Extraction of Essential Oils from Vietnam's Orange (*Citrus sinensis*) Peels by Hydrodistillation: Modeling and Process Optimization

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In this study, extraction of essential oil from orange peels was investigated by hydrodistillation method and combine technology software to optimize the process. By using the response surface methodology (RSM) based on Box-Behnken surface statistical design, the effect of parameters such as water-to-material ratio (2-4 mL/g), extraction time (45-75 min), and temperature (120-140 ℃) on the extraction of essential oil process from orange (*Citrus sinensis*) peels was examined . All factors were significantly affected to the extraction yield of essential orange peel oil. Optimum conditions for extraction oil yield including the ratio of water to material, temperature, extraction time achieved 3.19 mL/g, 130.08 ℃, 74.31 min, respectively. Below the optimal extraction condition, the maximum yield of orange oil of 3.21 % was obtained. Using gas chromatography/mass spectrometry (GC/MS), the results revealed that the essential orange peels oil is extremely rich in limonene (98.343 %).

Keywords: Orange peel, *Citrus sinensis*, Hydrodistillation, Response surface methodology, Central composite design, GC-MS.

INTRODUCTION

Essentials oils are hydrophobic compounds in oil sacs or oil gland, which contains aroma compounds and provides flavour, unique scent of the plants [1-10]. Essential oils are detected in various parts of the plant including roots, leaves, seeds, flowers and barks. Essential oils are applied for different purposes including flavouring food and drinks, cosmetics, bath products *etc*. In recent years, essential oils has been receiving a great deal of public attention due to the popularity of aromatherapy.

Essential oils are usually extracted from plants materials by different methods such as steam distillation, hydrodistillation, expression, simultaneous distillation extraction and organic solvent extraction [11-18]. Among these methods, hydrodistillation or steam distillation method is usually carried out to extract the essential oil from the medicinal herbs plants,

which is simple, easy to implement and gives relatively high oil recovery efficiency. The composition of oil may vary to a large extent depending on the used extraction methods. Yield and quality of essential oils depend on the extraction method by which natural products are treated. Moreover, different factors contribute to the efficacy of extraction of essential oils including liquid-to-solid ratio, particle size, temperature, *etc*. [19-22].

To achieve higher quality and higher yield of oil extraction from different plant materials, it is important to select adequate techniques and optimize appropriate extraction parameters. When different parameters may affect the desired response, the response surface methodology (RSM) is a useful tool to determine the optimal conditions for the process [23-30]. Response surface methodology is statistical and mathematical method which has been applied to determine the influences of different variables and optimize methods. The principal benefit

of RSM was to decrease the number of tests trials must estimate different variables and their interactions. Because of the various process variables interact with together, the RSM method is far better than "one to one factor" approach. The RSM method is based on the multivariate nonlinear model which has been broadly applied in different fields including biological, agricultural and chemical to anticipate the optimal conditions of the systems.

Orange (*Citrus sinensis*), belongs to the Rutaceae family which cultivated mainly in India, Malaysia, Thailand, Laos and Vietnam. Citrus fruits and their byproducts play a vital role in therapeutic and economic value due to their various uses including cosmetics, food industry, folk medicine, *etc*. These health benefits stem from vitamins C, phytochemical compounds like synephrine, polyphenols, pectin, hesperidin flavonoids, *etc*. The previous study demonstrates that orange peel has an excellent total radical antioxidative potential. Essential oils from orange (*Citrus sinensis*) peels have therapeutic, antiseptic, analgesic and anti-inflammation values [31-34].

Therefore, the objective of this work was to investigate the optimal essential oil extraction from orange (*Citrus sinensis*) peel by RSM. In this research, the extraction parameters of essential oils extraction from orange (*Citrus sinensis*) peel (extraction temperature, extraction time and water-to-material ratio) was examined and optimized applying a three-level, three variable central composite design (CCD). Moreover, we also determine the volatile constituents of essential oils from the peels of orange (*Citrus sinensis*) by GC-MS.

