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Green technology to optimize the extraction process of turmeric (*Curcuma longa L.*) oils

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Abstract. Turmeric oil has many uses in the field of medicine and food. There are many methods of extracting essential oils from plants. However, every traditional method has limitation regarding the heat transfer, time or quality of the oil obtained. In this study, the combination of microwave extraction and response surface methodology are used to extract turmeric oil and optimize the factors that influence the extraction process. The results showed that optimal influencing factors such as the ratio of water to the raw materials were 2.99:1 (mL/g), the microwave power used at 456.60 (W) for the extraction time was 102.43 (min) for the essential oil yield of 0.95 (%) with 100 % reliability

1. Introduction

In the current stage, green technology has always been the subject of many studies [1,2]. A particular attraction in this field is microwave extraction because of its ability to transfer energy to the material, its environmental-friendliness and most importantly, the ability to improve the quality and quantity of essential oils, as demonstrated by many studies [3-5]. However, to improve the yield in the extraction of essential oil, not only does the choice of extraction method matter, but it is also necessary to consider the optimization of the influencing conditions. In the present time, the application of computer software technology in the research process has brought about remarkable results, gradually replacing traditional methods. One of the methods used to optimize the conditions that affect the extraction of essential oil is the response surface methodology. Response surface methodology (RSM) is a method used to simultaneously optimize two or more factors affecting the experiment process. Compared with conventional methods, RSM is also capable of designing experiments. The designed experiments will consists of 12 or 20 experiments for optimization of 2 and 3 factors affecting the extraction process, respectively. In addition, RSM also facilitates the experiment detection and manipulation. Finally, with the simultaneous optimization of multiple factors, RSM results in the equations for prediction, and 3D models that describe the interaction of conditions on the response



factor. Because of these superior facets, response surface methodology has been widely utilized in many research areas. A few examples include studies regarding adsorption of toxic ions in the waste water, or in the extraction of some compounds in herbs such as essential oils, anthocyanin. Therefore, RSM is considered as an economical and efficient direction in producing empirical research results [6-13].

Curcuma longa L., commonly referred to as turmeric, is a plant which grows wild in the forests of South Asia and Southeast Asia, especially the south of India [14]. All parts of the turmeric are used for various purposes. The rhizomes are used in most fields, because of their remarkable biological properties and characteristic color and odor [15,16]. The leaves of turmeric act as protective membranes that cover food during cooking. Turmeric is split into two components including volatile and non-volatile compounds. The volatile compounds are compounds that are extracted from raw materials such as essential oils. Non-volatile compounds, for example turmeric powder, are often used as spices in food [17,18]. In this study, the content of volatile substances was emphasized through the extraction of essential oils from the rhizome. Tr-Turmerone, Turmerone, Tr-Curcumin, and Curlone are high-content components found in turmeric oils [19], which contribute to antioxidant and antimicrobial properties of the turmeric oil [20,21]. However, the content of these components may vary depending not only on the source but also on the extraction method. Therefore, the combination of microwave extraction with experimental design by RSM is considered as the efficient method for extracting essential oil from the rhizome of turmeric.

The aim of this work was to study the essential oil extraction process with microwave and to optimize the conditions that affect the extraction of turmeric oil by response surface methodology. Three conditions are examined in this study which are: the solvent to raw material ratio, extraction time and microwave power.

2. Materials and method

2.1. Plant Samples

The rhizome of turmeric is purchased in Ho Chi Minh City, Vietnam. After that, it is washed several times with water, the turmeric is retained and kept in a non-hygroscopic bag. Lastly, 50g of rhizomes are cut into 1-2 mm portions by cutting accessories (Sunhouse SHD5322, 220W, Vietnam).

2.2. Extraction Method

The material is extracted by an extraction system to obtain the essential oil. The system consists of a microwave oven MW71E manufactured by SAMSUNG (Vietnam), acting as a source of heat for the extraction process. The oven 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 collected after using anhydrous sodium sulfate (Na_2SO_4 , Sigma Aldrich).

2.3. Experimental design with RSM

With the aim of maximizing the yield of the turmeric oil, the response surface methodology was selected to optimize the factors affecting the yield of the oil. Three considered factors are the ratio of water and rhizomes of turmeric (Z_1), extraction time (Z_2), and microwave energy (Z_3). The central composite design (CCD) offers the experiment matrix designs, and the response variable is yield of oil obtained (Y), which were specified in Table 1. The experiment matrix designs were conducted by setting Z_1 , Z_2 , Z_3 in range of 2:1–4:1 mL/g, 60 to 120 min, and 300–600W, respectively. Design-Expert® software version 11, Minneapolis was used to conduct the experimental design and to analyze the data afterwards.

