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MODELING AND OPTIMIZATION OF THE ORANGE LEAVES OIL EXTRACTION PROCESS BY MICROWAVE-ASSISTED HYDRO-DISTILLATION: THE RESPONSE SURFACE METHOD BASED ON THE CENTRAL COMPOSITE APPROACH (RSM-CCD MODEL)

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ABSTRACT

Although being a by-product after the harvest, orange leaves could be used to produce essential oil through extraction. Application of the essential oil extracted from orange leaves is diverse ranging from food flavoring to cosmetics. This study aimed to develop optimal conditions for microwave assisted hydro-distillation of essential oil from orange leaves. The selected optimization method is Response Surface Methodology in conjunction with the central composite experiment design. The factors that were varied for the production of the orange leaves oil extraction were material-to-water ratio, extraction time, and microwave power. Accordingly, a statistical model was established and Analysis of variance (ANOVA) was carried out to identify the set of factors that gives the highest essential oil yield. Optimization results revealed optimal conditions as follows, material and water ratio of 3.46:1 (mL/g), extraction time of 100.47 min and operating power of 471.58 W. These conditions correspond to the essential oil yield of 0.43% with 92.1 % reliability. In addition, we also analyze the produced essential oils by gas chromatography-mass spectrometry (GC-MS). The GC-MS results revealed that major components of essential oil were Sabinene (30.556 %), Cis-Ocimene (10.139 %), and D-Limonene (9.682 %).

Keywords: Orange Leaves Oil, Microwave-assisted Hydro-distillation, Response Surface Methodology, GC-MS.

INTRODUCTION

Extraction technology plays a crucial role in the sustainability of the agro-food industry and the processing industry [1-4]. Nowadays, consumers tend to use products of natural origin which is health-beneficial and causes no side effects when taken accordingly.

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One of the components used in the production of such commodities is essential oils. Essential oils are valuable products composed of volatile substances. The oils are often isolated by various methods from plant organs or botanical species such as flowers, leaves, twigs, and seeds. Essential oils extracted from aromatic plants are often commercialized as export commodities and utilized in fragrance, cosmetics, pharmaceuticals and beverage industry. Popular products containing essential oils are air fresheners and deodorizers [5-10]. In medicine, almost all branches of medicine such as pharmacy, balneology, massage, and homeopathy recognized essential oils as important ingredients for drug production and popular components for various therapies and treatments.

Citrus fruits, similar to coffee and tea, are important goods for international trade and are widely cultivated globally. A significant proportion (60%) of produced citrus are oranges. Orange has its origin in South-East Asia and it is the most widely used species of citrus fruits there. Orange constitutes a wide range of vitamins, especially vitamin C, and is a rich source of flavonoids, terpenes, potassium and calcium [11-14]. Among these constituents, flavonoids have been utilized to produce health supplements and recently, are found to exhibit hypolipidemic and inhibitory effects in cancer cells. In the cosmetics industry, the orange essential oil is used to aromatize products such as fragrance and creams. In the food industry, orange essential oil gained popularity due to its antimicrobial effect against bacteria and fungi. Other applications of the orange essential oil could include a solvent for extraction of fats and oils from an olive.

Recently, major technological and economic obstacles have hindered the development of extraction techniques [15-18]. Such bottlenecks could be more expensive energy, strict law on emission and/or requirement in safety control. Traditionally, oil extraction processes include pressing, solvent extraction, and different distillation techniques where heat is involved with temperature ranging from 130 to 150°C. However, such techniques have various shortcomings including low oil yield, high toxicity stemming from hazardous solvents and extended extraction duration leading to increased costs [19]. To contribute to the environmental preservation and to enhance production efficiency, green techniques for extraction of oil from bio-products have been developed. Microwave-assisted extraction has been one of such technologies and is widely accepted in various industries due to its ability to reduce extraction time and to increase yield quantity and quality [20-26]. Due to electromagnetic waves with frequency ranging from 300MHz to 300GHz, polar molecules in the biomaterial are rapidly rotated, in turn generating heat in the interior of the material. The main advantage of microwave extraction is that it is capable of breaking cell walls and oil sacs, quickly freeing oil and constituents inside to the outside solvent medium. Therefore, the extraction efficiency could be improved.