EXPERIMENTAL

Fresh *Citrus sinensis* (orange) were collected from Ben Tre province ((latitudes 10°14′54″N and longitudes 106°22′34″E) of Vietnam in March 2019. Orange was cleaned and peeled to separate the external part, which is because of the reason that the majority of the essential oil in oil bag present in them. A 50 g of material was carried in all extractions.

Extraction of essential oils from orange (*Citrus sinensis*) peels by hydrodistillation: A quantity of 100 g of orange peels was subjected to hydrodistillation performed using a Clevenger type apparatus as described procedures [35,36]. The solvent was 1 L of distilled water and the extraction lasted about 180 min since essential oil began to be distilled in the output arm. Since, the oil is little in amount and sensitive to light, the product was carefully collected, dehydrated with anhydrous Na_2SO_4 and stored in sealed vials at 0 °C prior to gas chromatography-mass spectrometry analysis. Triplicate experiments were performed in each attempt and the mean value was reported. Yield of essential oil extraction was calculated as follows:

$$Y (\%) = \frac{V}{W} \times 100 \qquad (1)$$

where Y is the yield of essential oil was obtained, V is the volume of collected essential oil (mL) and W is the amount of raw material (g).

Experimental design with RSM of hydrodistillation: Response surface methodology (RSM) is a method that aims to describe experimental data statistically. The method is often utilized in describing correlation between a desired response with a set of numerous experimental parameters [3]. In this study, the aim of RSM was to optimize the experimental parameters for hydrodistillation of orange oil. The effect of three factors, namely water-to-material ratio, extraction time and temperature on the yield of extraction was investigated. Five different levels of parameters, varying from -1 and +1, was included. Table-1 describes the design in which parameters and corresponding levels were reported. Total, 20 experiments generated by combining 3 factors at 5 levels of variation were defined and attempted experimentally. The produced data will be subjected to regression to produce the final quadratic (second-order) polynomial regression model.

The Design-Expert software (version 11) was used to commence experiment design as well as produce statistical results of the model. ANOVA was adopted to calculate coefficient significance. Statistics of interest include the determination coefficients (R^2), the corresponding adjusted values (R^2 adj), and determination coefficient for prediction (R^2 pred).

Chemical composition of essential oil: The chemical profile of the obtained oil was analyzed by GC-MS. Prior to analysis, 25 µL of oil sample was mixed in 1 mL *n*-hexane. The instrument was GC Agilent 6890N, coupled with MS 5973 inert and HP5-MS column. The pressure of the head column was set to 9.3 psi. Following conditions were set in the GC-MS system. Carrier gas: helium; flow rate of 1.0 mL/min; split ratio of 1:100; injection volume of 1.0 µL and injection temperature of 250 °C. Temperature progress begins at 50 °C for 2 min, followed by a rise to 80 °C at 2 °C/min, then to 150 °C at 5 °C/min, then to 200 °C at 10 °C/min and to 300 °C at 20 °C/min for 5 min.

RESULTS AND DISCUSSION

Model and fitting the model: Because different factors potentially affect the extraction process, the optimization of the experimental conditions indicates a crucial step in the development of a hydrodistillation method. In this study, RSM-CCD approach illustrates independent three factors including water-to-material ratio (mL/g), temperature and time. Twenty experimental design with the RSM model was completed and displayed in Table-2. To determine the significance of each factor, F-value and "Prob. > F" (p-value) were calculated through ANOVA. Table-3 presents ANOVA results along with statistics of interest in which p-values of lower than 5 % indicates signi-

Name	Code	Levels				
		-α	-1	0	+1	+α
Water-to-material ratio (mL/g)	A	1.32	2	3	4	4.68
Extraction time (min)	B	34.78	45	60	75	85.23
Temperature (°C)	C	113	120	130	140	146