Table 1. Encodes of the values of the optimization factors.

Levels	Independent factors		
	Z ₁ (mL/g)	Z ₂ (Min)	Z ₃ (W)
Minimum point (-1)	2:1	60	300
Central point (0)	3:1	90	450
Maximum point (+1)	4:1	120	600

The yield of turmeric oil obtained (%) is calculated by following equation:

$$\text{Yield of turmeric oil (\%)} = \frac{\text{the volume of turmeric oil obtained (mL)}}{\text{the amount of turmeric originally used for the experiment (g)}}$$

3. Results and discussion

The results were analysed using Analysis of Variance (ANOVA). All results of the 20 experiments are presented in Table 2 corresponding to four minimum points, four maximum points, six axial points, and six center points. The actual yield of turmeric oil was compared with the predicted results of RSM software showing no significant difference. Statistically significant effects in the model were recognized for variables with the p-value lower than 1%. The lack of fit was insignificant at the 5% α -level only for the quadratic model described by the following equation (2):

$$\text{Yield of turmeric oil} = 0.9419 + 0.0208Z_1 + 0.0478Z_2 + 0.0062Z_3 - 0.0500Z_1Z_2 - 0.0250Z_1Z_3 - 0.0250Z_2Z_3 - 0.0605Z_1^2 - 0.0605Z_2^2 - 0.0428Z_3^2 \quad (2)$$

For any given set of levels of factors, a response for yield can be predicted by the above equation. By comparing factor coefficients, relative impact of the factor could be identified using the equation. By default, the high levels and low levels of the factors are coded as +1 and -1, respectively.

The Table 3 displayed results of ANOVA for Quadratic model for the process of extracting rhizome turmeric 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 57.75 implies that the model is significant and there is only a 0.01% chance that an F-value of this large could occur due to noise. P-values of less than 0.05 indicated that model terms are significant except for Z₃ whose p-value is greater than 0.1. The positive effect of water-to-material ratio on the yield can be explained by the increased contact surface between the material matrix and the solvent. To be specific, extra solvent caused microwave energy to be absorbed more efficiently by the material, which in turn enhances the swelling of the rhizome and extends the contact area. For extraction time, the positive relationship with oil yield can be explained by the fact that thermal accumulation, caused by prolonged exposure to microwave energy, facilitates the breaking down of cell wall, releasing more essential oils into the solution. If there exist many insignificant model terms (excluding those required to support hierarchy), an improved model may be produced by reducing model terms. Regarding the Lack of Fit, the F-value of 0.65 implies that the Lack of Fit is not significant relative to the pure error and there is a 67.66% chance that a Lack of Fit F-value of this large could occur due to noise. Therefore, no further specification of the model is required and it can be asserted that the yield of the extraction model produced by Design-Expert® software is fixed and suitable. In addition, the experimental model was considered reasonably fit when calculated residuals follow a random pattern as shown in Figure 1.A. Figure 1.B demonstrated that data points corresponding to predicted and actual values are scattered across the 45 degree line with close proximity, suggesting that the actual results are accurately predicted from the factor values. Thus, based on the data analysis of the oil yield from experiments, the RSM software results in optimized parameters as Z₁ = 2.99:1 (mL/g), Z₂ = 102.43 (min), and Z₃ = 456.60 (W) to obtain the yield of 0.951% with 100% reliability. This yield is in line with the oil content range (min: 0.05; max: 1.40; mean: 0.57) obtained from turmeric rhizomes of Bahl *et al.* (2014) who collected 84 accessions from various geographical locations in India to find various ranges of variation of oil yield [22]. Manzan *et al.* (2002) also found a similar yields of essential oil (0.45

wt %) and pigment (0.16 wt %) comprising curcumin, demethoxycurcumin, and bisdemethoxycurcumin in the condition of autoclave pressure of 1.0×10^5 Pa and steam distillation time of 2h [23]. On the other hand, Raina *et al.* (2002) performed hydro –distillation of leaves and rhizomes and obtained 2.2% oil yield consisting of more than 80 compounds [24].

