Yield Optimization and Supercritical CO_2 Extraction of Essential Oil from Jasmine Flower

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Abstract: *Supercritical fluid extraction (SFE) is an innovation that permits extraction of an extensive variety of different chemical composition from the plant grids. Extraction of essential oil from Jasmine flower was tentatively carried out using the supercritical CO_2 technique. The effect of extraction parameters which include pressure (100–300 bar) and temperature (300–350 K) on the oil recovery was explored. The extraction process was optimized using the response surface methodology (RSM). At the SFE optimal conditions, the chemical compositions of the extracted oil were examined using gas chromatography-mass spectrometry (GC-MS) analysis. The obtained result reflected that the optimal yield of oil from Jasmine flower was 12.18% mg oil extracted/100 g dry flower, which was achieved through an SFE optimal conditions of pressure at 200 bar and extraction temperature at 325 K. A total number of six chemical compounds were tentatively identified in the Jasmine flower extracted oil at the optimal SFE conditions.*

Keywords: *jasmine flower; response surface methodology (RSM); essential oil; supercritical fluid; gas chromatography-mass spectrometry (GC-MS)*

INTRODUCTION

The aromatic mixing of fluids through the refining of plant materials is known as a basic oil [1]. Industrially, fundamental oils are categorized as the unpredictable oils with fragrant, these oils are made up of substance mixes, such as phenols, esters, alcohols, ketones, aldehydes, and hydrocarbons [2]. Fundamental oils of various plants are accessible in the market [3]. Several studies had discussed the viability of fundamental oils in terms of biological activities even at lower measurements [4-5].

Jasmine is an evergreen, shrubby vine with glossy, extremely ornamental with pointed clears out that can grow up to 3 m high. The flower is white in color with a strong perfume that shows up in bunches consisting a little measure of basil oil. The fundamental oil of Jasmine is called "Otto" or "attar of jasmine". Jasmine trees are mostly grown in tropical nations, including Southeast Asia, Australia and Africa. In the present time, this plant can be found everywhere around the world. Jasmine flowers are mostly found in the gardens, houses for elaborate purposes as well as slice blossoms to make laurels. Jasmine possessed larger economic values because it can be explored as the florist restorative, vendor, scene, and pharmaceutical [6]. The oil from Jasmine flower comprises quintessence smell which is being used as a stimulant, expectorant, germicide, and antispasmodic [7]. It possesses different kinds of hydrocarbons and glycerides. The oil contains chemical compounds such as *cis*-jasmone, farnesol, linalool, benzyl liquor, eugenol, benzyl benzoate, benzyl acetic acid derivation, geraniol, isohytol, and phytol [8]. Moreover, the oil from this flower is utilized to cure a cough, despondency, migraine, delicate skin, and fatigue [9]. These are several species of Jasmine, out of several varieties, *Jasminum sambac*, *Jasminum auriculatum*, and *Jasminum grandiflorum* are used industrially for essential oil production [6]. In this study, Jasmine grown in Malaysia was explored.

In the extraction of Jasmine oil, different extraction methods had been employed which include hydrodistillation, solvent extraction, and maceration. Nevertheless, the methods had been reported to suffer some drawbacks which include lower yield of oil [10-11]. However, to an outcome this drawback, supercritical fluid extraction technique was employed for recovering oil from Jasmine flower.

Supercritical fluid extraction (SFE) is a modern technique used in the extraction of different plant samples. It operates based on the influence of temperature and pressure on the supercritical liquid state. This is vital to medicinal fields and nourishment. Mostly, supercritical fluid extraction utilized CO_2 because of its physical properties of moderately lower pressure and temperature conditions (304 K and 7.38 MPa, respectively) [12]. Likewise, the yield from this technique had been reported to be higher compared to the hydrodistillation and steam methods. Nevertheless, the main demerit of SFE is the hardware cost which restricts its utilization for profoundly mechanical parameters [13]. The SFE had shown higher efficiency in separating basic oils from apricot [14], myrtle [15], palm [16], juniperus [17], soybeans [18], rosemary [19], sunflower [20], jojoba [21], sesame [22], celery [23], parsley [24], almond, [25], and pistachio [26].