TABLE-1
INDEPENDENT VARIABLES MATRIX AND THEIR ENCODED LEVELS FOR MODEL

TABLE-2
MATRIX OF EXPERIMENTAL AND PREDICTED VALUES FOR MODEL

| Std. order | Run order | Point type | Coded variables | | | Experimental | RSM | |
			A (mL/g)	B (min)	C (°C)		Predicted	Residual
1	3	Factorial	2.00	45.00	120.00	2.00	1.97	0.0252
2	19	Factorial	4.00	45.00	120.00	1.80	1.73	0.0711
3	18	Factorial	2.00	75.00	120.00	2.20	2.25	-0.0465
4	1	Factorial	4.00	75.00	120.00	2.50	2.50	-0.0006
5	9	Factorial	2.00	45.00	140.00	1.90	1.89	0.0095
6	7	Factorial	4.00	45.00	140.00	2.00	1.94	0.0554
7	6	Factorial	2.00	75.00	140.00	2.40	2.46	-0.0622
8	16	Factorial	4.00	75.00	140.00	3.00	3.02	-0.0163
9	2	Axial	1.32	60.00	130.00	1.85	1.80	0.0483
10	12	Axial	4.58	60.00	130.00	2.00	2.06	-0.0609
11	11	Axial	3.00	34.78	130.00	1.90	1.99	-0.0915
12	14	Axial	3.00	85.23	130.00	3.20	3.12	0.0789
13	5	Axial	3.00	60.00	113.18	2.10	2.12	-0.0249
14	20	Axial	3.00	60.00	146.82	2.50	2.49	0.0123
15	4	Center	3.00	60.00	130.00	3.00	2.99	0.0087
16	8	Center	3.00	60.00	130.00	2.95	2.99	-0.0413
17	13	Center	3.00	60.00	130.00	3.00	2.99	0.0087
18	15	Center	3.00	60.00	130.00	3.00	2.99	0.0087
19	17	Center	3.00	60.00	130.00	3.00	2.99	0.0087
20	10	Center	3.00	60.00	130.00	3.00	2.99	0.0087

TABLE-3
ANALYSIS OF VARIANCE FOR RESPONSE SURFACE TO QUADRATIC MODEL FOR THE YIELD OF ORANGE OIL

Source	Sum of squares	Df	Mean	F-value	p-value	Significance
Model	4.7700	9	0.5302	137.26	< 0.0001	Significant
A-ratio	0.0811	1	0.0811	20.99	0.0010	Significant
B-time	1.5400	1	1.5400	398.73	< 0.0001	Significant
C-temperature	0.1588	1	0.1588	41.11	< 0.0001	Significant
AB	0.1250	1	0.1250	32.36	0.0002	Significant
AC	0.0450	1	0.0450	11.65	0.0066	Significant
BC	0.0450	1	0.0450	11.65	0.0066	Significant
A^2	2.0200	1	2.0200	524.00	< 0.0001	Significant
B^2	0.3409	1	0.3409	88.25	< 0.0001	Significant
C^2	0.8453	1	0.8453	218.83	< 0.0001	Significant
Residual	0.0386	10	0.0039			
Lack of fit	0.0365	5	0.0073	17.54	0.0035	
Pure error	0.0021	5	0.0004			
Core total	4.8100	19				

*P < 0.01 highly significant; 0.01 < P < 0.05 significant; P > 0.05 not significant
*Values obtained from Design-Expert 11

ficance of corresponding model terms. The significance of factor on the oil yield is presented in Fig. 1.

Preliminary inference from Table-3 reveals that all the examined first-order variables including water-to-material ratio (A), extraction time (B) and temperature (C) exerted significant impact on the extraction yield (p < 5 %). In addition, all three interactions and second order variables also showed significant relationship to the response. Regarding impact magnitude, Fig. 1 shows effect magnitude of variables as well as their signs. At first glance, greatest influences were observed in extraction time (B), quadratic ratio (A^2) and quadratic temperature (C^2). Apparently, one minute of increase in extraction time was associated with the most positive change in yield. This is contrast with the improvement in yield gained when rising temperature or ratio by one unit, which is marginal and economically inefficient. Furthermore, the presented R^2, R^2 predicted and R^2 adjusted suggested the relatively good performance of the final model. The R^2 predicted of 0.9417 agrees well with the
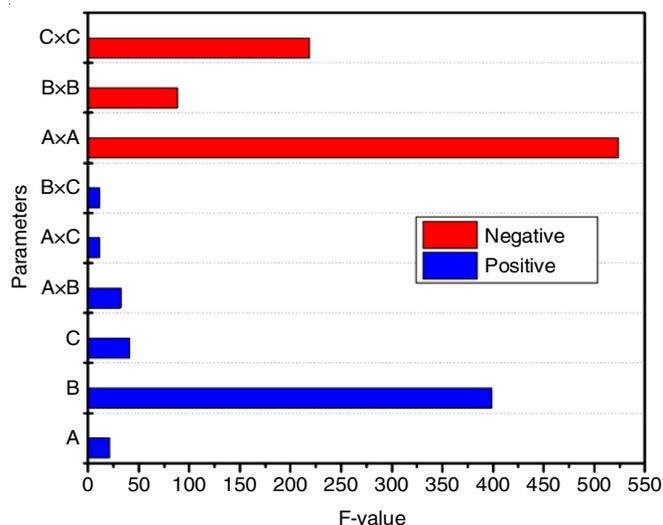
Fig. 1. Chars of the main effects (positive-negative) in the CCD

