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Optimization of Operating Conditions of Essential Oil Extraction of Vietnamese Pomelo (*Citrus grandis* L.) Peels by Hydrodistillation Process

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This study attempted the optimization of the extraction process involving essential oils from Vietnamese pomelo fruits. Three influential parameters including ratio of water and material, extraction time, and temperature were assumed to be impactful to the oil yield and were investigated by establishing a statistical model. A central composite design was adopted to generate dataset required for estimation of the model. Analysis of variance was used to calculate model significance. The results showed that optimum yield of pomelo oil is 4.46 % (v/w) corresponding to water ratio of 507 mL water to 100 g sample, temperature at 119.29 ºC and distillation time of 113.68 min. Predicted values proposed by the Design Expert 11 software well-agreed with the empirical data, suggesting the excellent predictability of the proposed models. In addition, the essential oil obtained under optimal conditions was analyzed by gas chromatography-mass spectrometry. The results indicated that D-limonene is the main component (97.318 %) of essential oil.

Keywords: Hydrodistillation, Pomelo Oil, *Citrus grandis* L., Response surface methodology, GC-MS.

INTRODUCTION

Essential oils are the mixture of various volatile compounds including hydrocarbons, alcohols, esters and aldehydes and could be obtained in various plant organs [1-8]. Essential oil of citrus is often located in oil glands of the peel and makes up of around 1-3 % of the fresh weight of fruit. Due to the presence of useful natural ingredients, essential oil from citrus find wide applications in manufacturing of cosmetics and supplement foods [9-11].

One of the species of citrus, *Citrus grandis* (L.) Osbeck, also known as pomelo, is a plant that bears large citrus fruits and belongs to the Rutaceae family that are native to China, Japan, Vietnam, Malaysia, Indonesia and Thailand [12-16]. The properties which are exhibited by pomelo essential oils include antioxidant, antiviral, anticancer, anti-inflammatory and antiallergenic properties. The peels of pomelos, which are regarded as by-products, occupy a large proportion of pomelo weight and have been found as a promising source of dietary fiber and health-beneficial compounds. Essential oils extracted from pomelo peels have also been widely used in production of foods, cosmetics, beverages and medicines as a popular flavourant. In medicine, pomelo oil is used in aromatherapy treatments.

To obtain essential oils, various techniques could be employed such as hydrodistillation, supercritical fluid extraction and microwave extraction [17-20]. Among which, hydrodistillation figures due to its viability, safety and suitability for herbs and plants. The method is facile and could be used to separate an oil from water. However, extraction efficiency of

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the method largely depends on operating conditions. As a result, selecting optimal experimental parameters becomes crucial for obtaining maximum oil yield. For chemical processes, response surface methodology (RSM) has been commonly employed. The optimization technique involves developing a statistical model that could accurately describe a desired variable relatively to its influential parameters. Results from RSM could be used to improve existing processes and contribute to existing product designs [21-30].

The aim of this work was to examine the relationship between hydrodistillation operation parameters on the efficiency of essential oil recovery process from peels of Vietnamese Pomelo (*Citrus grandis* L.) Peels. To be specific, conditional parameters generated from central composite design (CCD) will be experimentally attempted. The data will be used to establish a statistical model describing the yield from which, optimum condition would be calculated. This process is advantageous in terms of time and cost since it could reduce the required experiments to the feasible amount. The quality of obtained oil is evaluated by GC-MS analysis.

EXPERIMENTAL

Anhydrous sodium sulfate (Analytical grade) was purchased from Sigma-Aldrich (USA). Deionized water produced by Milli-Q purification system (Millipore, USA) was used as the solvent in this study. Pomelo peels (*Citrus grandis* L.) were collected from Tien Giang province of Vietnam in March 2019. The size of material used in this study includes original size, cut into fibre and grounded. The peels were then stored at room temperature as required.

Pomelo (*Citrus grandis* L.) peels essential oil extraction by hydrodistillation process: For extraction of oil by hydrodistillation, 100 g of peels were placed in a 1 L flask containing distilled water in a specified ratio. Following that hydrodistillation took place for 180 min using a Clevenger-type apparatus. The produced vapour was then subjected to condensation in the apparatus. The essential oil is separated from water by decantation, dehydrated with anhydrous sodium sulfate and stored at 4 ºC for further analysis. Each extraction was performed three times. Yield of essential oil (Y) was calculated as follows:

$$Y\ (\%) = \frac{\text{Volume of essential oil (mL)}}{\text{Amount of raw materials (g)}} \times 100 \qquad (1)$$

Optimization of the extraction process using central composite design model: Response surface methodology, in conjunction with central composite design (CCD), was employed to optimize the extraction process by generating a set of experimental trials. A calculation of experimental trials and optimum yield was performed using Design Expert 11. A central composite design approach was adopted incorporating three variables factor (Water-to-material ratio, extraction time and temperature) and one response (pomelo oil yield). The final set consists of 20 experiments corresponding 8 factorial, 6 axial point and 6 center points as shown in Table-1. For each variable, the initial level was determined from the acquaintance with the materials and preliminary experiments. Predicted optimal yield was then verified by further experiments.

