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Using soft computing approaches for orange (*Citrus nobilis* Lour. var. *nobilis*) oils extraction process

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Abstract. Orange oil with many applications in food and beautify people. In this sense, traditional methods of extracting essential oils have been less effective and gradually replaced by more optimal methods. The combination of microwave assisted hydro-distillation and treated with response surface methodology are considered as a new and modern method for extracting oranges and optimizing the factors that influence the extraction process. The results showed that influencing factors such as the ratio of water to the raw materials were 3.2: 1 (mL/g), the microwave power used at 465 (W) for the extraction time was 96.63 (min) for the essential oil yield of 4.0 (%) with 99.5 % reliability.

1. Introduction

Essential oils (EOs) are a blend of many volatile components, with characteristic odors ancillary on the source of the crude materials supplying essential oils, the greatest part which are derived from plants. Although essential oils have been fully developed in distillation technology since the early 13th century, however, the demand for essential oil in the world was impacted by many factors in last 50 years. The demand for materials for spices and perfumes naturally increases as the population grows, especially for food, beverage and beauty products because of their antimicrobial and antioxidant properties [1, 2]. The problem of food safety (which does not contain harmful substances in the immediate and long term) has made consumers more inclined to consume natural foods, in there, oranges oil is also of attention.

Citrus nobilis Lour. var. *nobilis* is the scientific name of the orange species grown mainly in the Mekong Delta of Vietnam. The leaves, flowers, and peels of oranges are components that can be extracted into essential oils (0.95%). Orange oil is extracted by various extraction methods such as mechanical methods: squeeze, press; Traditional methods: extraction with solvent; distillation with water; Modern methods: extraction by assistance of microwave or with a carbon dioxide solvent, in which, the microwave assisted hydro-distillation is believed to shorten the distillation time, both the composition and the yield of essential oils are significantly improved [3-5]. Orange essential oil contain mainly D-limonene, which is the main compound accountable used to deodorize the air, reduce stress, clear nasal decongestants, anti-aging, and detoxifying properties because of their high antioxidant and antimicrobial properties [6-9].



Response surface methodology (RSM) is a soft computing approaches, which is known as a tool for optimizing the effects of factors that occur in chemical reactions. RSM makes the survey not only easy but also effective because it minimizes the number of experiments performed, but still meet the full criteria to evaluate the optimal efficiency of the process. The researches have applied RSM in the optimization process can be included as extraction of essential oil from pomegranate (*Punica granatum* L.) [10], Neem Seed Oil Extraction [11], gmelina seed oil [12], and many other surveys for the optimization process.

In this way, the extraction of the orange oil has a combination of microwave assisted hydro-distillation and the use of RSM for optimization has never been studied. Thus, in this study, factors such as time for extraction, orange peels and water ratio, and microwave power were optimized by RSM, which is thought to affect the yield of orange oil obtained by microwave assisted hydro-distillation method.

2. Materials and method

2.1. Plant Samples

Orange fruit is purchased in Ho Chi Minh City, Vietnam. After that, the orange is washed several times with water, the orange peel is retained and kept in a non-hygroscopic bag. Last, weigh 100g orange peel and continue to cut to about 2 mm by cut accessories (Sunhouse SHD5322, 220W, Vietnam).

2.2. Extraction Method

The material is extracted to obtain the essential oil by the extraction system. The system consists of a microwave oven MW71E manufactured by SAMSUNG (Vietnam), which is considered as a source of heat for the extraction process. On top of that, the system is connected to the hydro-distillation apparatus (Clevenger type apparatus, Germany) used to condense and separate the extracted oil and water phase. Finally, the essential oil is anhydrous sodium sulfate (99%, Sigma Aldrich).

