using SC-CO_2.

Solvent extraction

A solvent extraction using *n*-hexane as a solvent was done as a reference extraction in order to compare the results on the oil composition. 20 g of dried red pitaya seed was ground, soaked in the *n*-hexane and left overnight. The solution was then evaporated to discard the *n*-hexane from the oil in a rotary evaporator.

Determination of fatty acid composition

Composition of fatty acids in the red pitaya seeds oil was determined by means of gas chromatography mass spectrometry (GC–MS) analysis using Agilent Technologies 7890A gas chromatograph coupled to Agilent Technologies 5975 mass spectrometer and equipped with HP-88 fused silica capillary column (100 m x 250 µm i.d; film thickness 0.25 µm; Agilent, USA). Beforehand the GC-MS analysis, methyl esters of fatty acids have been prepared by the method suggested by Malaysian Palm Oil Board (MPOB) Test Methods (MPOB Test Methods, 2005). Associated with chemical stability, methyl esters are often preferred over fatty acids for the GC-MS analysis. 1 µL aliquot of the prepared FAME was injected into a split mode GC. The oven temperature was maintained at 150ºC for 5 min and then the temperature was increased to 240ºC with a ramping rate of 4ºC/min and held for 15 min. The temperatures of 250ºC and 260ºC were set for injector and detector, respectively. Helium was utilized as carrier gas with total flow rate of 25 mL/min. Fatty acid peaks were identified using a standard 37 FAME mixture (Sigma, St. Louis, MO, USA).

Measurement of physicochemical properties

A specific gravity bottle and refractometer at room temperature of 25ºC were used to determine the specific gravity and refractive index, respectively. Iodine, saponification and acid values were determined according to standard Test Methods modified from American Oil Chemist's Society (AOCS) as well as Malaysian Palm Oil Board (MPOB) Test Methods (MPOB Test Methods, 2005). All measurements were performed in triplicate. Variations among the measurements were negligible and the mean values for each measurement were computed.

RESULTS AND DISCUSSION

Analysis of RSM model

The experimental data were analyzed and interpreted using Design Expert Software version 7.0.0 (Stat-Ease Inc., Statistics Made Easy, Minneapolis, MN, USA). Table 1 represents the design experiments in term of uncoded variables, experimental and predicted extraction yield for two-factor-five-level RCCD response surface analysis of red pitaya seed oil extraction.

From model fitting technique, it was observed that the predicted values obtained were satisfactorily correlated to the experimental values. After fitting of the obtained data to multiple models including linear, quadratic and cubic, *ANOVA* showed that the relation between the oil yield (response) and independent variables (temperature and pressure) was most suitably described with a quadratic polynomial model as follows:

$$\text{Oil yield (\%)} = 5.36 - 0.67A + 2.42B + 0.29AB - 0.52A^2 - 0.96B^2 \quad \text{(Eq. 3)}$$

where A is the temperature; B is the pressure. The results of model summary statistics and *ANOVA* of RSM are shown in Table 2. As compared to quadratic model, lower R^2, adjusted R^2 and predicted R^2 values were observed for linear and interactive (2FI) models, whereby, the cubic model was reported to be aliased. The high R^2 (0.9884), adjusted R^2 (0.9802) and predicted R^2 (0.9405) values and the good agreement between both predicted R^2 and adjusted R^2 values of quadratic model clearly demonstrates that the model could be used to analyze the relationship between independent variables and response (Maran *et al*., 2013; Sodeifian *et al*., 2016b). Hence, it can be

Table 1. Design experiments in term of uncoded variables, experimental and predicted values obtained for RCCD response surface analysis

Std. run	Point type	Temperature, A (°C)	Pressure, B (psi)	Experimental Yield (%)	Predicted Yield (%)
1	Factorial	39	2013	2.34	2.41
2	Factorial	61	2013	0.48	0.50
3	Factorial	39	4487	6.98	6.68
4	Factorial	61	4487	6.28	5.93
5	Axial	35	3250	5.16	5.26
6	Axial	65	3250	3.20	3.38
7	Axial	50	1500	0.13	0.008
8	Axial	50	5000	6.46	6.86
9	Central	50	3250	5.20	5.36
10	Central	50	3250	5.43	5.36
11	Central	50	3250	5.74	5.36
12	Central	50	3250	5.35	5.36
13	Central	50	3250	5.10	5.36