For statistical analysis, analysis of variance (ANOVA) was used to calculate model and coefficient significance. Fitting accuracy was evaluated through various statistics including significance, F-value, coefficient of determination and lack of fit ratio.

Chemical composition analysis GC-MS: Obtained essential oil was then assayed for chemical composition using GC-MS technique. Prior to analysis, 25 µL of oil sample was mixed into 1.0 mL of *n*-hexane. A GC Agilent 6890N apparatus was coupled with MS 5973 inert and HP5-MS column. The head column pressure was set at 9.3 psi. Helium gas was used as the carrier. The following conditions were designated: flow rate of 1.0 mL/min; split injection with the ratio of 1:100 and volume of 1.0 µL. Temperature of injection was set at 250 ºC. Temperature progress in the oven initiated at 50 ºC for 2 min, followed by a rise to 80 ºC at 2 ºC/min, then to 150 ºC at 5 ºC/min, then to 200 ºC at 10 ºC/min and to 300 ºC at 20 ºC/min for 5 min.

RESULTS AND DISCUSSION

Optimization of extraction process

Central composite design (CCD) and fitting the model: In CCD, different ratio (A: 3.32, 4, 5, 6 and 6.68 mL/g); extraction time (B: 79.77, 90, 105, 120 and 130.23 min) and temperature (C: 103.18, 110, 120, 130 and 136.82 ºC) were selected and ascendingly encoded as -α, -1, 0, +1, +α, respectively (Table-2). After running 20 experiments, ANOVA was performed (Table-3). The significance was recognized with p-value < 0.05. At first glance, it was found that ratio exhibited insignificant effect on yield, as demonstrated by high p-value of the coefficient. However, since there is only one insignificant model term, model reduction is not necessary to improve the prediction and fitting accuracy.

Non-significant "Lack of Fit" ($p > 0.05$) indicates that the proposed model fit the experimental data well. The value of the coefficient of determinations R^2, predicted R^2 and adjusted R^2 indicate the correlation between the actual data and the predicted values by the RSM model.

The second-order model based on condition variables was established as follows:

$$Y = 4.38 + 0.0062A + 0.2927B + 0.1005C + 0.0250AB - 0.1375AC + 0.0375BC - 0.2510A^2 - 0.2510\,B^2 - 0.3305C^2 \qquad (2)$$

As indicated in eqn. 2, extraction time exerted the most prominent effect on the oil yield from pomelo peels. The linear

TABLE-1
EXPERIMENTAL DESIGN VARIABLES AND THEIR ENCODED LEVELS FOR RSM MODEL

Variables	Code	Range and level				
		–α	–1	0	+1	+α
Water-to-material ratio (mL/g)	A	3.32	4	5	6	6.68
Extraction time (min)	B	79.77	90	105	120	130.23
Temperature (°C)	C	103.18	110	120	130	136.82

TABLE-2
MATRIX OF ACTUAL AND PREDICTED VALUES FOR RSM MODEL

Std. order	Run order	Code variables			Actual	RSM	
		A (mL/g)	B (min)	C (°C)		Predicted	Residual
1	18	–1	–1	–1	3.10	3.08	0.0234
2	16	1	–1	–1	3.30	3.31	–0.0139
3	20	–1	1	–1	3.55	3.54	0.0130
4	17	1	1	–1	3.90	3.87	0.0256
5	6	–1	–1	1	3.45	3.48	–0.0276
6	5	1	–1	1	3.15	3.16	–0.0149
7	19	–1	1	1	4.10	4.09	0.0119
8	7	1	1	1	3.85	3.88	–0.0254
9	11	–α	0	0	3.65	3.66	–0.0132
10	4	α	0	0	3.70	3.68	0.0160
11	15	0	–α	0	3.20	3.18	0.0187
12	10	0	α	0	4.15	4.17	–0.0159
13	12	0	0	–α	3.25	3.28	–0.0296
14	13	0	0	α	3.65	3.62	0.0324
15	9	0	0	0	4.35	4.38	–0.0334
16	1	0	0	0	4.40	4.38	0.0166
17	2	0	0	0	4.40	4.38	0.0166
18	8	0	0	0	4.40	4.38	0.0166
19	3	0	0	0	4.40	4.38	0.0166
20	14	0	0	0	4.35	4.38	–0.0334