2.3. Experimental design with RSM

The response surface methodology was chosen to optimize the effect factors of orange oil on the effect of the ratio of water and orange peel (A), extraction time (B), and microwave energy (C) to maximize essential oil yield. The central composite design (CCD) offers the experiment matrix designs, and the response variable is yield of orange oil obtained (Y), which were displayed in Table 1. The experiment matrix designs were conducted by parameter A was set in range of 2:1–4:1 mL/g, B was set from 60 to 120 min, and C was fixed in range of 300–600 W. For the statistical dissect, Design-Expert® software version 11, Minneapolis was practiced to assumed 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	60	300
Central point (0)	3	90	450
Maximum point (+1)	4	120	600

The yield of orange oil obtained (%) is calculated by quotient of the volume of attained oil (V, ml) and the amount of orange peel originally used for the experiment (m, g) were presented as in equation (1):

$$Y = \frac{V}{m} \times 100 \quad (1)$$

3. Results and discussion

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

	Independent variables			Y (%)		Independent variables			Y (%)		
	A	B	C	Actual	Predicted	A	B	C	Actual	Predicted	
1	2	60	300	2.8	2.81	11	3	40	450	3.0	2.98
2	4	60	300	3.2	3.21	12	3	140	450	3.4	3.39
3	2	120	300	3.0	3.01	13	3	90	198	3.1	3.09
4	4	120	300	3.3	3.3	14	3	90	702	3.3	3.27
5	2	60	600	2.9	2.92	15	3	90	450	4.0	3.97
6	4	60	600	3.1	3.12	16	3	90	450	4.0	3.97
7	2	120	600	3.3	3.32	17	3	90	450	3.9	3.97
8	4	120	600	3.4	3.41	18	3	90	450	4.0	3.97
9	1.3	90	450	3.0	2.98	19	3	90	450	3.9	3.97
10	4.7	90	450	3.4	3.39	20	3	90	450	4.0	3.97

A total of 20 experiments were carried out to estimate the response surface the orange oil yield such as eight factorial points, six axial points, and six center points. The response variable is that the orange oil yield Y depends on the independent variables realize through the experiments is indicated in Table 2. The results displayed that there is an contact between factors orange oils yield. To determine which of the effects in the model are statistically significant, the p-value of regression was significant with a commonly used α -level being 1%, whereas the lack of fit was not significant at the 5% α -level only for the quadratic model described by the following as in equation (2):

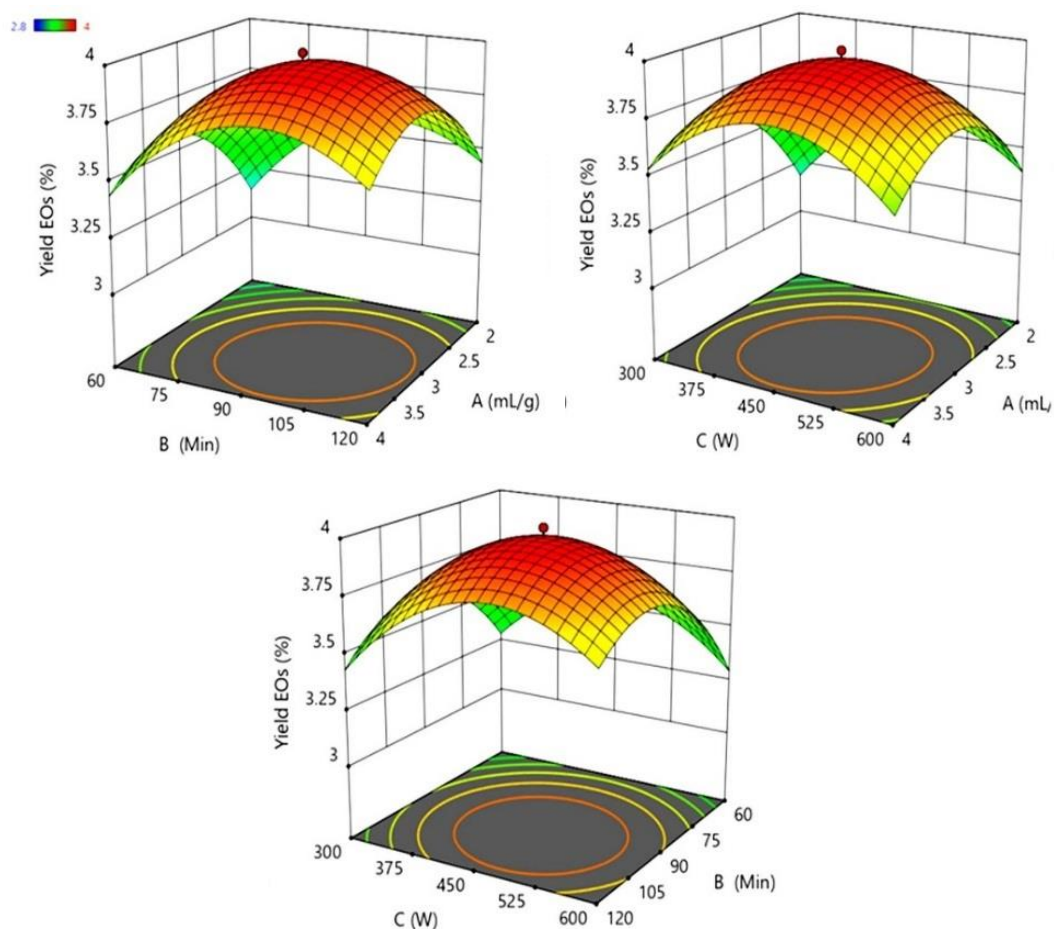
$$Y = 3.97 + 0.1225A + 0.1225B + 0.0539C - 0.0250AB - 0.05AC + 0.05BC - 0.2771A^2 - 0.2771B^2 - 0.2771C^2 \quad (2)$$