Table 2. RSM model summary and *ANOVA* of the SC-CO_2 extraction of red pitaya seed oil

Source	Std. Dev.	*R*-square	Adjusted *R*-square	Predicted *R*-square	*p*-Value Prob > *F*	PRESS
Linear	0.93	0.8548	0.8257	0.7566	<0.0001	14.40
2FI	0.96	0.8604	0.8139	0.7189	0.5597	16.63
Quadratic	**0.31**	**0.9884**	**0.9802**	**0.9405**	**0.0002**	**3.52**
Cubic	0.28	0.9932	0.9837	0.8209	0.2645	10.59
Source	Sum of squares	df	Mean square	*F*-value	Prob > *F*	Significance
Model	58.47	5	11.69	119.58	<0.0001	Significant
Residual	0.68	7	0.098			
Lack of fit	0.44	3	0.15	2.43	0.2054	Insignificant
Pure error	0.24	4	0.061			
Cor Total	59.15	12				

concluded that, quadratic model was most suitable for prediction of experiments.

Based on result of *ANOVA*, the higher model *F*-value of 119.58 with "Prob > *F*" value of <0.0001 implied the significance of the developed models. The Prob > *F* value of the lack of fit is 0.2054 indicated insignificant lack of fit, therefore the fitness of the developed model was proven. Adequate precision (signal to noise ratio) of 32.27 for the response indicated an adequate signal and the best fit of developed models as this value should be greater than 4 to navigate the design space excellently. From the results, it was found that this RSM model can be used for prediction of experimental data in the range of domain studied.

The coefficient of the empirical model is presented in Table 3. The higher regression coefficient and smaller Prob > *F* value (less than 0.05) for those variables and their interactions demonstrated that they have a greater impact on the oil yield (He *et al*., 2010). Based on the results obtained, linear term of pressure, *B* (Prob > *F* value of <0.0001) and quadratic term of pressure, B^2 (Prob > *F* value of 0.0001) showed that these variables gave more significant influence on the oil yield, followed by linear term of temperature, *A* (Prob > *F* value of 0.0005) and quadratic term

Table 3. Regression coefficient and *p*-value (Prob > *F*) of the model

Source	Coefficient	Prob > *F*
Intercept	5.36	
A	-0.67	0.0005[a]
B	2.42	<0.0001[a]
AB	0.29	0.1060
A^2	-0.52	0.0032[a]
B^2	-0.96	0.0001[a]

[a]Significant at "Prob > F" less than 0.05.

of temperature, A^2 (Prob > F value of 0.0032). The interaction temperature-pressure, AB variable showed no significant effect.

Effect of operating variables

Figure 1 illustrates the effect of varying temperature and pressure on the oil yield at fixed extraction time of 60 min. From the results, it was observed that at a constant temperature, an increase in pressure to about 4750 psi led to an increase in percentage of oil yield and the slope of the graph decreased afterward. Increasing pressure up to 4750 psi could increase the density of solvent (fluid). As a result, the distance between the molecules was reduced and causing stronger interactions between the solvent and solute (oil) matrix. This phenomenon caused an enhancement in SC-CO_2 solvating power, thus, increasing the solubility of solute within the SC-CO_2 fluid and therefore increased oil yield (Ixtaina *et al*., 2010; Lang & Wai, 2001; Sodeifian *et al*., 2016a). On the other hand, beyond 4750 psi, the oil yield was then decreased. This might be due to the enhancement of repulsion between solute and solvent causing from the high compression of CO_2. As described by Uquiche *et al*. (2015), this phenomenon happens when the system is dominated by repulsive part of the solute-solvent rather than solute-solvent interactions hence, causing both the solute and solvent to repel each other. As a result, the average density of solvent around a solute is less than the bulk density of solvent. Therefore, due to described opposing effect of pressure, commonly, there would be little impact on the oil yield at very high pressures (Porto *et al*., 2012; Liu *et al*., 2009; Sodeifian *et al*., 2016c).