Thus, this study investigated the extraction of oil from Jasmine flower using supercritical carbon dioxide (CO_2) extraction technique. The effects of SFE parameters which include pressure and temperature on the yield were as well studied using response surface methodology (RSM). In addition, the chemical compositions of the extracted oil at the optimal conditions were examined using gas chromatography-mass spectrometry (GC-MS).

■ EXPERIMENTAL SECTION

Materials

The Jasmine blooms were obtained from (Nature Flower Enterprise), Pahang, Malaysia. 100 g from flowers were washed using water to evacuate the contaminations. The blooms were dried in an oven at 90 °C for 1 h. The dried blooms were then crushed to expand the contact surface area.

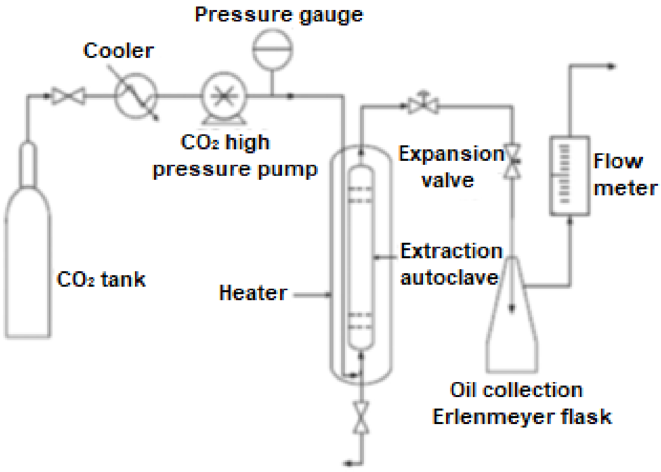

Fig 1. Supercritical CO_2 extraction

Instrumentation

Instrumentation were used are Germany Retsch Ultra Centrifugal Mill ZM 200 grinder machine, hot air drying oven (Thermo Seientific Heracus), supercritical fluid extraction (SFE) by Separex (champigneulles, France), Experimental Design, Gas Chromatography-Mass Spectroscopy analysis (GC-MS) (Agilent 5975C inert, USA).

Procedure

Supercritical CO_2 extraction

In the supercritical fluid extraction (SFE), extraction was carried out in a Separex 4219 extraction unit (Separex, France) as illustrated in Fig. 1. This device works with three autoclave breaking points (Axes) of 5, 10 and 20 cm^3. In this study, an autoclave breaking point of 5 cm^3 was used. A known mass of a homogeneous mix of Jasmine bloom powder was poured into the reactor, the pressure and temperature were set, and the autoclave was stacked. After the extraction, the reactor was cooled down before the oil was removed.

Experimental design

In order to optimize the yield (Y) of the supercritical CO_2 extraction of essential oil from Jasmine flower, the oil yield and its properties are functionally related to two factors: pressure and temperature. A 3^2 central composite design from RSM was used to determine the optimal factors of SFE in extracting oil

Table 1. Factors and standards examined for the exploratory outline (RSM)

Symbol	Independent variables	Coded levels		
		Low (-1)	Middle (0)	High (+1)
A	Pressure (bar)	100	200	300
B	Temperature (K)	300	325	350

from jasmine flower. The two independent variables namely pressure (100–300 bar, A) and temperature (300–350 K, B) as presented in Table 1 were being studied for their influence on the extraction yield. The Design Expert 7.1.6 was used for the experimental matrix (Table 1), ANOVA analysis, regression coefficient calculation and projection of the experimental data in graphical mode (response surface and contour plot). The experimental value and predicted values were compared to verify the adequacy of the developed model. Equation (1) which shows a second-order response surface model was used to determine the experimental yields.