Operating conditions in the extraction process have been investigated individually with respect to the production of essential oils. However, this approach is inefficient in terms of time and costs since the interaction between conditions is not taken into account and numerous experimental attempts are required. RSM is an optimization technique devised to overcome these disadvantages [27,28]. The method aims to describe a desired response or an outcome of interest with respect to a set of process variables through the use of statistical techniques. Benefits of RSM are numerous. In addition to readily available, efficient and simple experimental designs for the method [29-44]. RSM could also reduce the number of experiment trials and solve issues related to linear and non-linear multivariate regression.

The objective of the current study is to maximize the amount of extracted essential oil orange leaves. The method of extraction is microwave-assisted hydro-distillation method and the process is optimized by RSM. We considered variables that are relevant and useful to the possible up-scale process including material and water ratio, extraction time, microwave power and efficiency. The responses were the measured yield of essential oil. A statistical model was established to model extraction conditions and levels of experimental conditions were determined by central composite design (CCD). ANOVA analysis was adopted to assess the effect of the process variables on both of the responses. Optimal yields of essential oil were then predicted and experimentally verified.

EXPERIMENTAL

Materials and Chemicals

Orange leaves were taken from local markets in Vietnam. The material was washed several times with water to remove impurities and allowed to dry naturally. Then a grinder (Sunhouse, about 2-3mm) was used

to grind the material. Finally, the material was placed in a Clevenger type apparatus, connected to a domestic microwave oven (SAMSUNG MW71E) for microwave assisted hydro-distillation (MAHD) operation for extraction of essential oil as described in Fig.-1 and Fig.-2.
Anhydrous sodium sulfate (Na2SO4) was purchased from Sigma Aldrich (US). Deionized water produced by Milli-Q purification system (Millipore, USA) was used as a solvent to extract orange leaves oil.

