SUPERCRITICAL CO_2 EXTRACTION OF ESSENTIAL OIL FROM JASMINE FLOWER AND YIELD OPTIMIZATION

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Abstract

Supercritical liquid extraction (SFE) is an innovation that permits extraction of an extensive variety of different composition from an assortment of plant grids. Extraction of essential oil from Jasmine flower was done by used Supercritical CO_2 tentatively and the impact of working parameters, for example, pressure and temperature on the separating procedure were explored, thinking about three distinct esteems for every parameter (100 and 300 bar and 300 and 350 Kelvin for weight and temperature, individually). The extraction procedure was upgraded by methods for a trial configuration utilizing the measurable programming, Design Expert 7.1.6. A moment arrange Multiple was utilized to obvious the oil recuperation yield utilizing the reaction surface approach RSM and the acquired outcomes were near the test esteems with a relationship factor of 12.18, showing again the unwavering quality of this procedure. GC-MS examinations were done on tests got at the ideal conditions (200 bar and 325 K). The typical likelihood plan of the residuals and the plot of the residue against the anticipated reaction to Jasmine bloom oil yield was being. The examinations of the acquired reaction surface demonstrated that pressure had the most critical impact on the oil extraction yield, through the quadratic and straight terms. The temperature moderately affected the oil yield rather through its square term, which demonstrated a low importance oppositely to the straight and cooperation terms.

Keywords: Jasmine flower; Response Surface Methodology (RSM); Essential oil; Supercritical fluid; Gas Chromatography-Mass Spectrometry (GC-MS)

1. Introduction

The aromatic mixing of fluids got by means of refining of plant materials, is known as a basic oil (Burt, 2004). The fundamental oils are viewed in the industrial facility as unpredictable oils with fragrant parts that are comprised of substance mixes, for example, phenols, esters, alcohols, ketones, aldehydes, and hydrocarbons. These segments are the significant segments of fundamental oil (Younis, Riaz, Khan, Khan, & Pervez, 2006). Fundamental oils of various plants are accessible industrially in the market (Formaceck, 1982). Various investigations have shown the viability of fundamental oil in biological activity even at low measurements (Oussalah, Caillet, Saucier, & Lacroix, 2006, 2007).

Jasmine tree was showed in tropical nations, for example, Southeast Asia, Australia, and Africa. At present, everywhere throughout the world Jasmine plants are developed. In the present examination, basic oils extricated from Jasmine in Malaysia, for example, Melur and Melati were explored. The oil of Jasmine displays the quintessence of flavor and smell of Jasmine blossom. Its properties are subject to the kinds of hydrocarbon and glycerides. Fundamental oils of Jasmine have been broadly utilized as a stimulant, an expectorant (for skin), antispasmodic and germicide. Likewise, these basic oils are utilized to cure a cough, despondency, migraine, delicate skin, and fatigue.

The sort Jasminum is accounted for to incorporate around 64 species, in Indian subcontinent out of which 40 (Irulappan, 1994). Jasmine is of great economic value as a field crop for the florist restorative, vendor, scene, medicinal and pharmaceutical (Green & Miller, 2009). For the most part, jasmine flower is developed in houses and gardens for elaborate purposes and are some of the time likewise utilized for slice blossoms to make laurels. Among these species, J. sambac, J. auriculatum, and J. grandiflorum are industrially developed for oil extraction (Green & Miller, 2009). Jasminum is growing up to 3 m high and is an evergreen, shrubby vine with glossy, extremely ornamental and pointed clears out. Its white, strongly

perfumed blossoms show up in light bunches that contain a little measure of basic oil. The fundamental oil of jasmine is called "Otto" or "attar of jasmine"; it contains more than 100 types however the principle concoction parts in charge of the fragrance are cis-jasmone, Farnesol, Linalool, benzyl liquor, eugenol, benzyl benzoate, benzyl acetic acid derivation, Geraniol, and follow measures of isohytol and phytol (Lawless, 1995).