$$Y = a_0 + \sum_{i=1}^{2} a_i A_i + \sum_{j=1}^{2} a_j B_j + \sum_{i=1}^{2}\sum_{j=1}^{2} a_{ij} A_i B_j + \sum_{i=1}^{2} a_{ii} A^2{}_i + \sum_{j=1}^{2} a_{jj} B^2{}_j + \sum_{i=1}^{2}\sum_{j=1}^{2} a_{ij} A^2{}_i B_j + \sum_{i=1}^{2}\sum_{j=1}^{2} a_{ij} A_i B^2{}_j \qquad (1)$$

where Y refers to the response for variables, a_0, a_i, a_{ii}, a_{ij}, and a_{jj} are consistent coefficients of the object, direct, quadratic and intelligent terms, respectively. A_i and B_j are the autonomous factors of pressure and temperature, respectively.

GC-MS analysis

The Agilent 5975C Series GC/MS used to examine the chemical composition of the oil at optimal SFE condition is made up of 100% dimethylpolysiloxane and DB-WAX (30 m × 0.25 mm ID × 2.5 µm). For 10 min, the working set-up temperature was set at 60 °C and expanded at a rate of 20 °C/min to accomplish 250 °C, held for 10 min. The helium gas speed of 30 cm/sec was utilized. The oils obtained at the two conditions (200 bar, 325 K) and (200 bar, 300 K) were analysed. The compositions were distinguished compared the mass spectral data obtained from the oil with that of pure commercially available standards injected under similar conditions. National Institute Standard and Technology (NIST) library was used to identify the chemical compounds.

Table 2. The yield of Jasmine oil (mg oil extracted/100 g dry flower)

Run	Pressure (bar)	Temperature (K)	Observed yield (%)
1	100	300	8.1
2	100	350	3.8
3	100	325	8.8
4	200	325	12.18
5	200	300	11.32
6	200	350	9.1
7	300	300	9.8
8	300	325	7.9
9	300	350	6.7

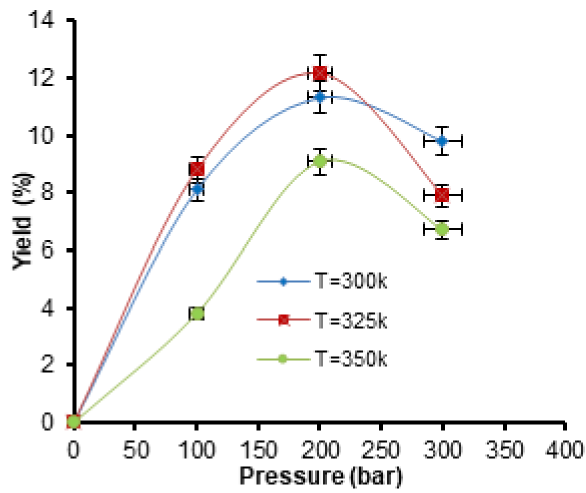

Fig 2. The yield for oil at various pressures and temperatures

■ RESULTS AND DISCUSSION

Supercritical CO_2 Extraction

The supercritical CO_2 extraction yield as influenced by pressure and temperature was studied. The obtained yields for the experimental runs are shown in Table 2. The oil yield was affected by both the pressure and temperature. The pressure significantly affected the oil yield by increasing from 100 to 200 bar. The highest oil yield was obtained at 200 bar. However, the oil yield

Table 3. Major compositions in the Jasmine flower oil extracted

RT	Compounds	Composition at (200 bar, 300 K) (%)	Composition at (200 bar, 325 K) (%)	Composition at (300 bar, 350 K) (%)
6.253	Acetaldehyde	9.52	12.70	10.74
33.029	2-Phenylthiolane	55.32	57.31	53.43
40.656	Propanamide	8.87	6.79	4.68
45.587	Cyclohexene, 3-ethenyl	22.61	25.91	27.29
50.118	*N*-Methylallylamine	11.54	9.99	10.11
60.371	Phthalic acid, bis(7-methyloctyl) ester	4.69	5.21	7.55