TABLE-4						
REGRESSION ANALYSIS OF MODELING FOR THE YIELD OF ORANGE OIL						
Std. Dev.	Mean	CV (%)	R^2	Adjusted R^2	Predicted R^2	Adeq Precision
0.0622	2.47	2.52	0.9920	0.9847	0.9417	31.6773

R^2 adjusted of 0.9847. Regarding precision adequacy, the obtained ratio of 31.6773 far exceeds 4, implying the results are adequately predicted (Table-4).

Fig. 2 compares actual oil yield relative to predicted yield, showing agreed values between predicted and actual numbers. This indicates the high prediction capability of the model. Assembling calculated coefficients, the second order polynomial model could be presented as follows:

$$Y = 299 + 0.0771A + 0.3358B + 0.1078C + 0.1250AB + 0.0750 AC + 0.0750 BC - 0.3748A^2 - 0.1538B^2 - 0.2422C^2 \quad (2)$$
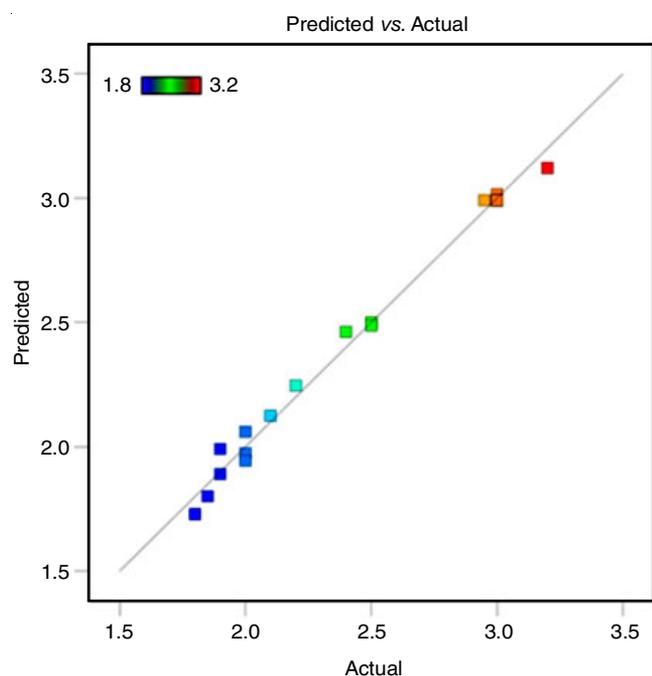
Fig. 2. Scatter diagram that compares the experimental data actual against the predicted values for the yield of extraction using RSM

For any given levels of experimental parameters, a single value of yield could be predicting using above equation. In addition, this model could also be used to conduct sensitivity analysis as one or more factors vary.

Optimization of extraction parameters: Contour plots and three-dimensional plots were constructed based on eqn. 2 (Fig. 3). By examining the plots, the interactions between independent variables could be interpreted and optimal values in

which the response reaches the highest values could be speculated.