For the extraction of Jasmine oil composition, different methods from its characteristic source do exist and like hydrodistillation, solvent extraction, maceration, and so on. In any case, these strategies have some reasonable hindrances like the trouble to recuperate all the natural dissolvable from the last item (Ernesto Reverchon, 1997; Zizovic, Stamenić, Orlović, & Skala, 2007) for the instance of the dissolvable extraction by used high temperatures included which may led fractional hydrolysis and warm debasement of a few constituents for the instance of hydrodistillation and steam (Lawrence & Shu, 1993; E Reverchon & Senatore, 1992). These procedures additionally require promote unit tasks, for example, decantation, centrifugation, and others (Meireles, 2003), henceforth inciting further expenses.

Supercritical liquid extraction can be viewed as the other option to the dissolvable extraction of different mixes from common strong networks with no effect of dissolvable. It achievement the influence of temperature and pressure on the supercritical liquid state and properties, especially its thickness to improve its dissolvability This is especially vital for pharmaceutical fields and nourishment.

As of most cases in a supercritical liquid utilized Co_2 is because of its physical properties and substance to its moderately low basic pressure and temperature esteems (304 K and 7.38 MPa, individually) (Carvalho Jr, Moura, Rosa, & Meireles, 2005). Likewise extricates got by methods for this strategy are of a greatly quality than those got by dissolvable extraction or by hydrodistillation and steam or use another method. In any case one of the real downsides of this system is the hardware cost which restricts its utilization for profoundly touchy

mechanical parameters were high and also characteristics and purities for some elements (Meireles, 2003). This procedure has additionally indicated superior exhibitions in separating basic oils from an awesome assortment of sources as announced in the writing and one can refer to apricot (Özkal, Yener, & Bayındırlı, 2005), Myrtle (A Zermane, Larkeche, Meniai, Crampon, & Badens, 2014), Palm (Md Zaidul, Nik Norulaini, & Mohd Omar, 2006), Juniperus (Larkeche, Zermane, Meniai, Crampon, & Badens, 2015), Soybeans (E Reverchon & Osséo, 1994), Rosemary (Ahmed Zermane, Meniai, & Barth, 2010), Sunflower (Calvo, Cocero, & Diez, 1994), Jojoba (Salgın, 2007), Sesame (Xu, Chen, & Hu, 2005), Celery (Papamichail, Louli, & Magoulas, 2000), Parsley (Louli, Folas, Voutsas, & Magoulas, 2004), Almond, (Marrone, Poletto, Reverchon, & Stassi, 1998), Pistachio (Palazoglu TK, 1998), and so on.

This work present interest to the extraction of Jasmine blossom oil utilizing supercritical carbon dioxide (CO_2). The got test comes about were utilized to examine and advance the impact of key working factors pressure and temperature on the yield from extraction through utilizing the response surface procedure (RSM). The extracted oil at ideal extraction conditions was studied via (GC-MS).

2. Materials and methods

2.1. Raw Materials

The Jasmine blooms were obtained from (Kingdom of Malaysia, Pahang). The flowers were washed by water keeping in mind the end goal to evacuate the contaminations. The blooms were dried in a stove working at 90 ^{o}C for 1h. The dried blooms were then crushed keeping in mind the end goal to expand the contact territory between the solvent and the blossom (powdered shape).

2.2. Supercritical CO_2 extraction

For supercritical fluid extraction (SFE), extraction was finished in the extraction unit (Separex 4219) gave by Separex (France, champigneulles) and showed up in Figure 1. This device offers the probability to work with three autoclave breaking points (Axes) of 5, 10 and 20 cm^3. In this work according to the progress and stream rate, the pressure, the temperature was used at an enduring in 5 cm^3:

a. By using a known mass of a homogeneous mix of Jasmine bloom powder and warmed up to the coveted temperature, the autoclave was stacked;

b. By a cryogenic shower over (- 4 oC) CO2 liquid was already cooled, isolated and after that drew into the extractor until the point that the working weight was come to. CO_2 was pre-warmed to the coveted extraction temperature, before entering the extraction autoclave;

c. The extension valve was opened for SC-CO_2 flow. By a stream meter situated toward the finish of the extraction line, the CO_2 stream was estimated. Also from through utilizing the extension valve the pressure and the stream rate were kept up steady.

d. The autoclave was detached and the autoclave was permitted to chill off to room temperature toward the finish of the extraction time. The autoclave was again pressured to derive the expelled solute mass In the wake of cooling and desorption of CO_2. Did not differ any longer on the systematic adjust, add up to CO_2 desorption was expected when the mass of the autoclave. This was complete after 5 min.

e. To acquire the extraction bend, the above advances were rehashed to acquire the extraction bend;

f. In Hexane, oil extracts were gathered and put away far from light in a cooler at (- 10 ^oC).