TABLE-3
ANOVA FOR RESPONSE SURFACE TO QUADRATIC MODEL FOR THE YIELD OF POMELO OIL

Source	Sum of Squares	df	Mean	F-value	p-value	Significance
Model	4.31	9	0.4801	498.60	< 0.0001	Significant
A-ratio	0.005	1	0.0005	0.5377	0.4082	Not significant
B-time	1.17	1	1.17	1215.32	< 0.0001	Significant
C-temperature	0.1380	1	0.1380	143.30	< 0.0001	Significant
AB	0.0050	1	0.0050	5.19	0.0459	Significant
AC	0.1513	1	0.1513	157.08	< 0.0001	Significant
BC	0.0112	1	0.0112	11.68	0.0066	Significant
A^2	0.9076	1	0.9076	942.60	< 0.0001	Significant
B^2	0.9076	1	0.9076	942.60	< 0.0001	Significant
C^2	1.57	1	1.57	1634.88	< 0.0001	Significant
Residual	0.0096	10	0.0010			
Lack of fit	0.0063	5	0.0013	1.89	0.2510	Not significant
Pure error	0.0033	5	0.0007			
Core total	4.33	19	Std. Dev	0.0310	R^2	0.9978
			Mean	3.81	Adjusted R^2	0.9958
			C.V. (%)	0.8134	Predicted R^2	0.9874
					Adeq precision	59.5582

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

effects of A, B, C and interaction of AB, BC are also significant and positive, suggesting that oil yield could be improved with increasing examined parameters.

Model fitting was evaluated based on residual analysis and coefficient of determination. It is expected that residuals, which are defined as difference between predicted and actual data, are random and should follow normal distribution. Fig. 1 plotting studentized residuals against probability shows that the data points are distributed across a line, indicating that residuals are likely to follow a normal distribution. This is contrasted with the situation where an S shape curve is observed, which often indicates non-normal distribution of residuals and the need for model transformation. In Fig. 2, data points corresponding to actual yield values and predicted yield values are scattered across the 45-degree line and are situated equally on both sides of the line. This indicates that the obtained model can predict actual extraction yield well. For independence verification, Fig. 3 further elaborates the distribution of residuals with respect to run number and predicted value, respectively.

Analysis of response surface methodology: Mutual interactions of independent variables were visualized using contour plots and three-dimensional response surface plot. In addition, the plots could be used to speculate the optimal yields. In each plot, one parameter is kept constant at the central level and the other two are allowed to vary. Interactive effects of extraction time and temperature on extraction yield of pomelo oil were displayed in Fig. 4a. Apparently, increasing extraction time exhibits a larger impact on oil yield than elevating temperature does, which is in line with the result of the quadratic model. The contour plot indicated that the oil yield could reach approximately 4.4 % when time ranges from 101.05 to 124.74 min and temperature ranges from 115.53 to 128.05 ºC.