RT: Retention Time

declined beyond 200 bar. An expansion in the pressure builds the dissolvable energy of the supercritical liquid because of its thickness increment that prompt higher extraction [27]. The decline in the yield above 200 bar might be because at times a pressure increment may lessen the diffusivity and cause liquid particles to diffuse into the strong pores by breaking up the solute. Additionally, a pressure increment may initiate a decline in the strong grid free space and influence the extraction yield. In the case of temperature variation, the yield increases as the temperature increases from 300 to 325 K, beyond 325 K, a net reduction in the oil yield was observed [28]. However, at 100 bar, the oil yield was nearly temperature free. In this case, the multifaceted nature of temperature impact on the oil yield since an expansion in temperature may prompts two consequences for the dissolvability of solids in supercritical liquids which include the expansion of strong unpredictability and the lessening of the dissolvable thickness [28]. It should be noticed that the oil dissolvability is controlled by a balance between the solute vapor pressure and the dissolvable thickness [29].

Characterization of the Extracted Jasmine Flower Oil

Table 3 show the identified chemical compounds which include acetaldehyde, 2-phenylthiolane, propanamide, cyclohexene, 3-ethenyl, N-methylallylamine, and phthalic corrosive, and bis (7-methyloctyl) ester. This clarified the extent of oil and mass at a pressure of 200 bar achieved 40%. This might be due to the way that the oil yield had been influenced by different variables such as atmospheric conditions, height, precipitation, and so on. Additionally, this study demonstrated that the Jasmine blossom oil portrayed a high aliphatic aldehyde and vegetal hydrocarbon.

Analysis using RSM

The results showed that the oil yield (Jasmine blossom oil) varied between 3.8 and 12.18%, this affirmed the effect of pressure and temperature on the oil yield and affirms their significance. By applying the analysis of variance, a second-degree was obtained as shown in Equation (2):

$$Y = +12.09 + 1.16 * A - 1.52 * B + 0.35 * A * B - 3.52 * A^2 - 1.01 * B^2 - 0.238 * A^2 * B + 0.034 * A * B^2 \quad (2)$$

The multiple correlation coefficient R was 0.9986 indicating a quite satisfactory adjustment of the model. The design matrix is shown in Table 4 consisting of nine tests with their observed and predicted responses. Most of the representing points are close enough to the first bisector line, confirming the agreement between the observed and predicted data. Henceforth, the model of unwavering quality can be evaluated using analysis of variance (ANOVA) in terms of Fisher (F-test). This is performed by the Fisher variety proportion (F-value), which is the proportion of the mean square of the model and the residual error. F-value of 503.68 indicates that the model is significant. The estimation of "Prob > F or p-value" for the model was less than 0.0500, demonstrated that the model was noteworthy as shown in Table 4. In a comparable way, the primary impact of pressure (A), principle impact of temperature (B), the two-level associations of pressure and temperature (AB), the

Table 4. Predicted and observed values for oil yield of Jasmine flower (mg oil extracted/g of dry flower)

Run	Pressure (bar)	Temperature (K)	Observed yield (%)	Predicted yield (%)
1	100	300	8.1	8.59
2	100	350	3.8	4.4
3	100	325	8.8	9.21
4	200	325	12.18	12.39
5	200	300	11.32	12.1
6	200	350	9.1	7.8
7	300	300	9.8	10.28
8	300	325	7.9	6.56
9	300	350	6.7	7.47