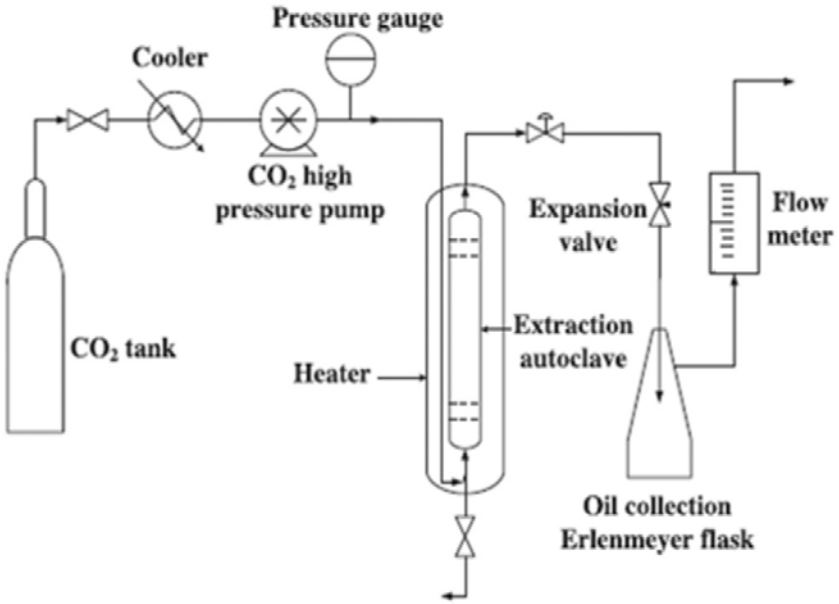

Figure 1 - Supercritical CO2 extraction contraption

2.3. Experimental Design

In order to optimize the yield (Y) of the supercritical CO_2 extraction of essential oil from Jasmine flower, of initial dry matter, expressed as mg of oil jasmine/g, an experimental design was applied for the determination of the corresponding extraction pressure (A) and temperature (B). A full arrangement made out of 3^2 tests was performed, for each considered factor three levels were tried, as appeared in Table 1.

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

By using the following equation (1) the experimental yields were calculated to a second-order response surface model.

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

Where Y refer 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, separately. A_i and B_j are the autonomous factors of Pressure and Temperature.

2.4. GC-SM analysis

The Agilent 5975C Series GC/MS utilized as a part of the present examination utilized a melded silica section pressed with 100% dimethyl poly siloxane and DB-WAX (30 m × 0.25 mm ID × 2.5µm). For 10 min, the working setup temperature was set as 60 ^{o}C and expanded at a rate of 20 ^{o}C/min to accomplish 250 ^{o}C, held for 10 min. The helium gas speed 30 cm/s was utilized. Three examples got at (200 bar, 325 K) and (200 bar, 300 K) were considered for examinations because of the moderately high extraction yields got at these working conditions. The structure comes about are appeared in Table 3. The compositions were distinguished through comparison of mass spectral data obtained from the sample with that taken from pure commercially available standards injected under similar conditions. By utilizing the information of National Institute Standard and Technology (NIST) for data, the general range was translated and the range segments were examined by this library..

2.5. The yield of oil extraction

The of extracted oil in mg over the of initial dry flower was determine through Jasmine flower oil extraction yield as a proportion expressed. It is noticed that the removed oil mass was considered as the loss in the extractor. Various examples of pulverized grains of a mean size under one mm were subjected to extraction with Sc-CO_2 observance a steady stream rate of 0.252 kg h^{-1} after the of the cluster loss of the autoclave at each 20 min, till the adjustment of the mass of autoclave for 2 h.