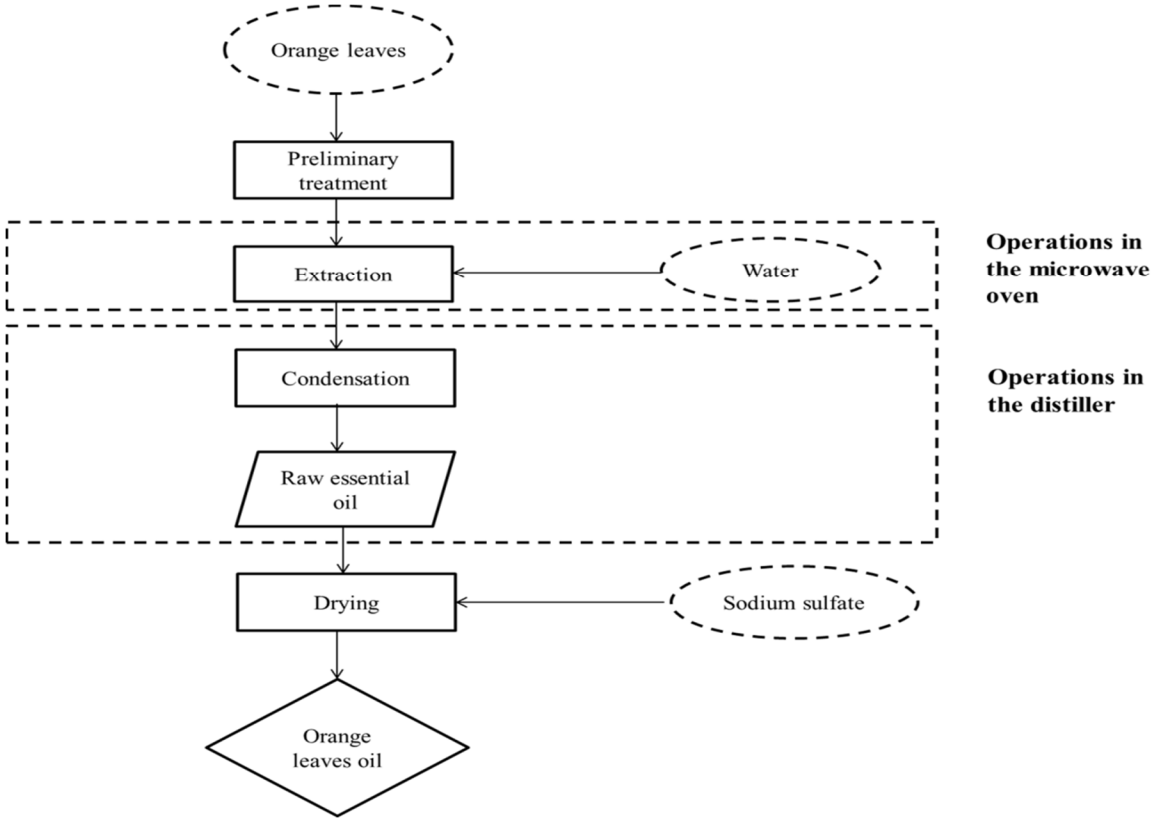

Fig.-1: Diagram of the Orange Leaves Oil Extraction Process

Experimental Design with RSM

To optimize factors influencing the hydro-distillation process, the response surface methodology was adopted to maximize essential oil yield. Considered factors include water and material ratio (A), extraction time (B), and microwave power (C). MAHD optimal code was determined following the central composite design, where the response variable and the experiment matrix designs were shown in Table-1. Design Expert software version 11 was employed to carry out ANOVA, regression, statistical tests and plotting. In order to verify the adequacy of the developed model, optimal conditions were verified by an actual experimental attempt.

Table-1: Independent Variables Matrix and their Encoded Levels for RSM Model.

Code	Name	Units	Levels				
			-α	-1	0	+1	+α
A	Material and water ratio	mL/g	1.3	2	3	4	4.7
B	Extraction time	Min	40	60	90	120	140
C	Microwave power	W	198	300	450	600	702

The yield of orange leaves oil extracted (Y) was calculated as follows to evaluate the performance of MAHD:

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

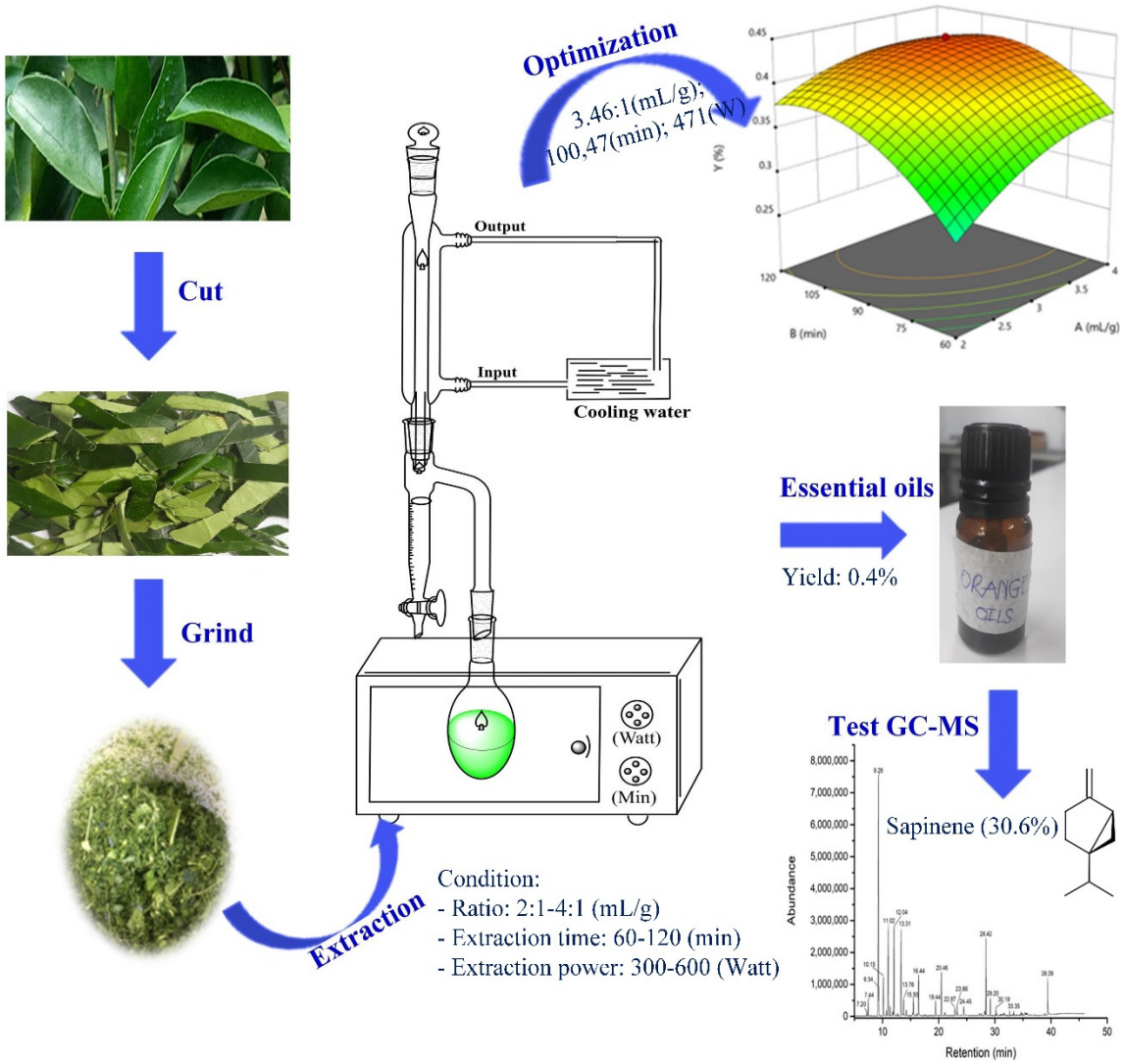

Fig.-2: The experimental process including preparation of orange leaves, microwave-assisted hydro-distillation unit and analysis of the obtained oil samples.

Analysis of Sample

Gas Chromatography-Mass Spectrometry (GC-MS) was used to analyze the composition of the essential oils of all extraction methods. 25 µL sample of essential oil in 1.0 mL n-hexane. Name of the equipment: GC Agilent 6890N, MS 5973 inert with HP5-MS column, head column pressure 9.3psi. GC-MS system operated at the following conditions: carrier gas He; flow rate 1.0 mL/min; split 1:100; injection volume 1.0 µL; injection temperature 250°C. Oven temperature progressed from an initial hold at 50°C for 2 min and a rise to 80°C at 2°C/min, and then to 150°C at 5oC/min, continue rising to 200°C at 10°C/min and rise to 300°C at 20°C/min for 5 min.

RESULTS AND DISCUSSION

Building Response Surface Model

Experimental results (20 experiments), produced by the design method of complex CCD center, and predictions by Design-Expert 11 are shown in Table-2. To be specific, 20 experiments including six axial points, six center points, and eight factorials, were devised and attempted to derive the input data for the approximation of response function. The experimental and predicted result of Table-2 suggested the impact of the three process factors on the yield. The estimated quadratic model is described as follows (2):

$Y = 0.4162 + 0.0270A + 0.0343B + 0.0270C - 0.0125AB - 0.025AC - 0.0125BC - 0.0205A^2 - 0.0382B^2 - 0.0382C^2$ (2).

The ANOVA results for the quadratic model of essential oil yield were summarized in Table-3. The main terms in the ANOVA table included: water and material ratio (A), microwave power level (B), extraction time (C), interaction terms (AB, BC, AC) and second-order effects (A^2, B^2, and C^2). Based on the F-value, it is suggested that the model was significant and the odds of noise that could cause such F-value is minimal, approximately 0.01%. The LOF F-value of 0.6782 is also desirable, implying that the LOF was not significant relative to the pure error and this experimental design model is suitable. In addition, the predicted R^2 of 0.7344 concurred with the adjusted R^2 of 0.9918. AP ratio was also greater than 4, which indicates signal adequacy. Therefore, this model could be used to navigate the design space.

Table-2: Box-Behnken Design and Observed Responses

S. No.	Experimental Parameters			Y (%)		
	A (Material and Water Ratio, mL/g)	B (Extraction Time, Min)	C (Microwave Power, W)	Actual	Predicted	Residual
1	2.0	60	300	0.20	0.1811	0.0189
2	4.0	60	300	0.30	0.3100	-0.0100
3	2.0	120	300	0.30	0.2996	0.0004
4	4.0	120	300	0.40	0.3785	0.0215
5	2.0	60	600	0.30	0.3100	-0.0100
6	4.0	60	600	0.35	0.3389	0.0111
7	2.0	120	600	0.40	0.3785	0.0215
8	4.0	120	600	0.35	0.3575	-0.0075
9	1.3	90	450	0.30	0.3128	-0.0128
10	4.7	90	450	0.40	0.4035	-0.0035
11	3.0	40	450	0.25	0.2505	-0.0005
12	3.0	140	450	0.35	0.3658	-0.0158
13	3.0	90	198	0.25	0.2628	-0.0128
14	3.0	90	702	0.35	0.3535	-0.0035
15	3.0	90	450	0.40	0.4162	-0.0162
16	3.0	90	450	0.40	0.4162	-0.0162
17	3.0	90	450	0.40	0.4162	-0.0162
18	3.0	90	450	0.40	0.4162	-0.0162
19	3.0	90	450	0.45	0.4162	-0.0162
20	3.0	90	450	0.45	0.4162	-0.0162