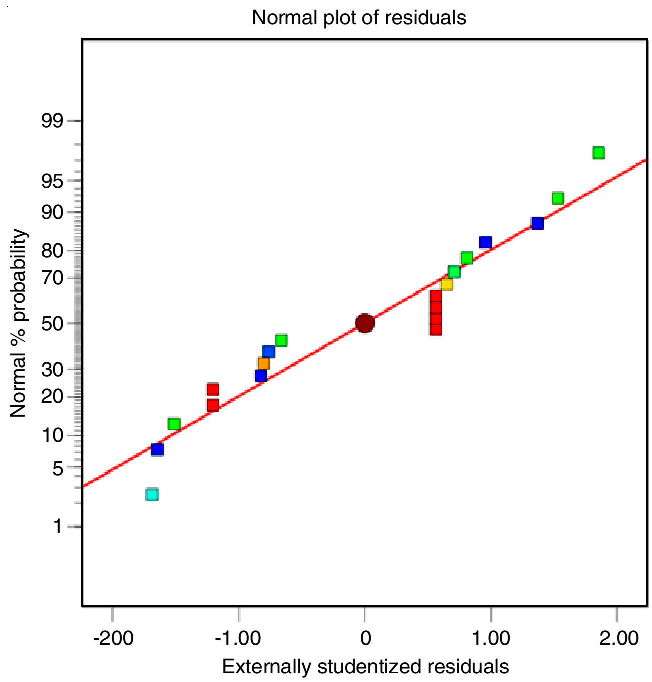

Fig. 1. Normal plot of residuals

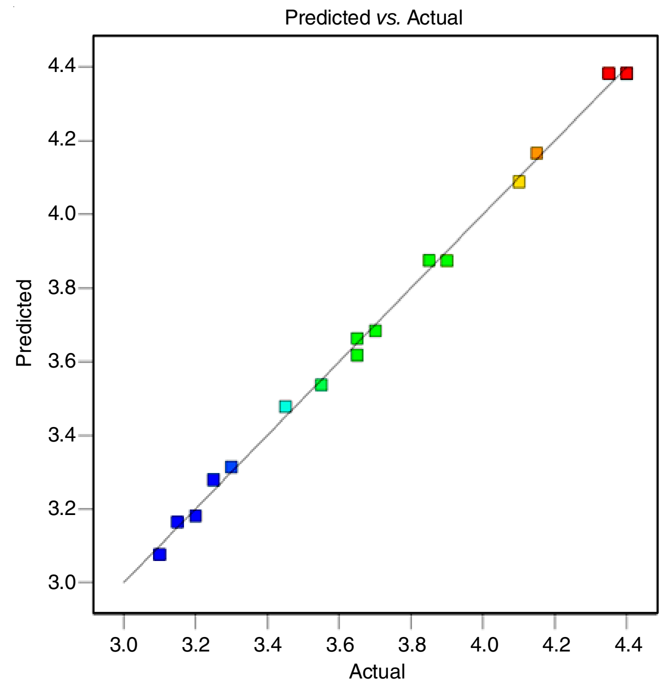

Fig. 2. Predicted and actual plot

The interaction effects of water-to-material ratio and extraction time on the pomelo oil yield are presented in Fig. 4b. The results showed that in the ratio range from 4.38 to 5.80 mL/g and time range from 101.05 to 124.74 min, the extraction yield peaked. This improved yield is caused by enlarged interfacial area when increasing the quantity of solvent, facilitating the release from disrupted cells in the materials [31,32].

Surface plot demonstrating effect of temperature and water-to-material ratio is shown in Fig. 4c. Similar to previous plots, the oil yield was shown to be positively responsive by elevated temperature until 126.26 ºC, where the yield started to decline thereafter. The positive influence of heat on extraction yield could be explained by the rapid destruction of the plant cells, which in turn causes intracellular components to quickly diffuse into the solvent. On the other hands, rapid acceleration of temperature could also cause components in the essential oil to partially decompose, leading to reduced yield. From three plots and further calculation from the quadratic model, it could be concluded that the highest pomelo oil extraction yield (4.46 %) could be achieved at water-to-material ratio of 5.07 mL/g, extraction time of 113.68 min and temperature at 119.29 ºC with desirability of 1.

Verification experiment: The calculated optimal conditions were then experimentally verified with almost identical parameters (Table-4). Under those conditions, actual yields of pomelo oil extraction ranged from 4.45 to 4.50 (%, v/w) and