3. Results and Discussion

3.1. Supercritical CO_2 extraction

The supercritical CO2 extraction yield was learned at the working conditions, the impact of used pressure and temperature. The acquired yield esteems from the different test runs as see Table 2, communicated by weight (mg) of extract oil/ weight (g) of dry blossom x 100. Figure 2 demonstrates unmistakably that the yield changed altogether with pressure and temperature. A pressure, significantly affected the Jasmine extraction yield increment from 100 to 300 bar demonstrating an expansion in pressures to 200 bar and after that a lessening of pressures to 300 bar. For the most part, an expansion in the pressure builds the dissolvable energy of the supercritical liquid because of its thickness increment, prompting higher extraction. The decline of the yield past 200 and up to 300 bar might be clarified by the way that at times a pressure increment may likewise lessen the diffusivity and henceforth the liquid particles will discover challenges to diffuse into the strong pores to break up the solute. Additionally, a pressure increment may initiate a diminishment of the strong grid free space and henceforth the extraction yield. For the impact of temperature variations, a surprising pattern was watched where an expansion of temperature from 300 to 325 K prompted higher yield esteems, oppositely to an expansion from 325 to 350 K which demonstrated a net reduction in

the extraction yield. However, at 100 bar, the extraction yield was nearly temperature free. In this way, one can see the multifaceted nature of the impact of temperature on the extraction yield since an expansion in temperature may prompt two contending consequences for the dissolvability of solids in supercritical liquids: the expansion of strong unpredictability and the lessening of the dissolvable thickness (Pereda, Bottini, & Brignole, 2008).

pressure and temperature as a general rule about the impacts of the cooperations of these two parameters, it ought to be noticed that the oil dissolvability is controlled by a balance between the solute vapor pressure and the dissolvable thickness, clear up the retroactive area as revealed in Ref. (Roy, Goto, & Hirose, 1996). There were a few reparations between the lessening in the SC-CO_2 thickness as the pressure diminished and the expansion in vapor pressure of the compositions as the temperature expanded.

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

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

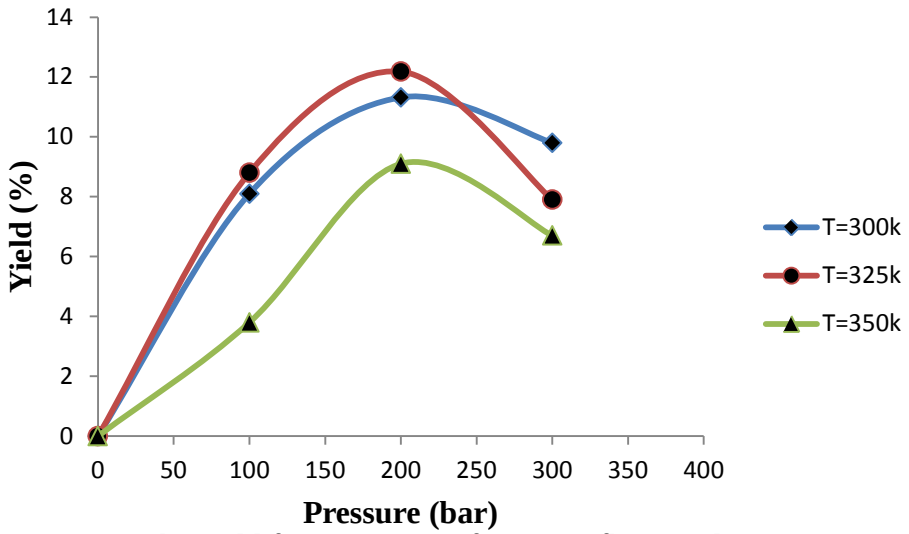

Figure 2 - The yield for extraction of Jasmine flower oil at various pressures and temperatures.

3.2. Characterization of the Jasmine flower oil extracted

The screening of the indistinguishable chromatograms of Figure 3 additionally as the related synthesis tables, coming about because of such examinations, indicated real compounds in particular Acetaldehyde, 2-Phenylthiolane, Propanamide, Cyclohexene, 3-ethenyl, N-Methylallylamine and Phthalic corrosive, bis(7-methyloctyl) ester see Table 3 where to demonstrate the extents significant segments by utilized distinctive parameters to removed oil from Jasmine bloom. This clarified their awesome extent of the oil and mass at a pressure of 200 bar achieved 40%. This can be clarified by the way that the oil piece may likewise rely upon different variables like the atmosphere conditions, the height, the precipitation, and so on. Additionally, the investigations demonstrated that the Jasmine blossom oil was portrayed by a high aliphatic aldehyde mix and vegetal hydrocarbon.