The yield of essential oil could be predicted using the above model. To validate the model, residuals of 20 runs and yields of oil were plotted in Fig.-3. Figure-3A plotted actual experiment yield values against predicted values. Visually, the distribution of data points follows the 45-degree line, indicating the consistency between the predicted value and the actual experimental value. Figure-3B indicated that the residuals of experimental yields clearly follow a random pattern. Figure-3A, which plotted predicted versus against actual values, also indicated close proximity of scattered data points to the 45-degree line, suggesting the reasonable predictive accuracy of the model and no violation of assumptions regarding the independence of variables and constant variance. Figure 3C depicted studentized residuals against corresponding probabilities. It is revealed that data points were almost on a straight line, suggesting no serious deviation and reasonable fit of the model.

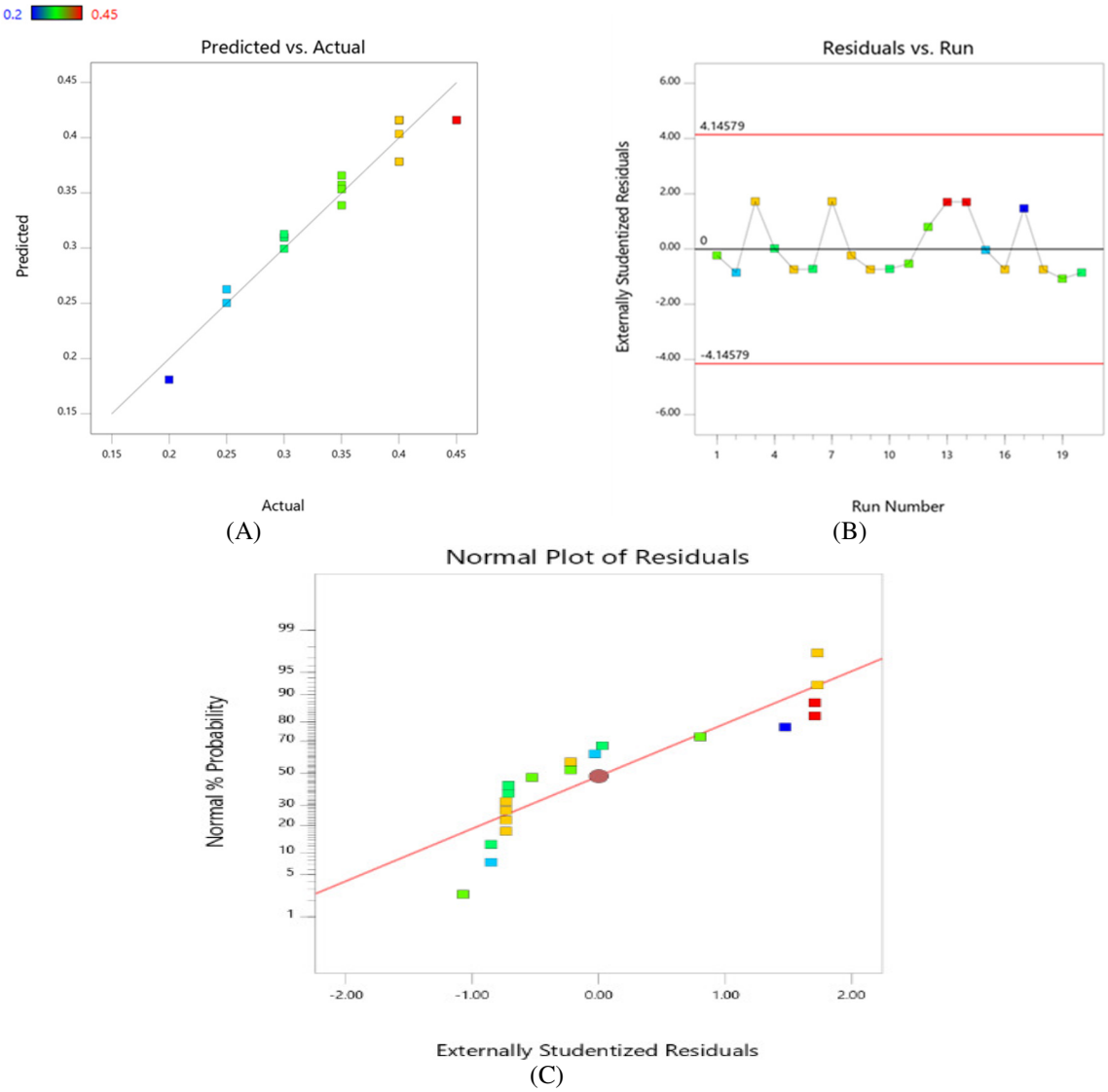

Fig.-3: Estimation of Model Precision (A) Comparison between Actual Values and Predicted Values and (B) Plot of Internally Studentized Residuals versus the Actual Run, and (C) The Normal % Probability Plot.