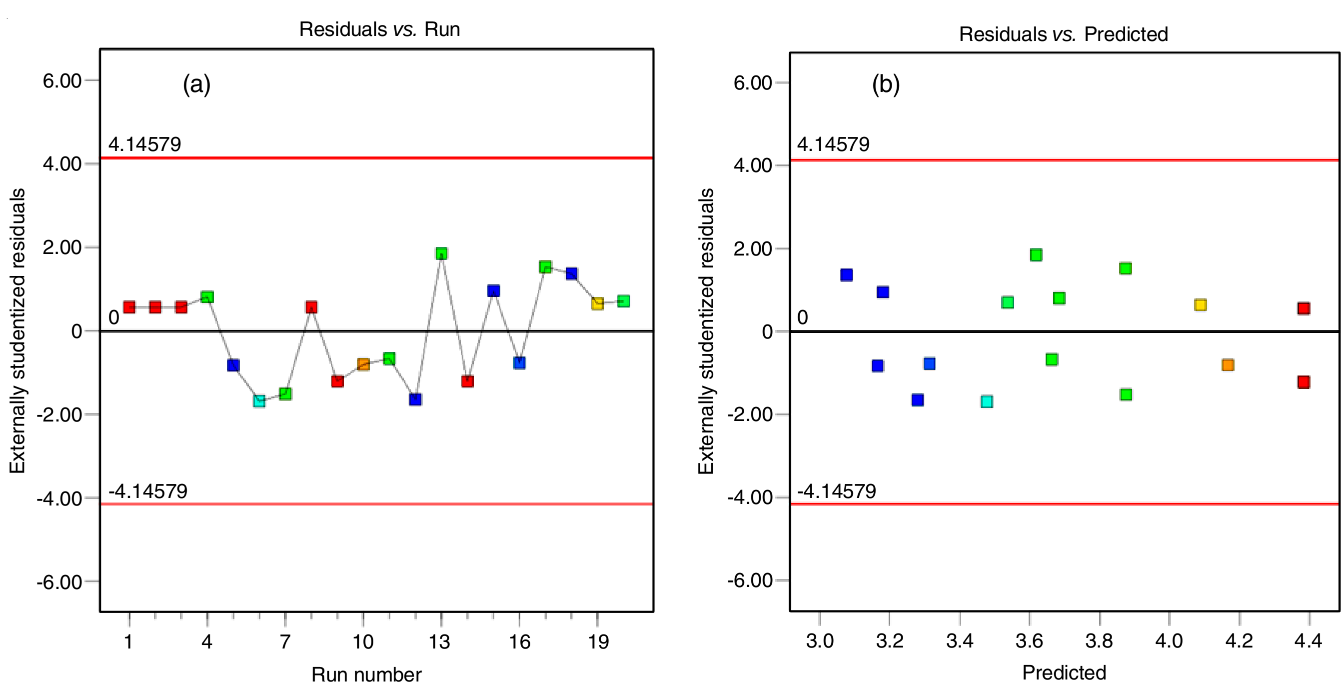

Fig. 3. Studentized residuals *versus* (A) Run number, (B) Predicted responses

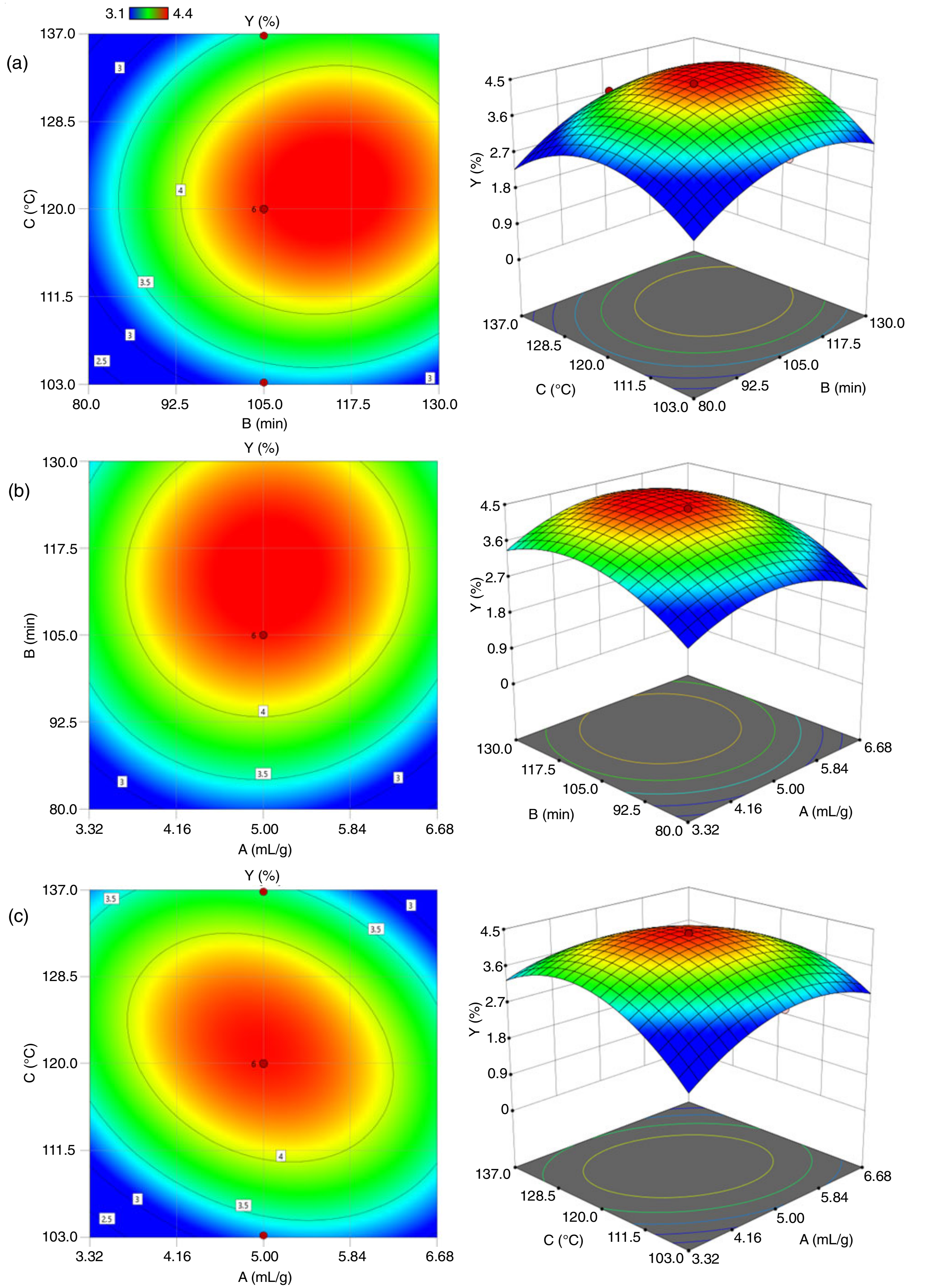

Fig. 4. 2D and 3D response surface plots of interaction relationship of yield (Y) with (a) Extraction and temperature, (b) Water-to-material ratio and extraction time, (c) Water-to-material ratio and temperature

TABLE-4
COMPARISON BETWEEN PREDICTED BY RSM MODEL AND EXPERIMENTAL RESULTS

	A (ratio, mL/g)	B (time, min)	C (heat, °C)	Y (Yield, %)	Error (%)
Predicted	5.07	113.68	119.29	4.46	–
Experiment 1	5.07	113.70	120.00	4.50	0.8969
Experiment 2	5.07	113.70	120.00	4.45	-0.2242
Experiment 3	5.07	113.70	120.00	4.45	-0.2242
Average exp	5.07	113.70	120.00	4.47	0.2242

TABLE-5
CHEMICAL CONSTITUENTS OF POMELO OIL

Peak	Retention time (min)	Compounds	This study	Turkey HD [Ref. 7]	Hong Kong HD [Ref. 8]
1	7.272	α-Pinene	0.636	0.70	0.45
2	9.008	Sabinene	0.227	0.60	0.14
3	9.949	β-Myrcene	1.350	0.90	2.49
4	10.503	α-Phellandrene	0.469	0.10	0.20
5	11.946	D-Limonene	97.318	88.60	82.58

the predicted yield of 4.46 approximatly with very small errors of 0.22 %. The low deviation indicated that the developed model was reliable and reasonable.

GC-MS: The composition of essential oil from pomelo was identified by GC-MS analysis. The produced retention times and GC-MS spectra were compared with spectra of authentic samples and the mass spectra library. In this study, the essential oil has been obtained by hydrodistillation at optimal conditions, giving the yield of 4.5 %. Overall, five constituents were detected from peels, accounting for 100 % of the total content. D-Limonene was the major compound accounting for 97.318 %. Limonene was reportedly insecticidal to cath fleas and may play a part in conferring resistance to trees against attack by insect. Limonene is commonly