Fig. 3a indicates that at a fixed extraction time, an increase in ratio or extraction temperature induces the yield to rise slightly. The yield seems to reach the maximum value at the moderate temperature in the range of 130-135 ºC and water-to-material ratio of 3 mL/g. On the other hand, when fixing the ratio at the central level, interaction effects could be observed in Fig. 3b, showing similar trend to that in Fig. 3a. The figure shows that optimal yield could be attained at 130 ºC after 60-75 min. This is also in line with Fig. 3c where maximum recovery of orange oil can be achieved when ratio water to material and extraction time (60-65 min) were around 3-3.5 mL/g.

The observed trend is generally positive, which is an increase of any of three examined factors results in an increase of oil yield. This could be explained by permeation of oil-containing epidermises, caused by water vapour. However, if the water is insufficient, essential oil bags will not be freed from colloids and salt, making the oil unable move outside. On the contrary, excess water may impair the yield by contributing to the dissolution and emulsification of oil. Therefore, a suitable amount of water could sufficiently facilitate solubility and diffusion of oil into water, leading to the improved yield. Furthermore, temperature could influence the drain of the oil to the solvent. However, extreme temperature may undermine the escape rate and in turn reduce the extraction efficiency due to saturation [37].

It could be concluded from Design-Expert 11 that optimal extraction conditions included temperature of 130.08 ºC, extraction time of 74.31 min and water to material ratio of 3.19 mL/g (Fig. 4). At this optimized condition, the predicted yield of the orange was 3.21 mL/100 g.

To evaluate this model, we conducted the experiment three times the optimal parameters predicted by response surface methodology and the results are presented in Table-5. Repeated experiments show that the error between experiment and predictions by Design-Expert 11 is not large (< ± 5 %). The yield of orange oil obtained under the proposed conditions in the laboratory (3.20-3.30 %) approximated the predicted value, suggesting that the obtained quadratic model could accurately predict the real yields (Table-6).

Results analysis of GC-MS: Gas chromatography mass spectrometry was employed to confirm solute identities in the orange oils. The resulting spectra and retention time are shown in Fig. 5 (Table-7). The analysis could be used to give