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients. Figure 1 shows a 3D model of the relationship between the attained orange oil yield and three independent factors water and orange peels ratio (A), time extraction (B), and power microwave (C). By that we see that not only A or B, but also when C increased the yield of the oil increased. However, if it overcome 4.0 point which has been optimized by DE11, the factors that affect the process are ineffective and result in reduced yield of the essential oil. Optimization results are given with the orange oil yield of 3.996% with A = 3.2:1 (mL/G), B = 96.63 (min), C = 464.75 (W) for desirability of 99.70%.

In Table 3, the model is significant because the model F-value implies 220.20 value. There is only a 0.01% chance that an F-value this large could occur due to noise. In addition, model terms are significant base on P-values less than 5% and if this values greater than 1% indicate the model terms are not significant. Analytical results showed that the factors interacting with the essential oil function with significance level $R^2 = 0.9831$ and confidence level was 95% as can be seen in Table 3. If there are many not significant model terms, the model is not good and the model needs to be improved. The Lack of Fit F-value of 0.24 implies the Lack of Fit is not significant relative to the pure error. There is a 93.10% chance that a Lack of Fit F-value this large could occur due to noise. In addition, as observed in Figure 2, the high correlation coefficient represents a high secure of the actual and predicted values because most of the points are closely aligned with the straight line. This has proven that the results of the investigations during the extraction process are accurate and the optimization is highly effective.

Table 3. ANOVA for Quadratic model.

Source	Sum of Squares	Degree of freedom	Mean Square	F-value	Prob. > F		Comment
Model	3.26	9	0.3626	220.2	< 0.0001	significant	SD = 0.0406 Mean = 3.40 CV (%) = 1.19 R ² = 0.995 AP = 40.2545
A	0.2049	1	0.2049	124.41	< 0.0001		
B	0.2049	1	0.2049	124.41	< 0.0001		
C	0.0397	1	0.0397	24.11	0.0006		
AB	0.005	1	0.005	3.04	0.112		
AC	0.02	1	0.02	12.14	0.0059		
BC	0.02	1	0.02	12.14	0.0059		
A ²	1.11	1	1.11	671.81	< 0.0001		
B ²	1.11	1	1.11	671.81	< 0.0001		
C ²	1.11	1	1.11	671.81	< 0.0001		
Residual	0.0165	10	0.0016				
Lack of Fit	0.0031	5	0.0006	0.2351	0.931	not significant	
Pure Error	0.0133	5	0.0027				

**Figure 1.** A 3D model of the interaction relationship between Yield EOs and factor A (water and orange peels ratio), B (extraction time), C (microwave energy).