used as a dietary supplement and fragrance ingredient for cosmetics products, polymers and adhesives [33]. Additionally, four other compounds were also identified at a retention time of 7.272, 9.008, 9.949 and 10.503 min, which corresponds to α-pinene (0.636 %), sabinene (0.227 %), β-myrcene (1.350 %) and α-phellandrene (0.469 %), respectively (Fig. 5). In comparison with published results, reported pomelo oil composition seems to be different [7,8]. To be specific, hydrodistilled oil from pomelo of Turkey and Hong Kong was found to be abundantly composed of D-Limonene at 88.60 and 82.58, respectively, which are lower than the content in present study. Such discrepancies may suggest harvest timing, geographical location and age of the plant, which contribute the difference of chemical composition of pomelo oils (Table-5).

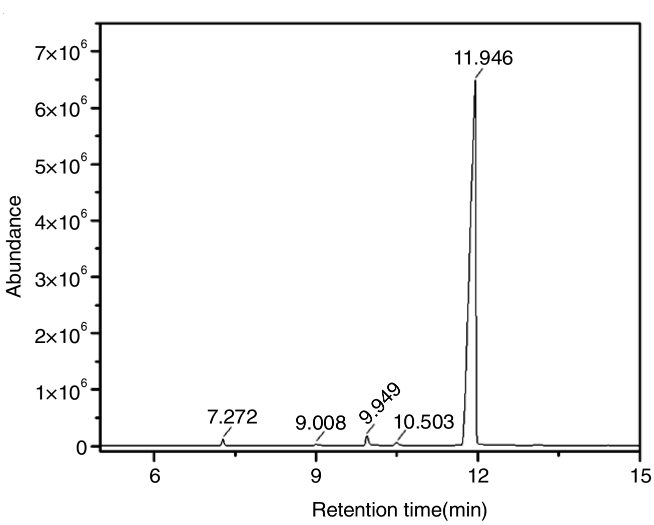

Fig. 5. GC-MS chromatogram of pomelo oil

Conclusion

The hydrodistillation of essential oils from Vietnamese pomelo (*Citrus grandis* L.) peels is reported. In addition, RSM was adopted to optimize the process, giving the highest possible yield.Three condition parameters that were considered include water-to-material ratio, extraction time, and temperature. From RSM analysis, it is concluded that water-to-material ratio, extraction time, temperature and interaction of ratio and time, time and temperature contributes significantly for efficient production of pomelo oil. Furthermore, under optimal condition, confirmed yield of extraction was 4.46 %, which also agrees well with the predicted results. The composition of pomelo oils was determined by GC-MS and five components were identified. Compared with other reported works, the obtained results indicated pomelo peels in Vietnam extracted by hydrodistillation consist of a high content of D-limonene.