TABLE-5					
COMPARISON BETWEEN EXPERIMENTAL RESULTS USING OPTIMUM CONDITION AND PREDICTED RESULTS					
	A (Ratio, mL/g)	B (Time, min)	C (Heat, ºC)	Y (Yield, %)	Error (%)
RSM	3.19	74.31	130.08	3.21	
Experiment 1	3.2	74	130	3.20	-0.3115
Experiment 2	3.2	74	130	3.30	2.8037
Experiment 3	3.2	74	130	3.30	2.8037
Average Exp	3.2	74	130	3.27	1.8692
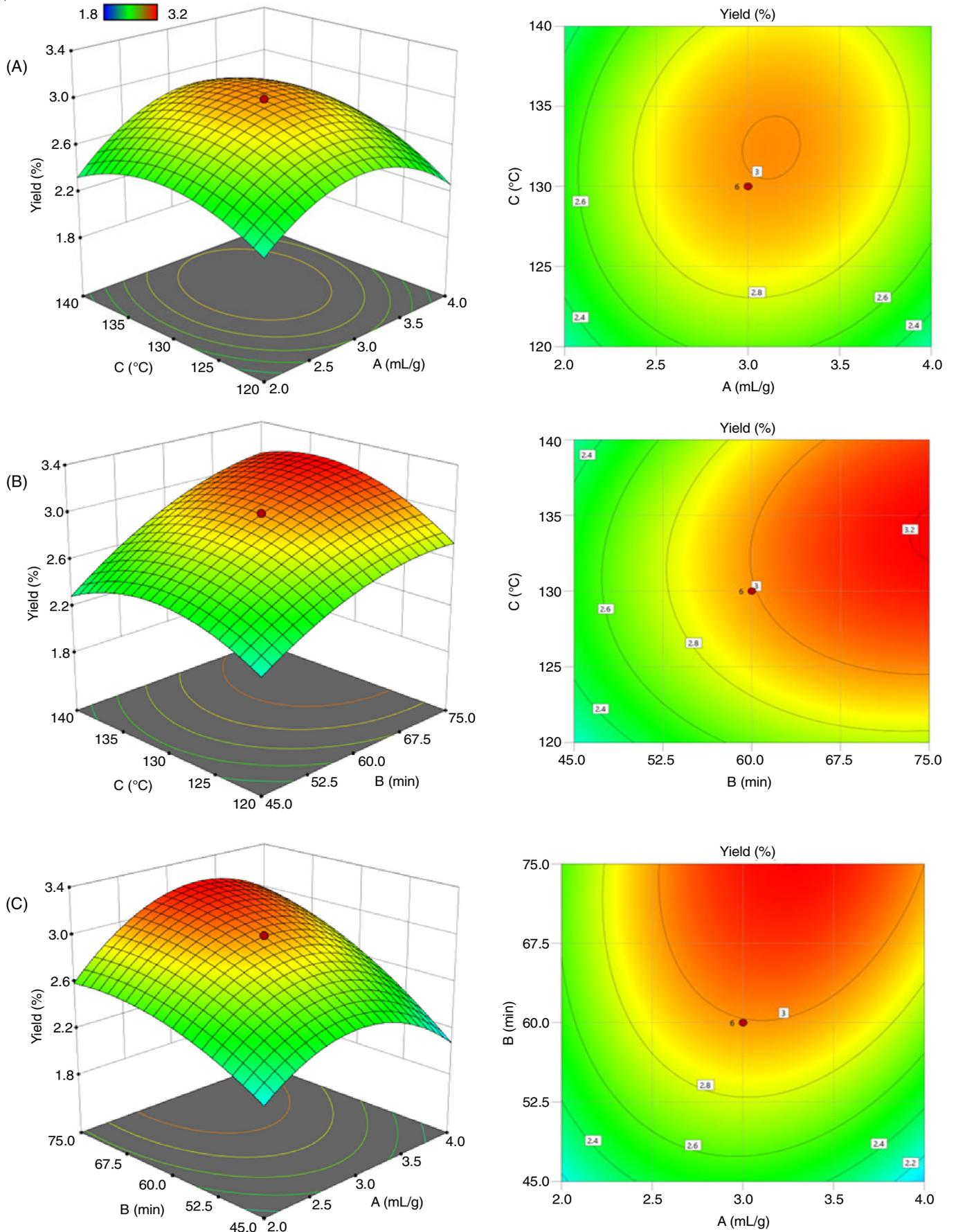
Fig. 3. 3D and 2D response surface plots of interaction relationship of yield with (A) water-to-material ratio and temperature, (B) extraction time and temperature, (C) water-to-material ratio and extraction time

Cube
Desirability
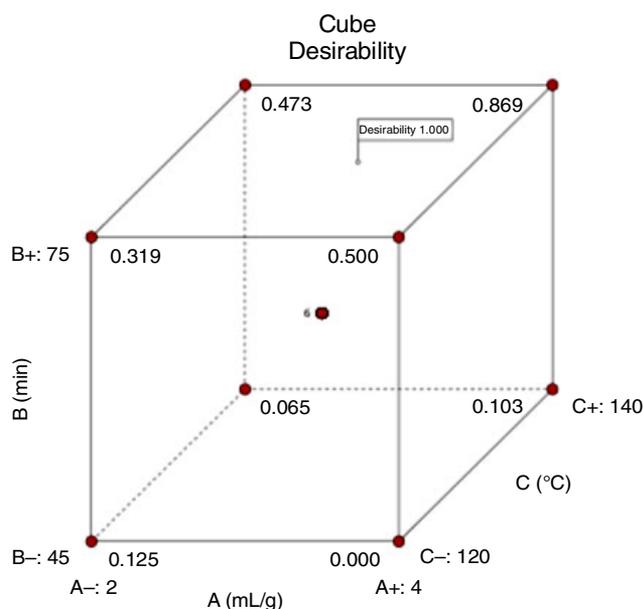
Fig. 4. Graphical optimization of extraction essential oil from orange peels and it's desirability

TABLE-6
NEGATIVE, POSITIVE LINEAR AND QUADRATIC
EFFECT OF FACTOR ON THE EXTRACTION YIELD

Factor	Coefficient estimate	df	Standard error
Intercept	2.99	1	0.0253
A-Ratio	0.0771	1	0.0168
B-Time	0.3358	1	0.0168
C-Temperature	0.1078	1	0.0168
AB	0.1250	1	0.0220
AC	0.0750	1	0.0220
BC	0.0750	1	0.0220
A^2	-0.3748	1	0.0164
B^2	-0.1538	1	0.0164
C^2	-0.2422	1	0.0164