Based on the optimized parameters, Figure 2 shows the mutual interactions of the factors and interaction of factors with the yield of the turmeric oil obtained. In general, the efficiency of the attained oil increases proportionally with condition parameters. However, as these conditions exceed the optimal point (2.99:1 mL/g, 102.43 min, and 456.60 W); the obtained turmeric oil content ceases to rise, and eventually, starts diminishing.

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

	Independent variables			Y (%)			Independent variables			Y (%)	
	Z ₁	Z ₂	Z ₃	Actual	Predicted		Z ₁	Z ₂	Z ₃	Actual	Predicted
1	2	60	300	0.60	0.60	11	3	40	450	0.70	0.69
2	4	60	300	0.80	0.79	12	3	140	450	0.85	0.85
3	2	120	300	0.85	0.85	13	3	90	198	0.80	0.81
4	4	120	300	0.85	0.84	14	3	90	702	0.85	0.83
5	2	60	600	0.70	0.72	15	3	90	450	0.95	0.94
6	4	60	600	0.80	0.81	16	3	90	450	0.95	0.94
7	2	120	600	0.85	0.86	17	3	90	450	0.95	0.94
8	4	120	600	0.75	0.75	18	3	90	450	0.95	0.94
9	1.3	90	450	0.75	0.76	19	3	90	450	0.90	0.94
10	4.7	90	450	0.8	0.81	20	3	90	450	0.95	0.94

Table 3. ANOVA for Quadratic model

Source	Sum of Squares	Degree of freedom	Mean Square	F-value	Prob. > F	Comment
Model	0.1786	9	0.0198	57.75	< 0.0001	significant
Z ₁	0.0059	1	0.0059	17.20	0.0020	
Z ₂	0.0312	1	0.0312	90.68	< 0.0001	
Z ₃	0.0005	1	0.0005	1.51	0.2477	
Z ₁ Z ₂	0.0200	1	0.0200	58.21	< 0.0001	
Z ₁ Z ₃	0.0050	1	0.0050	14.55	0.0034	
Z ₂ Z ₃	0.0050	1	0.0050	14.55	0.0034	
Z ₁ ²	0.0528	1	0.0528	153.65	< 0.0001	
Z ₂ ²	0.0528	1	0.0528	153.65	< 0.0001	
Z ₃ ²	0.0265	1	0.0265	77.00	< 0.0001	
Residual	0.0034	10	0.0003			not significant
Lack of Fit	0.0014	5	0.0003	0.6491	0.6766	
Pure Error	0.0021	5	0.0004			
Std. Dev.= 0.0185		Mean = 0.8300		C.V. % = 2.23		R² = 0.9811
Adjusted R²= 0.9641		Predicted R² = 0.9271		Adeq Precision = 25.8352		

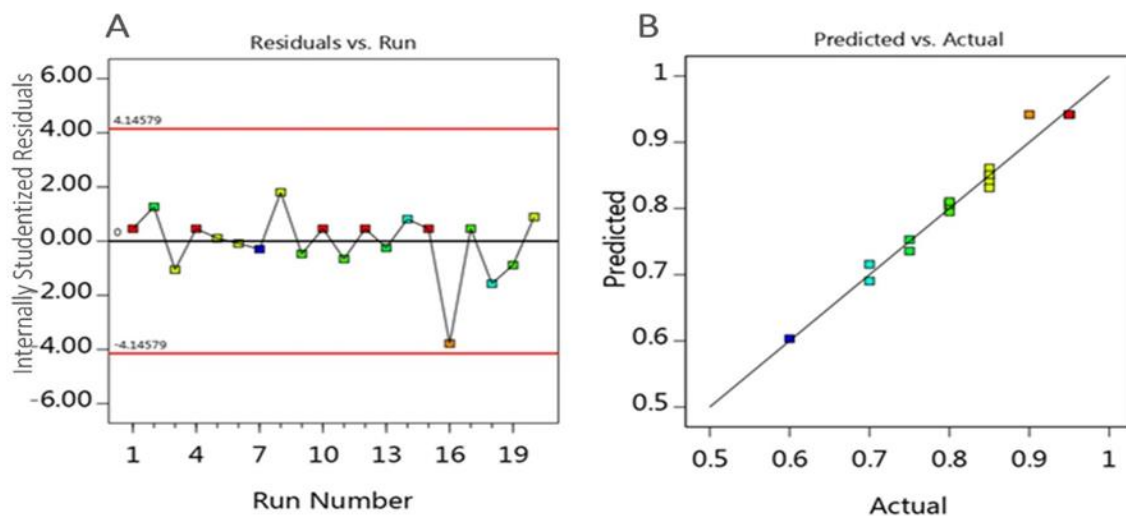


Figure 1. (A) Normal plot of Residuals with Run number (B) Comparison between actual values and predicted values.