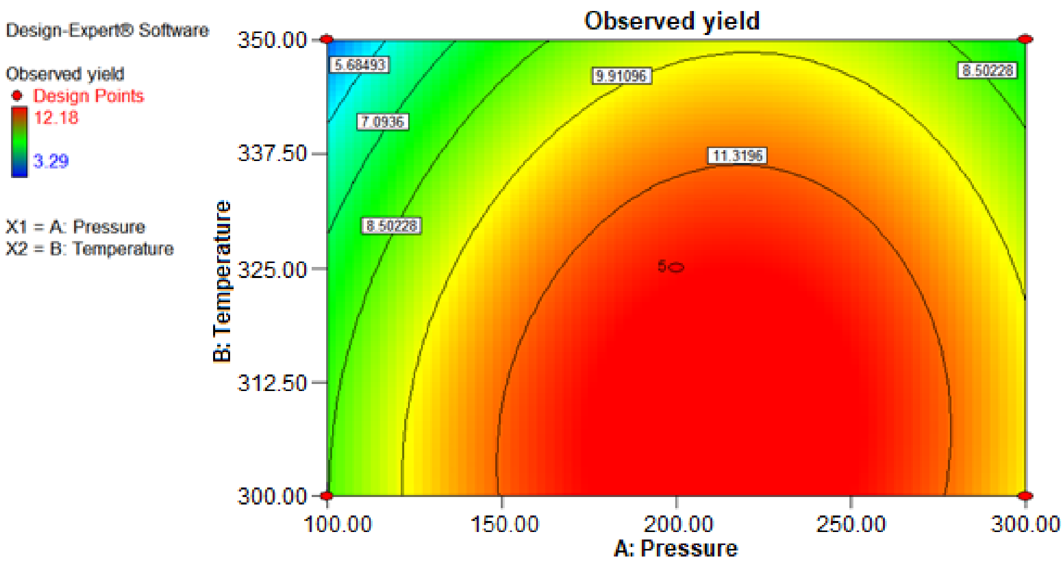

Fig 3. Optimum yield for oil extraction

second-arrange impact of the pressure (A^2), and the second request impact of Temperature (B^2) were significant. Other model terms whose esteems were more noteworthy than 0.1000 show the model terms are not significant. Among the noteworthy model expression which was A, B, AB, A^2, and B^2, the primary impact of Pressure (A) was the most significant factor to the oil yield of Jasmine bloom since pressure has polar nature which could interface inexhaustibly with temperature. This finding is in concurrence with [30]. The noteworthy elements were being positioned to the estimated F-value. Consequently, in this study, the ordering of the significances of the elements are as per the following: $A^2 > B > B^2 > A > AB > A^2B > AB^2$. Moreover, the lack of fit in not significant with a p-value of 3.49%. The R^2 value is utilized to choose whether a relapse is proper. In this study, the R^2-value being ascertained was 0.9986, sensibly near 1, which is satisfactory. The anticipated R^2 of 0.9349 was insensible concurrence with the adjusted R^2 of 0.9966. A proportion more noteworthy than 4 is desirable. For this situation, the esteem was well over 4 where the proportion of 60.510 showed a satisfactory indicative.

Optimum Conditions

The main purpose of using response surface methodology is to estimate the effect of extraction parameters of oil yield of Jasmine bloom. The optimal conditions to obtain the highest oil yield of 12.18% was a pressure (A) of 200 bar and a temperature (B) of 325 K.

Fig. 3 showed the three-dimensional response surface obtained involving the interaction between pressure and temperature in achieving optimal oil yield. For the three considered pressures (100, 200 and 300 bar), the oil yield expanded before diminishing with temperature. This showed that pressure and temperature influence the Jasmine flower oil yield. At the optimum conditions of 200 bar and 325 K resulted in 12.18% mg extracted oil/g dry sample.

CONCLUSION

The supercritical CO_2 extraction of oil from Jasmine bloom had been studied. The two SFE parameters, namely pressure and temperature were optimized using RSM. The pressure had a close impact on the extracted oil yield while temperature moderately affected the oil. The optimum oil yield of 12.18% mg oil extracted/100 g dry flower was acquired using 325 K and 200 bar of temperature and pressure, respectively. A total number of six chemical compounds were tentatively identified in the Jasmine flower extracted oil at the optimal SFE conditions. Thus, Jasmine bloom is endowed with the appreciable quantity of oil using SFE.

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