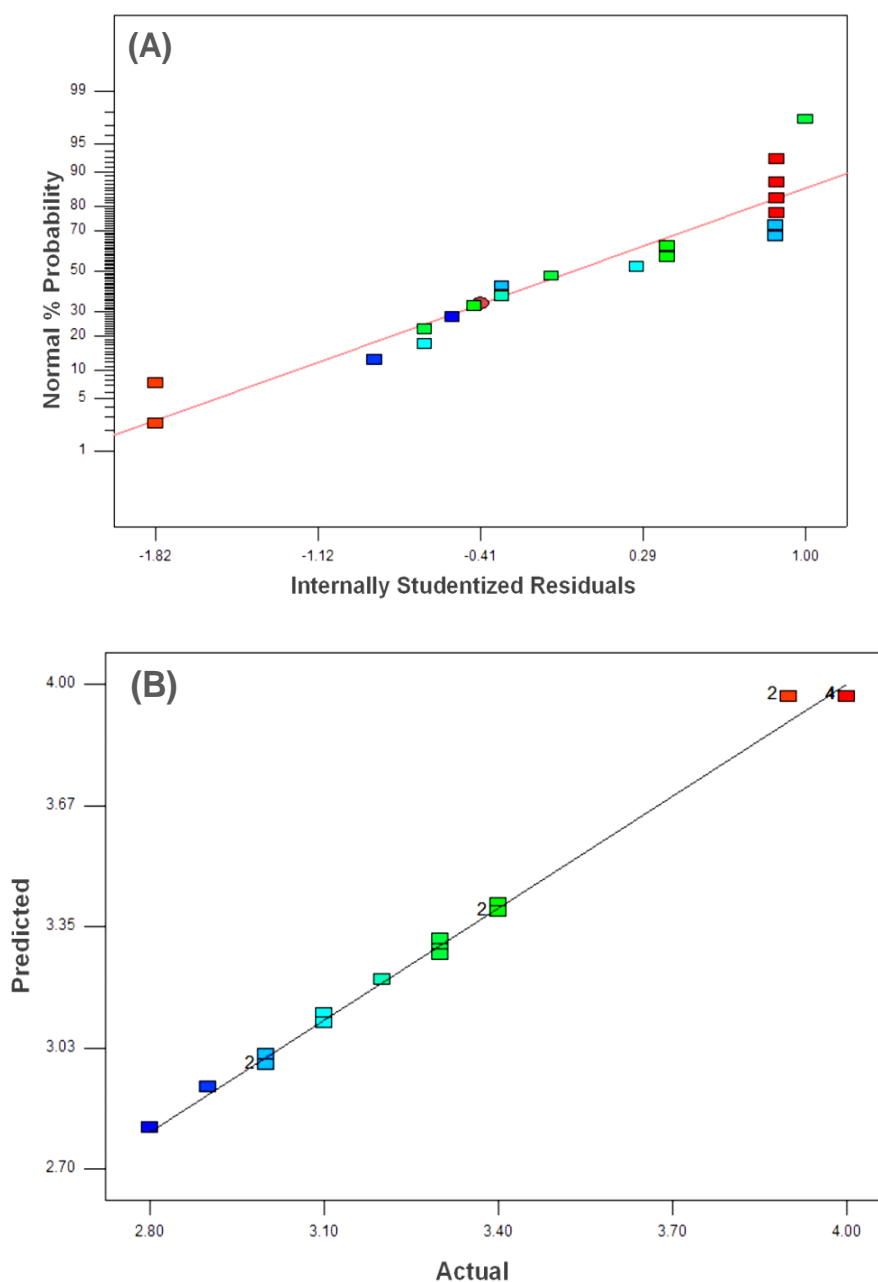


Figure 2. (A) Normal plot of Residuals (B) Comparison between actual values and predicted values.

4. Conclusions

Under the optimization performed by the response surface methodology, the conditions for extraction of the orange oil using microwave oven heat have been suggested as follows: the ratio of water and orange peel is 3.2: 1; extraction time at 96.63 min, and microwave power for this process is 464.75W with yield of 3.996%. RSM and microwave assisted hydro-distillation showed not only the convenience of the experiments, but also the optimization of the three parameters in detail, which made the yield of orange oil obtained more efficiently both quantity and quality.

Acknowledgments

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