When temperature rises in the range of 35–47ºC, it was observed that the oil yield increases with a mild slope and the oil yield decreases as the temperature was further increased up to 65ºC. Maran & Priya (2015) mentioned that these two different effects on oil yield are due to counter effect of solute vapour pressure (volatility) and the density of supercritical fluid. In other word, increasing temperature plays a dual role (Sodeifian *et al.*, 2016c). Firstly, increasing temperature up to 47ºC increases the oil vapour pressure (volatility) and enhancing its solubility in the SC-CO_2 (Brunner, 1994; Uquiche *et al*., 2015). In this region, the increase of the oil vapour pressure prevailed over the decrease in SC-CO_2 density, therefore, led to enhance the oil yield. However, above 47ºC, the effect of oil vapour pressure and CO_2 density becomes equivalent, and consequently beyond that, the decrease of SC-CO_2 density prevailed over vapour pressure enhancement (Maran & Priya, 2015). Here is where the second role of increasing temperature is in action. The density of SC-CO_2 is decreased due to an increment of gas volume and resulting in a decreased solvent power, hence led to lower oil yield.

Optimum operating conditions and validation of optimised condition

Within the experimental ranges studied, optimal conditions for oil extraction were predicted by optimization function of the Design Expert Software to be: temperature = 47ºC and pressure = 4750 psi which resulted in predicted oil yield of 6.93% (by applying Eq. 3) after 60 min of extraction time. At optimal condition, the experiments were carried out

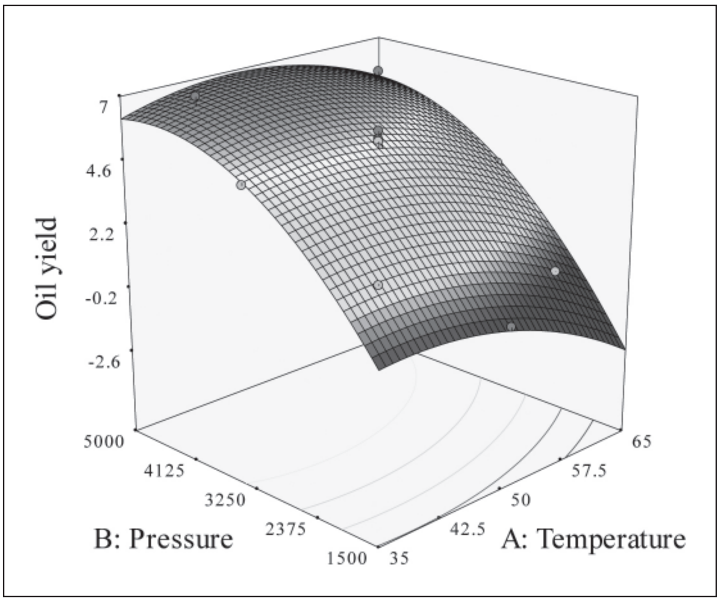

Fig. 1. Three-dimensional (3D) surface plot showing the effect of temperature and pressure on the oil yield.

in triplicate and the experimental data were then compared with the predicted result. Experimentally, the percentage yield of oil obtained was 6.88 ± 0.06% and in a good agreement with the predicted value. Well agreed experimental and predicted results, thus confirming that mathematical model derived from RSM can be used to adequately describe the relationship between the independent variables and response of the oil yield.