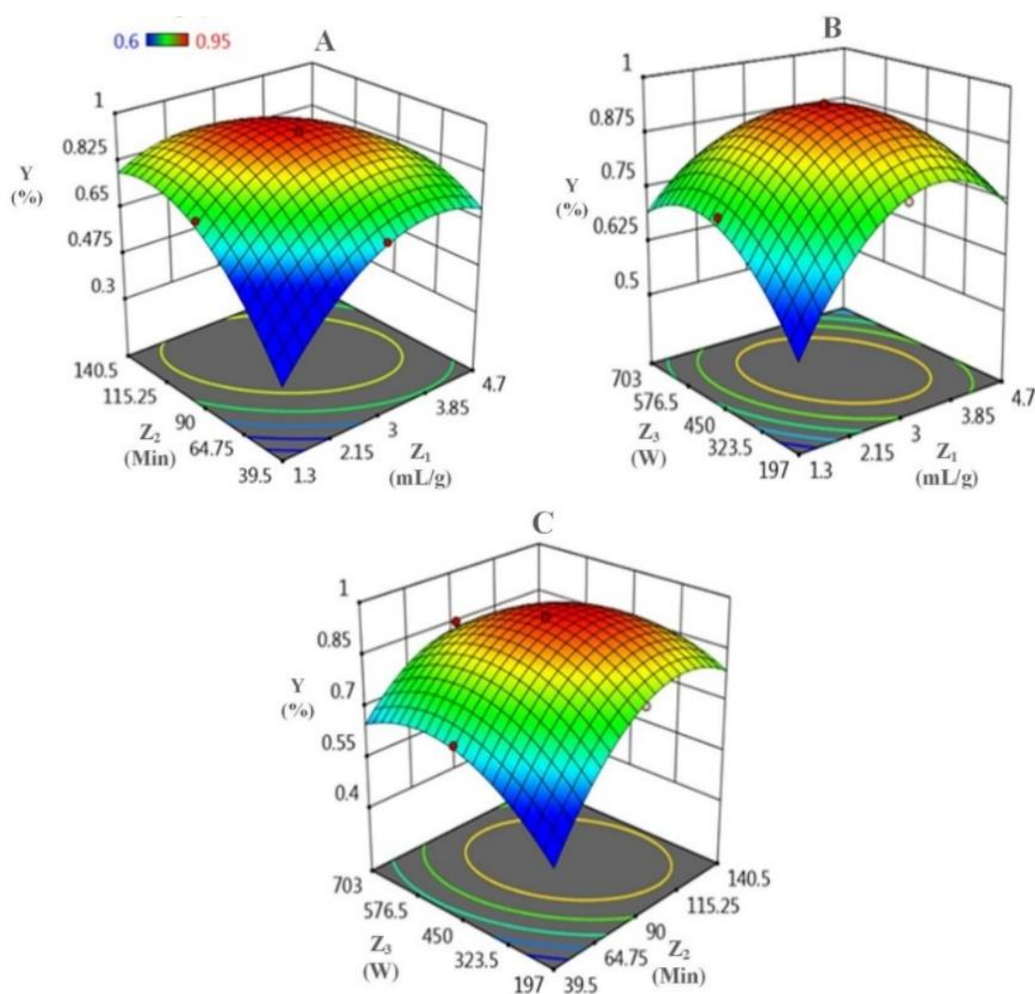


Figure 2. 3D models of the interaction relationship between Yield and factor Z_1 (water and turmeric ratio), Z_2 (extraction time), Z_3 (microwave energy).

4. Conclusions

To conclude, the parameters that affect the extraction of the turmeric extract using microwaves during extraction were optimized by the RSM method. The yield of turmeric oil is at 0.95% with 100% confidence when the water-to-material ratio reached 2.99:1 mL/g, at the extraction time of 102.43 min and microwave power of 456.60 W. The combination of RSM and microwave assisted extraction showed not only the convenience, but also the ability to optimize condition parameters, which made the yield of turmeric oil obtained more efficiently both terms of both quantity and quality.

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