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Response Surface Methodology for Optimization Studies of Microwave-assisted hydrodistillation of essential oil from Vietnamese *Citrus aurantifolia* (Lemon fruit)

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Abstract. In this study, microwave-assisted hydrodistillation extraction technique was applied to extract the essential oil from Vietnamese *Citrus aurantifolia* (Lemon fruit). The optimal conditions for microwave-assisted extraction of lemon fruit oil was determined by response surface methodology. A central composite design (CCD) was used to estimate the effects of three independent variables plant material to extraction time (B: 30, 60, 90 min), microwave power (C: 300, 450, 600 W), and solvent ratio (A: 2, 3, 4 mL/g), and on the extraction yield of Lemon fruit oil. The optimal extraction conditions of *Citrus aurantifolia* oil was plant material to solvent ratio 2.80 mL/g, extraction time 63.29 min and microwave power 477.82W. The maximum Lemon fruit oil yield was 2.427%. Under the extraction condition, the experimental values matched with the anticipated results by analysis of variance. It showed high fitness of the model used and the benefit of response surface methodology for optimizing and indicate the expected extraction condition. .

1. Introduction

Nowadays, the application of the microwave-assisted method in order to extract constituents from plant material has revealed enormous research potential and interest. [1-4]. A previous study demonstrated that using the microwave extraction method is a choice that could be advanced than the conventional methods due to the high levels of product purity, reduced extraction time and more efficient heating. [5-7]. To achieve higher quality and higher yield of oil extraction from different plant materials, it is important to



select suitable techniques and optimize extraction parameters. Given that a certain response, such as oil yields, could be significantly influenced by various individual experimental conditions and interactions of such variables, the response surface methodology may prevail as an appropriate method to identify the optimal conditions. RSM involves establishing a regression equation that describes the relationship between controllable experimental parameters, their interactions and desired response. Thanks to the widespread application of RSM, development of new and improved products has been rapid and increasingly efficient [8-15]. Citrus fruits play an important role in commercial crops grown in around the world. It has gained attention not only for their medicinal value but also for their nutritional characteristics. Citrus species have a broad spectrum of biological activities, including antioxidant, analgesic, antiviral, antibacterial and so on. Citrus fruits are one of the best sources of ascorbic acid and different bioactive compounds such as limonoids, coumarins, and flavonoid [16]. Thus, the objectives of this work was to use the microwave-assisted hydrodistillation techniques for the extraction of essential oil from Vietnam *Citrus aurantifolia* (Lemon fruit) commonly used in the food, pharmaceutical and cosmetic industries. The microwave-assisted extraction parameters of essential oils from the Lemon fruit was examined and optimized using a three-level, three variable central composite design (CCD).

2. Materials and Method

2.1 Plant sample preparation

Citrus aurantifolia (Lemon fruit) was collected from Tien Giang province, Vietnam. It was then soaked with water to eliminate impurities and was dried. After that, the peels were separated from the lemon sarcocarp. All raw materials are stored in a non-hygroscopic bag, which is stored in a cooler (LC-1416B, Alaska, Vietnam) at temperatures under 10°C.

2.2 Extraction method

The lemon peels is extracted to obtain the essential oil by a system consisting of a microwave oven MW71E (manufactured by SAMSUNG, Vietnam), which acts as a source of heat for the extraction process and a hydro-distillation apparatus (Clevenger type apparatus, Germany) used to condense and separate the extracted oil and water phase. Finally, the lemon essential oil was anhydrated using sodium sulfate purchased from Sigma Aldrich (US).

2.3 Experimental design with RSM

The response surface methodology was chosen to optimize the effect factors of lemon oil on the effect of the ratio of water and lemon peels (A), time extraction (B), and microwave power (C) to maximize essential oil yield (Y). The central composite design (CCD) offers the experiment matrix designs comprising variation levels of experimental parameters, which were displayed in Table 1. Parameter A was set in range of 2:1–4:1 mL/g, B was set from 30 to 90 min, and C was fixed in range of 300 to 600 W. The raw materials in the RSM experiments were cut to a size of about 1-2 mm. For the statistical dissect, Design-Expert® software version 11, Minneapolis was adopted to assume the experimental design and to check complex multinomial to analogue the data.