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CONFLICT OF INTEREST

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

REFERENCES

1. H. Zhai, H. Liu, S. Wang, J. Wu and A.M. Kluenter, *An. Nutr.*, **4**, 179 (2018); https://doi.org/10.1016/j.aninu.2018.01.005.
2. T.H. Tran, H.H.H. Nguyen, D.C. Nguyen, T.Q. Nguyen, H. Tan, L.T.H. Nhan, D.H. Nguyen, L.D. Tran, S.T. Do and T.D. Nguyen, *Processes*, **6**, 206 (2018); https://doi.org/10.3390/pr6110206.
3. T.H. Tran, P.T.N. Nguyen, T.N. Pham, D.C. Nguyen, T.P. Dao, T.D. Nguyen, D.H. Nguyen, D.V.N. Vo, X.T. Le, N.T.H. Le and L.G. Bach, *IOP Conf. Series Mater. Sci. Eng.*, **479**, 012002 (2019); https://doi.org/10.1088/1757-899X/479/1/012002.

4. T.H. Tran, P.T.N. Nguyen, V.T.T. Ho, T.H.N. Le, L.G. Bach and T.D. Nguyen, *IOP Conf. Series Mater. Sci. Eng.*, **479**, 012015 (2019); https://doi.org/10.1088/1757-899X/479/1/012015.
5. M. Perricone, E. Arace, M.R. Corbo, M. Sinigaglia and A. Bevilacqua, *Front. Microbiol*., **6**, 76 (2015); https://doi.org/10.3389/fmicb.2015.00076.
6. J.F. Ayala-Zavala, G.A. Gonzalez-Aguilar and L. Del Toro-Sànchez, *J. Food Sci*., **74**, 84 ((2009) https://doi.org/10.1111/j.1750-3841.2009.01294.x.
7. F. Bakkali, S. Averbeck, D. Averbeck and M. Idaomar, *Food Chem. Toxicol*., **46**, 446 (2008); https://doi.org/10.1016/j.fct.2007.09.106.
8. S. Burt, *Int. J. Food Microbiol*., **94**, 223 (2004); https://doi.org/10.1016/j.ijfoodmicro.2004.03.022.
9. A.K. Maurya, S. Mohanty, A. Pal, C.S. Chanotiya and D.U. Bawankule, *J. Ethnopharmacol.*, **212**, 86 (2018); https://doi.org/10.1016/j.jep.2017.10.018.
10. M.S.A. Aamri, N.M.A. Abousi, S.S.A. Jabri, T. Alam and S.A. Khan, *J. Taibah Univ. Med. Sci.*, **13**, 108 (2018).
11. C.E. Palma, P.S. Cruz, D.T.C. Cruz, A.M.S. Bugayong and A.L. Castillo, *Ind. Crops Prod.*, **128**, 108 (2019); https://doi.org/10.1016/j.indcrop.2018.11.010.
12. L. Chen, Y. Lai, L. Dong, S. Kang and X. Chen, *Microb. Pathog.*, **113**, 365 (2017); https://doi.org/10.1016/j.micpath.2017.11.018.
13. K. Hosni, I. Hassen, Y. M'Rabet, H. Sebei and H. Casabianca, *Biochem. Syst. Ecol.*, **50**, 65 (2013); https://doi.org/10.1016/j.bse.2013.03.035.
14. H.D. Jang, K.S. Chang, T.C. Chang and C.L. Hsu, *Food Chem.*, **118**, 554 (2010); https://doi.org/10.1016/j.foodchem.2009.05.020.
15. M. Razzaghi-Abyaneh, M. Shams-Ghahfarokhi, M.-B. Rezaee, K. Jaimand, S. Alinezhad, R. Saberi and T. Yoshinari, *Food Control*, **20**, 1018 (2009); https://doi.org/10.1016/j.foodcont.2008.12.007.
16. M.R. Cruz-Valenzuela, M.R. Tapia-Rodríguez, F.J. Vazquez-Armenta, B.A. Silva-Espinoza and J.F. Ayala-Zavala, Lime (*Citrus aurantifolia*) Oils, In: Essential Oils in Food Preservation, Flavor and Safety, Cahp. 61, pp. 531-537 (2016).