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

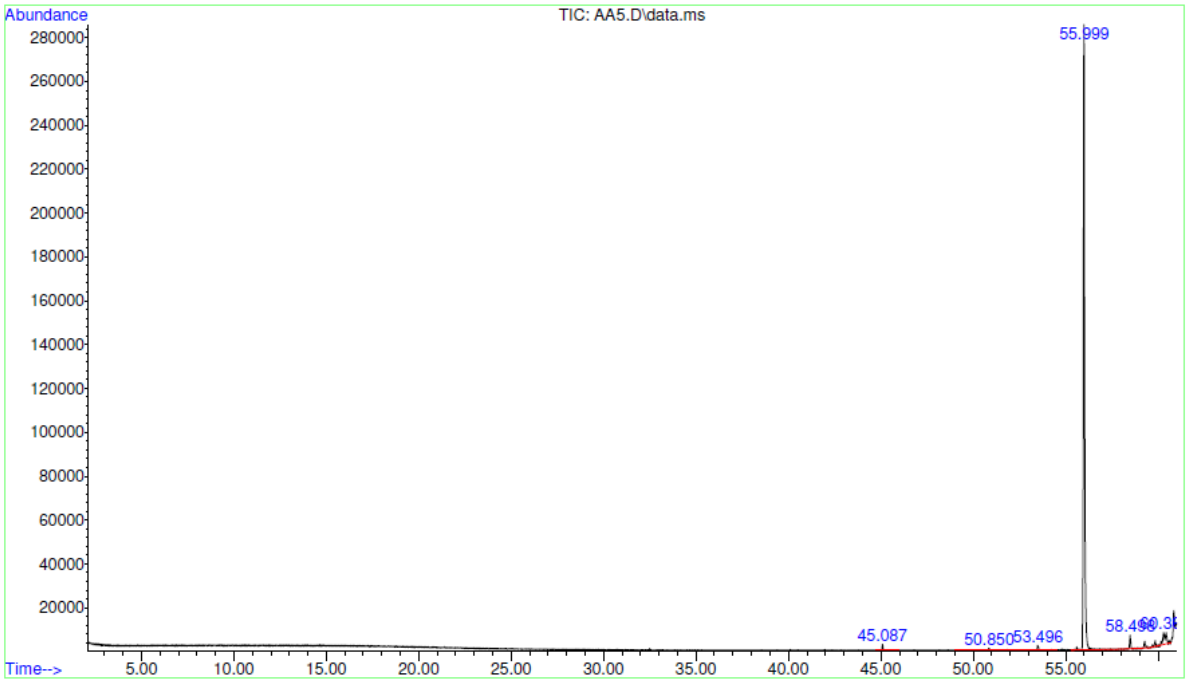

Figure 3 - Chromatogram of Jasmine flower oil by GC-MS at (200 bar, 325 K)

3.3. Analysis by use (RSM)

The outcomes demonstrate that the trial oil extraction yield oil (Jasmine blossom oil) differed in the vicinity of 3.8 and 12.18 %, this affirms the impact of temperature and weight coefficients and affirms their significance as showed ahead of time.

By applying different relapse examination of the exploratory information, a second- degree condition was gotten as takes after 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 various correlation coefficient R was 0.9986 though that of the assurance was, demonstrating a very good change of the model. The planned display is appeared in Table 4

and comprises of nine tests with their watched and anticipated reactions, with Figure 4 demonstrating their correlation. The investigations of various through the analysis of the watched and processed estimations of the reaction which is, for this situation, the oil extraction yield, is appeared in Figure 4 where the greater part of the speaking to indicates are sufficiently close the primary bisector line, affirming the tradition between the watched and the anticipated information.

The outcomes precision, henceforth the model unwavering quality can likewise be finely evaluated by methods for an examination of difference (ANOVA) in light of the trial of Fisher (F-test). This is performed by the Fisher variety proportion (F-value), which is the proportion between the mean square of the model and the residual error.