Table 1. Encodes the values of the optimization factors

Levels	Independent factors		
	A (mL/g)	B (Min)	C (W)
Minimum point (-1)	2	30	300
Central point (0)	3	60	450
Maximum point (+1)	4	90	600

3. Results and Discussion

The results of 20 experimental runs with the RSM model were completed design and shown in Table 2. The table 2 results displayed was the yield oils extracted would be severely changed when the survey parameters changed (ratio, time, and microwave power). In Table 2, we see not only the actual experimental results shown, but also the predicted results made by DX11 software.

Table 2. Results of the actual and predicted values for the RSM model

	Independent variables			Y (%)			Independent variables			Y (%)	
	A	B	C	Actual	Predicted		A	B	C	Actual	Predicted
1	2	30	300	1.90	1.90	11	3	10	450	1.85	1.86
2	4	30	300	1.85	1.84	12	3	111	450	2.00	1.99
3	2	90	300	2.00	2.00	13	3	60	198	1.90	1.91
4	4	90	300	1.95	1.95	14	3	60	702	2.10	2.09
5	2	30	600	2.05	2.05	15	3	60	450	2.45	2.42
6	4	30	600	1.95	1.95	16	3	60	450	2.40	2.42
7	2	90	600	2.10	2.11	17	3	60	450	2.45	2.42
8	4	90	600	2.00	2.00	18	3	60	450	2.40	2.42
9	1.3	60	450	2.15	2.14	19	3	60	450	2.40	2.42
10	4.7	60	450	2.00	2.01	20	3	60	450	2.40	2.42

Table 3. ANOVA for Quadratic model

Source	Sum of Square	Degree of freedom	Mean Square	F-value	Prob. > F	Comment
Model	0.8915	9	0.0991	249.91	< 0.0001	significant
A-A	0.0223	1	0.0223	56.34	< 0.0001	
B-B	0.0223	1	0.0223	56.34	< 0.0001	
C-C	0.0397	1	0.0397	100.17	< 0.0001	
AB	0.0000	1	0.0000	0.0000	1.0000	
AC	0.0012	1	0.0012	3.15	0.1061	
BC	0.0012	1	0.0012	3.15	0.1061	
A ²	0.2101	1	0.2101	530.02	< 0.0001	
B ²	0.4352	1	0.4352	1097.88	< 0.0001	
C ²	0.3125	1	0.3125	788.38	< 0.0001	
Residual	0.0040	10	0.0004			
Lack of Fit	0.0006	5	0.0001	0.1891	0.9542	not significant
Pure Error	0.0033	5	0.0007			
Std. Dev.= 0.0199		Mean = 2.11		C.V. % = 0.9413		R ² = 0.9956
Adjusted R ² = 0.9916		Predicted R ² = 0.9893		Adeq Precision = 40.9550		

The Table 3 displayed results of ANOVA for Quadratic model for the process of extracting lemon oil. In this table, F-value, P-values and Lack of Fit F-value are parameters that exhibit the interaction of the conditions. The Model F-value of 249.91 implies the model by DX11 software is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. In this case A, B, C, A², B², C² are significant model terms. The Lack of Fit F-value of 0.19 implies the Lack of Fit is not significant relative to the pure error. The Predicted R² of 0.9893 is in agreement with the Adjusted R² of 0.9916. The 40.955 value shows an signal and this model can be applied to navigate the design space. In addition, the experimental model was considered fit when calculated residuals follow a random pattern as shown in Figure 1A. Figure 1B presented that data points corresponding to anticipated and actual values are scattered across the 45 degree line with close proximity, suggesting that the actual results are accurately anticipated from the factor values. Thus, based on the data analysis of the oil yield from experiments, the RSM software results in optimized parameters as A = 2.80:1 (mL/g), B = 63.29 (min), and C = 477.82 (W) to obtain the yield of 2.427% with 96.2% reliability with the quadratic model described by the following equation: Yield of lemon oil = $2.42 - 0.0404A + 0.0404B + 0.0539C - 0.0125AC - 0.0125BC - 0.1207A^2 - 0.1738B^2 - 0.1473C^2$ (2)