17. B. Uysal, F. Sozmen, O. Aktas, B.S. Oksal and E.O. Kose, *Int. J. Food Sci. Technol.*, **46**, 1455 (2011); https://doi.org/10.1111/j.1365-2621.2011.02640.x.
18. W. Chen and A.M. Viljoen, *S. Afr. J. Bot.*, **76**, 643 (2010); https://doi.org/10.1016/j.sajb.2010.05.008.
19. H. Zhang, Z. Lou, X. Chen, Y. Cui, H. Wang, X. Kou and C. Ma, *J. Food Eng.*, **244**, 126 (2019); https://doi.org/10.1016/j.jfoodeng.2018.09.014.
20. A. Mohagheghniapour, M.J. Saharkhiz, M.T. Golmakani and M. Niakousari, *Sustain. Chem. Pharm.*, **10**, 118 (2018); https://doi.org/10.1016/j.scp.2018.10.008.
21. A. Franco-Vega, N. Ramírez-Corona, A. López-Malo and E. Palou, *J. Food Eng.*, **247**, 1 (2019); https://doi.org/10.1016/j.jfoodeng.2018.11.007.
22. T. Van Tran, Q.T.P. Bui, T.D. Nguyen, N.T.H. Le and L.G. Bach, *Adsorpt. Sci. Technol.*, **35**, 72 (2017); https://doi.org/10.1177/0263617416669152.
23. T. Van Tran, Q.T.P. Bui, T.D. Nguyen, V.T.T. Ho and L.G. Bach, *Water Sci. Technol.*, **75**, 2047 (2017); https://doi.org/10.2166/wst.2017.066.
24. T. Van Thuan, B.T.P. Quynh, T.D. Nguyen, V.T.T. Ho and L.G. Bach, *Surf. Interf.*, **6**, 209 (2017); https://doi.org/10.1016/j.surfin.2016.10.007.
25. N.P.T. Nhan, T.T. Hien, L.T.H. Nhan, P.N.Q. Anh, L.T. Huy, T.C.T. Nguyen, D.T. Nguyen and L.G. Bach, *Diffus. Defect Data Solid State Data Pt. B Solid State Phenom.*, **279**, 235 (2018); https://doi.org/10.4028/www.scientific.net/SSP.279.235.
26. F.A. Mustapha, J. Jai, N.H. Nik Raikhan, Z.I.M. Sharif and N.M. Yusof, *Food Control*, **99**, 106 (2019); https://doi.org/10.1016/j.foodcont.2018.12.042.
27. M.A. Bezerra, R.E. Santelli, E.P. Oliveira, L.S. Villar and L.A. Escaleira, *Talanta*, **76**, 965 (2008); https://doi.org/10.1016/j.talanta.2008.05.019.
28. O. Larkeche, A. Zermane, A.-H. Meniai, C. Crampon and E. Badens, *J. Supercrit. Fluids*, **99**, 8 (2015); https://doi.org/10.1016/j.supflu.2015.01.026.
29. F. Valderrama and F. Ruiz, *Comput. Chem. Eng.*, **117**, 25 (2018); https://doi.org/10.1016/j.compchemeng.2018.05.009.
30. D. Belhachat, L. Mekimene, M. Belhachat, A. Ferradji and F. Aid, *J. Appl. Res. Med. Arom. Plants*, **9**, 132 (2018); https://doi.org/10.1016/j.jarmap.2018.04.003.
31. T.H. Tran, L.K. Ha, D.C. Nguyen, T.P. Dao, L.T.H. Nhan, D.H. Nguyen, T.D. Nguyen, D.V.N. Vo, Q.T. Tran and L.G. Bach, *Processes*, **7**, 56 (2019); https://doi.org/10.3390/pr7020056.
32. T.P. Dao, D.C. Nguyen, D.T. Nguyen, T.H. Tran, P.T. Nhan Nguyen, N.T. Hong Le, X.T. Le, D.H. Nguyen, D.V. N. Vo and L.G. Bach, *Asian J. Chem.*, **31**, 977 (2019); https://doi.org/10.14233/ajchem.2019.21667.
33. H.C. Chen, L.W. Peng, M.J. Sheu, L.Y. Lin, H.M. Chiang, C.T. Wu, C.S. Wu and Y.C. Chen, *J. Food Drug Anal.*, **21**, 363 (2013); https://doi.org/10.1016/j.jfda.2013.08.003.