Table-3: ANOVA Results of the Response Function

Source	Sum of Squares	dF	Mean Square	F-Value	p-Value	Comment	
Model	0.0844	9	0.0094	16.77	< 0.0001	Significant	SD = 0.0237
A	0.0099	1	0.0099	17.74	0.0018	Significant	Mean = 0.3500
B	0.0160	1	0.0160	28.69	0.0003	Significant	CV (%) = 6.76
C	0.0099	1	0.0099	17.74	0.0018	Significant	R^2 = 0.9378
AB	0.0013	1	0.0013	2.23	0.1658		AP =14.0606
AC	0.0050	1	0.0050	8.94	0.0136	Significant	Adj R^2 =0.8819
BC	0.0013	1	0.0013	2.23	0.1658		Pred R^2= 0.7344
A^2	0.0061	1	0.0061	10.86	0.0081	Significant	

B^2	0.0210	1	0.0210	37.61	0.0001	Significant	
C^2	0.0210	1	0.0210	37.61	0.0001	Significant	
Residual	0.0056	10	0.0006				
Lack of Fit	0.0023	5	0.0005	0.6782	0.6598	Not Significant	
Pure Error	0.0033	5	0.0007				
Cor Total	0.0900	19					

Optimization of Experimental Procedures

The interaction effects of parameters on the response were demonstrated by three-axis response surfaces and two-axis plots. From Fig.-4, it is revealed that all three experimental parameters exerted significant influence on the yield of the Orange leaves oil extraction. In addition, the interactions between different functions (ratio water and raw materials and extraction time, ratio water and raw materials and microwave power, microwave power and extraction time) also exhibited very significant influence on the extraction yield. From Fig.-4, it could be observed that general trends of the three factors are similar. To be specific, an increase in any of the three factors induces oil yield to rise until oil yield reaches a certain point, where yield stops rising, and eventually, starts diminishing. Optimization results were calculated as: A= 3.46 (mL/g), B= 100.47 (min), and C= 471.58 (W) with desirability of 92.1%. These correspond with the orange leaves oil yield of 0.43%.

Validation of the Predictive Model

The data from Table-4 display the optimum conditions resulted from optimization. Accordingly, material and water ratio of 3.46:1 (mL/g), the time of 100.47 minutes and 471.58W operating power yielded the highest efficiency of 0.43%. This number approximates to the actual yield, conducted with almost identical conditions, of 0.4%. This result reaffirmed the validity of the model, suggesting that the model accurately predicted yield values. These results are in line with previous research results in which the yield of essential oils extracted from orange leaves ranged from 0.19-0.28% using steam distillation for 2h [45-46], and reached 0.23% for steam distillation for 5h [47]. Obviously, MAHD showed higher efficiency and shorter extraction time. More specific, the yield of orange leaves oil (0.43%) using MAHD was also higher than that of steam distillation (0.19-0.28%) with an extraction time of 100 min. These results confirmed the suitability MAHD when it comes to essential oil extraction from orange leaves.

Table-4: The Experimental Results using Optimum Condition Comparison with Predicted Results

	Material and Water Ratios (g/mL)	Extraction Time (min)	Microwave Power (W)	The Yield of Essential Oil (%)
Predicted	3.46	100.47	471.58	0.43
Actual	3.46	100	471	0.4

GC-MS Analysis Results

The chemical composition of orange leaves oil was presented together with the retention indices in Table-5 and Fig.-5. The GC-MS analysis identified 28 components in total. The major chemical compounds were Sabinene (30.556%) followed by Cis-Ocimene (10.139%), D-Limonene (9.682%), 3-Carene (9.102%), β-Elemenne (6.060%), Linalool (5.240%).

In a previous study [1], the aforementioned components were also found in the orange leaves oil, although in varying amounts. To be specific, previously recognized components were Sabinene (16.03%), 3-Carene (7.53%), and limonene (3.71%). It also showed that the number of components found in this study is higher than that in previous research. It is worth nothing that chemical composition of the essential oil could vary depending on geographical location and season of harvest, plant age and method extraction [48].