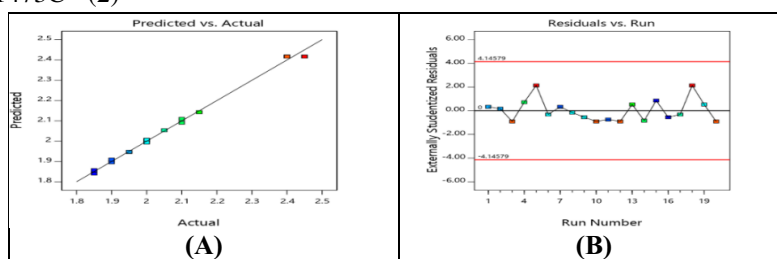


Figure 1 (A) Normal plot of Residuals with Run number (B) Comparison between actual values and predicted values

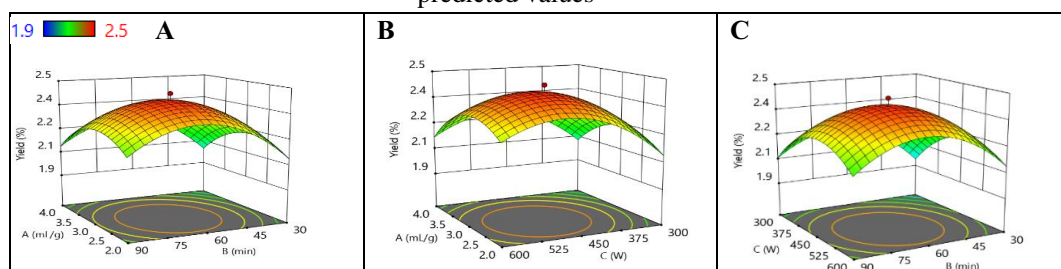


Figure 2. 3D models of the interaction relationship between Yield and factor A (water and turmeric ratio), B (extraction time), C (microwave power)

Based on the optimized parameters, Figure 2 shows the mutual interactions of the factors and the yield of the lemon oil obtained. In general, the efficiency of the attained oil increases proportionally with condition parameters. However, as these conditions exceed the optimal point (2.80:1 mL/g, 63.29 min, and 477.82 W); the obtained lemon oil content ceases to rise, and eventually, starts diminishing. The parameters that DX11 software predicted were made to extract lemon essential oil and the results are shown in Table 4. With that result, the DX11 software predicted accurate results in comparison with empirical values.

Table 4. Compare lemon essential oil yield results between DX11 software and actual experiments

	A (mL/g)	B (min)	C (W)	Yield (%)
DX11 software	2.80:1	63.29	477.82	2.472

Experiment	2.80:1	63.30	478.00	2.500
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4. Conclusion

Response surface methodology has demonstrated to be useful in determining the result of three independent variables including extraction time, microwave power and plant material to solvent ratio on the lemon essential oils yield and for anticipating the optimal operational conditions. To conclude, the parameters that affect the extraction of the lemon extract using microwaves during extraction were optimized by the RSM method. The yield of lemon oil is at 2.472% with 96.2% confidence when the water-to-material ratio reached 2.80:1 mL/g, at the extraction time of 63.29 min and microwave power of 477.82 W. Through the agreement of the experimental and the analysis of variance and anticipated results, it can be assumed that the generated model was suitable for the simulation of microwave-assisted extraction of lemon with any combination of tested variables.

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