F-value of 503.68 for the model is significant. A 0.01% shot that an F-value this extensive could happen because of commotion The estimations of "Prob > F or p-value" for the model was under 0.0500 demonstrated that the model was noteworthy show 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 second-arrange impact of the Pressure (A^2) and the second request impact of Temperature (B^2) were a critical model expression. Other model terms whose esteems were more noteworthy than 0.1000 show the model terms are not critical. The consequences of the diminished quadratic model were all the while being shown that the model was as yet huge (see Table 5). Among the noteworthy model expression which were A, B, AB, A^2 and B^2, the primary impact of Pressure (A) was the most critical factor being related with a yield of Jasmine bloom oil since Pressure has polar nature which could interface inexhaustibly with Temperature, hence, to a great extent impact the proficiency of extraction. This finding is in concurrence with (Zhu, Heo, & Row, 2012). The noteworthy elements were being positioned in light of the estimation of F-value.

Consequently, in this examination, the positioning of the significances of the elements are as per the following: $A^2 > B > B^2 > A > AB > A^2B > AB^2$.

Table 4 - Predicted and observed Jasmine flower oil extraction values (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

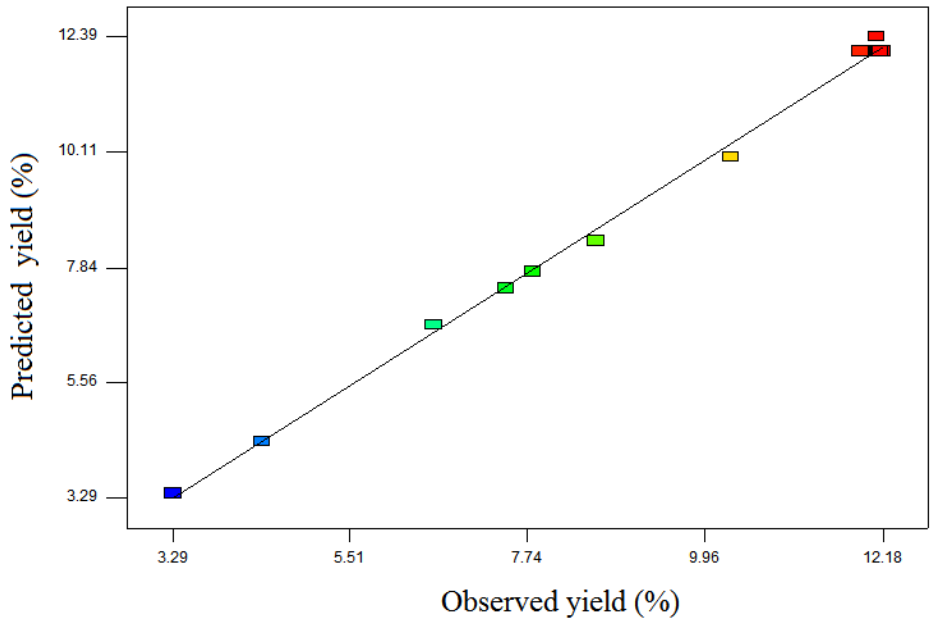

Figure 4 - Compare of observed and predicted yield values.

The "Lack of Fit F-value" of 9.86 infers the Lack of Fit is important (significant). There is just a 3.49% shot that a "Lack of Fit F-value" this vast could happen because of clamor. All in all, R^2 is being utilized to choose whether a relapse show is proper. In the current examination, 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. Satisfactory accuracy thinks about the scope of the anticipated esteems at the plan focuses on the normal expectation blunder. 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.

Table 4 - ANOVA table for a quadratic model for a yield of Jasmine flower Oil

Source	Sum of Squares	dF	Mean Square	F-Value	P-Value (P > F)	
Model	121.45	7	1735	503.68	0.0001	significant
A-Pressure	5.35	1	5.35	155.21	0.0041	
B-Temperature	9.24	1	9.24	268.39	0.0023	
AB	0.48	1	0.48	13.82	0.0137	
A^2	86.32	1	86.32	2505.99	0.0001	
B^2	7.10	1	7.10	206.06	0.0001	
A^2B	0.11	1	0.11	3.06	0.1404	
AB^2	2.29	1	2.29	0.067	0.8066	
Residual	0.17	5	0.034			
Lack of Fit	0.12	1	0.12	9.86	0.0349	Significant
Pure Error	0.050	4	0.012			
Cor Total	121.62	12				
Std. Dev	0.19		R^2	0.9986		
Mean	9.30		Adjusted R^2	0.9966		
C.V %	1.99		Pred R^2	0.9349		
Press	7.92		Adeq Precision	60.510		