*Values obtained from Design-Expert 11
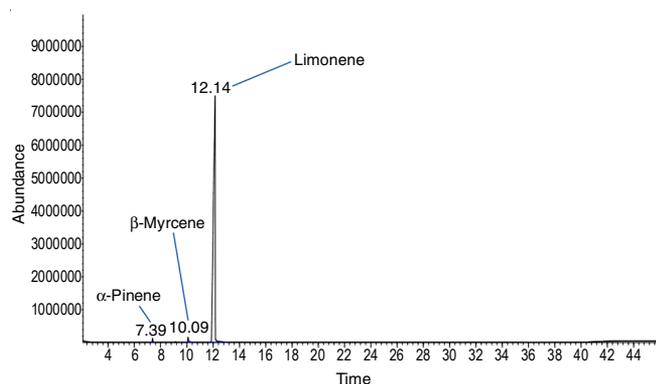
Fig 5. GC-MS chromatogram of orange oil by HD

an approximation regarding quality of essential oils. In total, three detectable compounds was found in the composition of the obtained oil. A total of three components were identified. The major shared components of orange oil belong to the mono-terpene hydrocarbons, such as limonene (98.343 %). Some components were identified only in a certain plant (Table-7). For example, α-pinene was a characteristic component of orange peel oil. In addition, we referred to other studies from China, Spain and Turkey of orange oil composition. The results of present study were consistent with previous studies in that limonene was major constituents in the orange extracts. Besides, Table-7 showed that orange oil grown in Vietnam has the highest limonene content. It is worth nothing that the chemical composition of essential oils could vary depending on geographical location and season of harvest, plant age and method of extraction [38].

The biological activities of essential oil largely depends on major components in the oil. For the essential oils of orange oil, limonene was the dominant components. It was shown that limonene figures for its toxicity to cat fleas and takes an important role in providing insecticidal resistance. In addition, the compound could be utilized as an valuable ingredient in manufacture of cosmetics products, polymers and adhesives [39]. For β-myrcene, the compound could be used as a natural base [40] and an important intermediate used in the perfumery industry. Lastly, α-pinene exhibits excellent anti-inflammatory and antimicrobial activities [41]. To conclude, the composition of oil from orange peels suggests that the oil holds significant potential in the pesticide industries. This is in line with a previous study [42] where evidence for pesticidal characteristic of an essential oil from the orange oil was presented.

Conclusion

In this research, the efficiency of hydrodistillation extraction from orange peels (*Citrus sinensis*) are determined by RSM-CCD method. There are three independent factors in the model including the ratio of water and materials, extraction time, and temperature. From this study, the extraction yield within the experimental range can predict by the quadratic polynomial model. The correlation coefficients "R2", "R2 adj." and "R2 pred." achieved 0.9920, 0.9847 and 0.9417, respectively, showing well-fitness of the model to the experimental data. The optimal conditions for extraction oil yield including the ratio of water to material, temperature, extraction time achieved 3.19 mL/g, 130.08 ℃, 74.31 min, respectively. The optimum accessible yields of extraction are achieved by 3.21 % by RSM. The orange peels are rich in limonene content which plays a pivotal function in different fields including cosmetics, food, beverage and pharmaceuticals industries. Hydrodistillation acts as a green method of extraction due to less energy-intensive process.

TABLE-7
VOLATILE CONSTITUENTS OF ESSENTIAL OIL FROM ORANGE PEELS

Peak	Retention time (min)	Compounds	Present study	Steam Distillation, China [Ref. 11]	Cold-Pressed, Spain [Ref. 12]	Cold-Pressed, Turkey [Ref. 13]
1	7.387	α-Pinene	0.520	1.49	0.54	0.42
2	10.085	β-Myrcene	1.137	6.27	1.87	1.80
3	12.145	Limonene	98.343	77.49	95.24	94.08

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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