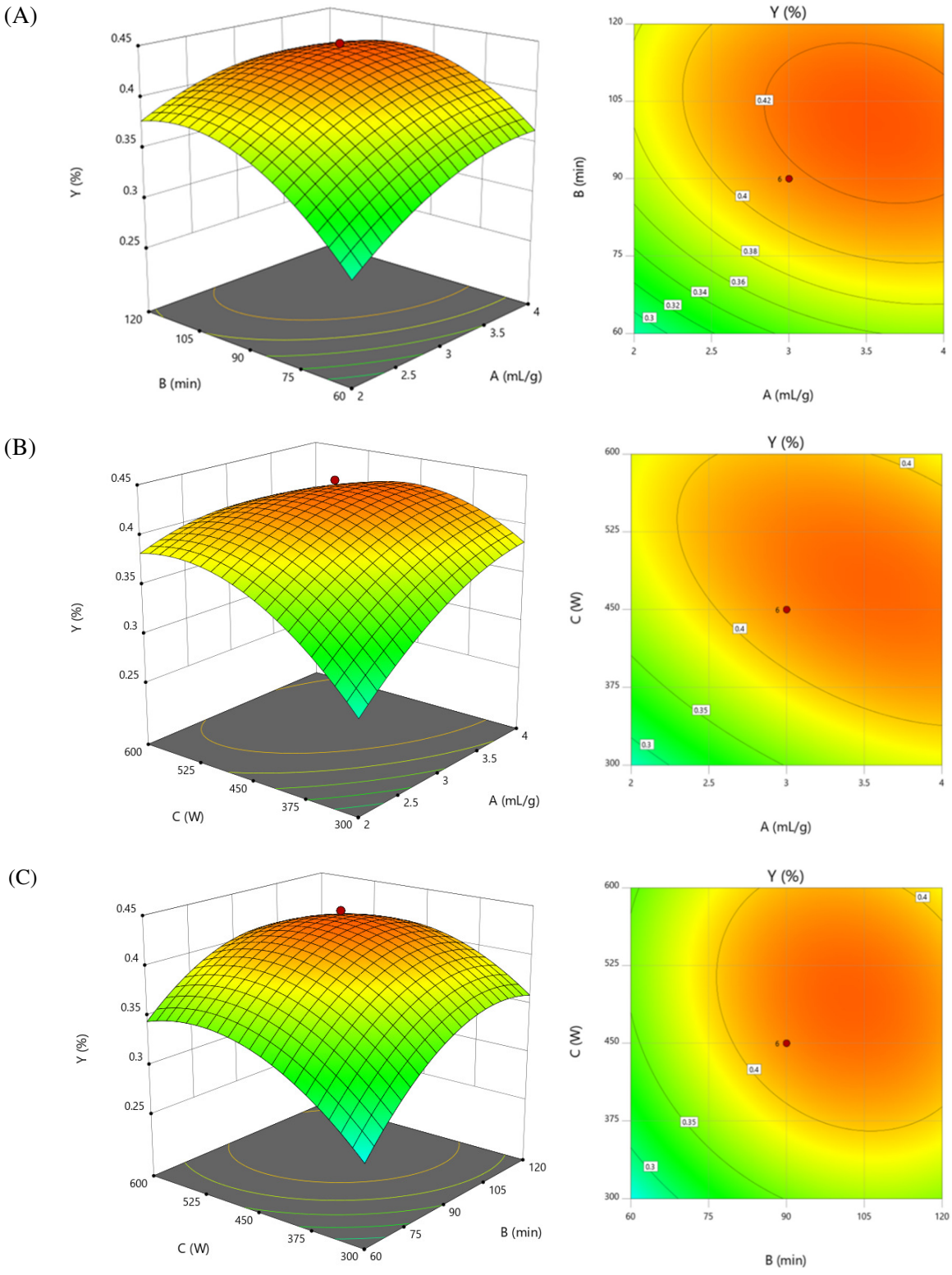

Fig.-4: 3D Response Surface Plots of the Interaction of Y with (A) Ratio Water and Raw Materials and Extraction Time, (B) Ratio Water and Raw Materials and Microwave Power, (C) Microwave Power and Extraction Time