3.4. Optimum conditions

The major goal of utilizing Response Surface Methodology was to decide the estimations of the working parameters which prompt the ideal estimation of the oil extricating yield (the reaction). The sample examination and specialized determination in the Design Expert 7.1.6 software(numerical improvement), prompted an ideal oil yield estimation of 12.18% for a pressure (A) of 200 bar and a temperature (B) of 325 K. Can be found for optimal oil yield graphically inside the working factors areas, by methods for the curves appeared as shown in Figure 5.

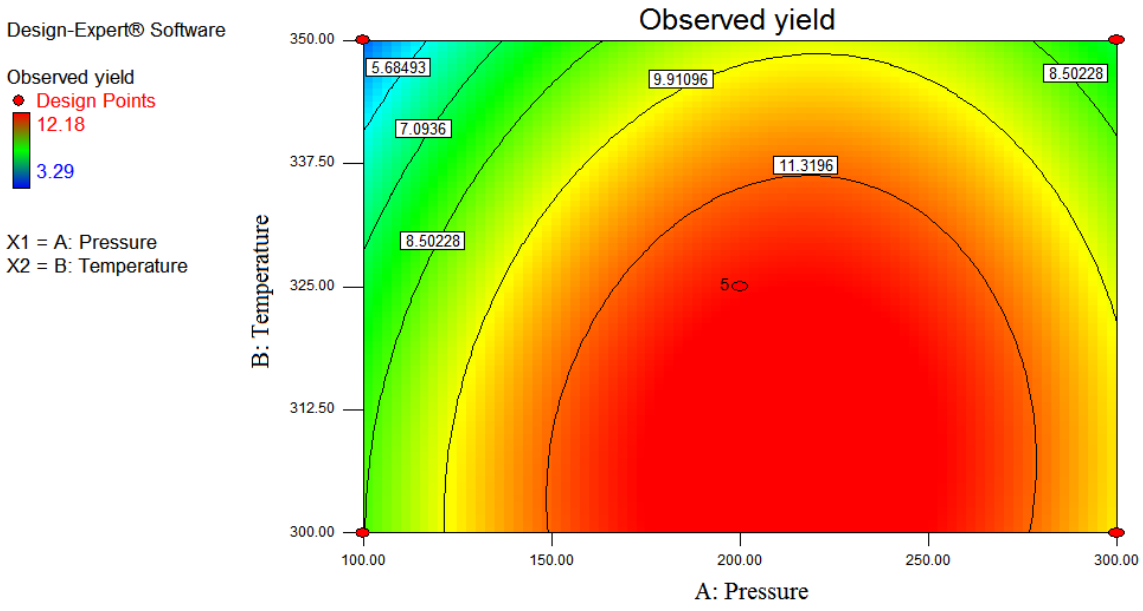

Figure 5 - Optimum yield for oil extraction

3.5. Response surface

It can be seen that for the three considered pressures(100, 200 and 300 bar), the extraction yield expanded before diminishing with temperature, demonstrating a recessive area described by a dissolvability diminish rather than an increase.For this demonstrates the pressure and temperature consequences for the Jasmine blossom oil extraction, after 2 h and a settled supercritical carbon dioxide (CO_2) mass stream rate of 0.252 kg h-1. As can be visible, an ideal extracted estimation of 12.18% was gotten by using 200 bar and 325 K.

4. Conclusions

Through the examination of the acquired reaction surface particular, that pressure had the closely impact on the extracted oil yield, via the quadratic and straight expression and temperature moderately affected the oil separating yield rather by using its quadratic expression, oppositely to the direct and communication expression which demonstrated a low hugeness. This implies supercritical carbon dioxide has been turned out to be compelling in extricating oil from jasmine bloom. The greatest yield was acquired by using 325 K and 200 bar, accomplishing a product of 12.18 %. At last, one can infer that the reaction superficies

system for just from a set number of tests, is a solid strategy which gives helpful data on the impacts of the autonomous parameters.

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