Fatty acid composition and physicochemical properties

Fatty acid (FA) compositions of oil extracted from red pitaya seeds by *n*-hexane and SC-CO_2 at optimised conditions are summarized in Table 4. Both extraction methods produced oils that were yellow in color, but the yellow color of SC-CO_2 extracted oil was much lighter. It was disclosed from results that there was no significant difference between the composition of FA in oil obtained by *n*-hexane and SC-CO_2 extraction. Similar findings have been reported by Sodeifian *et al.* (2016a) for the extraction of oil from *Pistacia khinduk*, Maran and Priya (2015) for the extraction of muskmelon seed oil and Rai *et al.* (2016) for the extraction of sunflower seed oil. Since CO_2 and *n*-hexane were both of non-polar solvents, therefore, they were expected to extract the chemical compounds from the solid plant materials in similar way. For both oils, it was found that the composition of unsaturated fatty acids (UFA) including linoleic (C18:2), oleic (C18:1) and linolenic acids (C18:3) was more than that of saturated fatty acids (SFA) consisting palmitic (16:0) and stearic acids (18:0). The major component was linoleic acid with concentration averagely at 49.76% which is considered as very high as those discovered by Ariffin *et al*. (2009), Chemah *et al*. (2010) and in correspondence when compared with white pitaya (*Hylocereus undutus*) seeds reported by (Rui *et al*., 2009). The second major component was oleic acid, while the major component of SFA was palmitic acid with average content of 16.42%.

Table 5 shows the physicochemical properties of red pitaya seed oil extracted by *n*-hexane and SC-CO_2 at optimised conditions. Based on result, there were no significant differences in specific gravity and refractive index values for both extracted oil. Relatively high iodine values (105.1 and 113.6 g of I_2/100 g of oil) were obtained designating high percentages of UFA in the oils. Saponification value measures the molecular weight (chain length) of FA in the oil. The saponification values of 133.4 and 128.4 mg of KOH/g of oil with acid values 3.09 and 2.35 of NaOH/g of oil were recorded for oil extracted by SC-CO_2 and *n*-hexane, respectively. The acid values were found within desirable limits for edible oil (Sodeifian *et al*., 2016a).

Table 4. Fatty acid composition of red pitaya seed oil extracted by SC-CO_2 and *n*-hexane at optimised conditions

Fatty acids (wt%)	SC-CO_2	*n*-Hexane
Palmitic acid (C16:0)	15.71 ± 0.79	17.13 ± 1.07
Stearic acid (C18:0)	4.28 ± 0.22	7.61 ± 0.25
Oleic acid (C18:1)	26.86 ± 0.86	23.95 ± 1.00
Linoleic acid (C18:2)	50.94 ± 1.30	48.57 ± 1.21
Linolenic acid (C18: 3)	1.07 ± 0.11	1.05 ± 0.14
Other	1.14 ± 0.20	1.69 ± 0.42
ΣSFA	19.99 ± 1.01	24.74 ± 1.32
ΣUFA	78.87 ± 2.27	73.57 ± 2.35

Values are means ± standard error (n = 3).
SFA: saturated fatty acid; UFA: unsaturated fatty acid.

Table 5. Physicochemical properties of red pitaya seed oil extracted by SC-CO_2 and *n*-hexane

Properties	SC-CO_2	*n*-Hexane
Specific gravity (kg/m^3) (25°C)	0.857 ± 0.0010	0.858 ± 0.0010
Refractive index (25°C)	1.4675 ± 0.0005	1.4673 ± 0.0003
Iodine value (g of I_2/100 g of oil)	105.1 ± 2.5	113.6 ± 3.2
Saponification value (mg of KOH/g of oil)	133.4 ± 2.1	128.4 ± 1.0
Acid value (mg of NaOH/g of oil)	3.09 ± 0.10	2.35 ± 0.10

Values are means ± standard error (n = 3).

CONCLUSION

In this study, the effect of temperature and pressure on the SC-CO_2 extraction of oil from red pitaya (*H. polyrhizus*) seeds was investigated using CCD-RSM. The optimal conditions for extraction with dynamic extraction time of 60 min were 4750 psi and 47ºC which gave maximum oil yield of 6.93%. The predicted value of extraction yield obtained from the model adequately in agreement with the experimental yield obtained under investigated conditions. GC-MS analysis showed both oil extracted by SC-CO_2 and *n*-hexane had negligible difference in fatty acid composition. The extracted oil was mainly composed of unsaturated fatty acids mainly linoleic acid and demonstrating that the red pitaya (*H. polyrhizus*) seed oil can be a promising candidate for applications in the food and cosmetic industries.

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