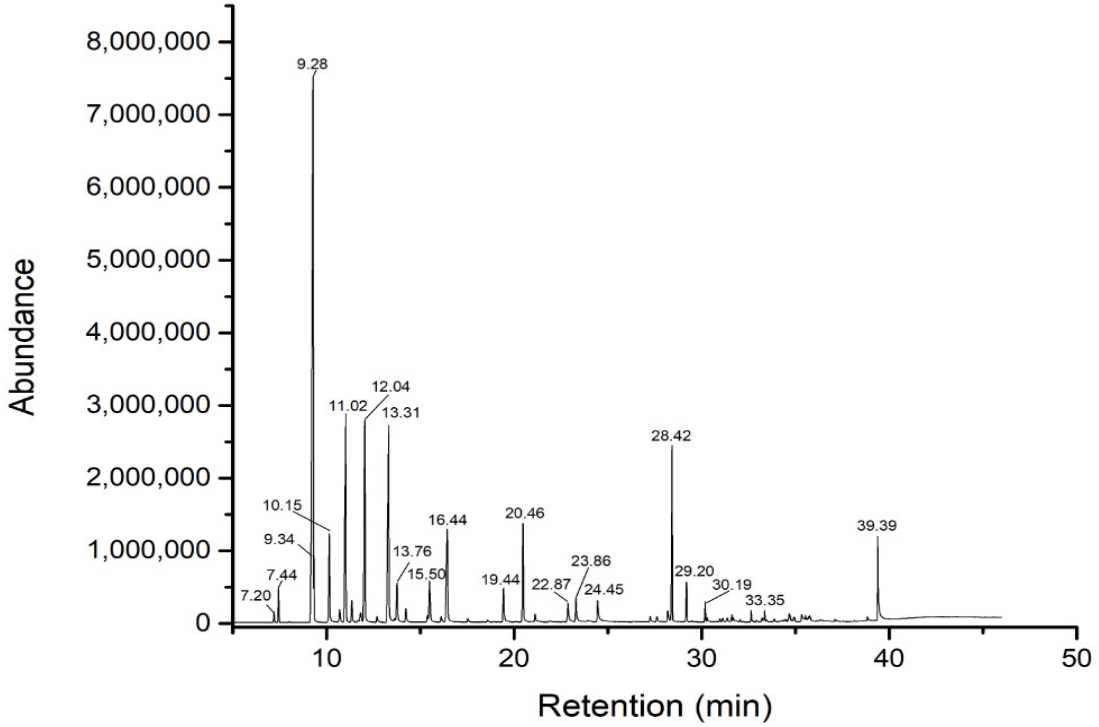

Fig.-5: GC-MS Results of Orange Leaves Oil Extraction by MAHD Method

Table-5: Chemical Composition of Orange Leaves Oil

No.	Component	MAHD(%)	No.	Component	MAHD(%)
1	2,4(10)-Thujadiene	0.339	15	Linalool	5.240
2	1R-α-Pinene	1.090	16	β-Citronellal	1.552
3	Sabinene	30.556	17	L-4-terpineneol	4.391
4	β-Pinene	1.618	18	α-Terpineol	0.318
5	β-Mycene	3.654	19	β-Cotronellol	1.059
6	α-Phellandrene	0.588	20	β-Citral	1.123
7	3-Carene	9.102	21	α-Citral	1.258
8	α-Terpinen	0.939	22	β-Elemen	0.609
9	o-Cymol	0.542	23	β-Elemenne	6.060
10	D-Limonene	9.682	24	Caryophyllene	1.325
11	Cis-Ocimene	10.139	25	α-Caryophyllene	0.617
12	γ-Terpinene	1.911	26	Elemol	0.277
13	Terpineol	0.658	27	Caryophyllene oxide	0.353
14	Terpinolene	2.139	28	Phytol	2.859

CONCLUSION

The present study explore microwave-assisted hydro-distillation of essential oil from orange leaves using response surface methodology (RSM). A total of 20 experimental runs following the Box-Behnken design was generated and attempted to generate the data for RSM procedure. The condition obtained an optimum yield of 0.43% with the material and water ratio of 3.46:1 (mL/g), the extraction time of 100.47 min, and 471.58 W operating power. The validity of the constructed model was verified by the determination

coefficients (R^2 = 0.9378, Adj. R^2 =0.8819) and the significance of the lack of fit ($p > 0.05$). This study serves as the precursor for the production of industrial scale by discovering optimal conditions of orange leaf oil extraction. In addition, not only did the MAHD method give very high oil yield, but the results of GC-MS also showed that the beneficial components existed in very high content in the essential oil.

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