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Optimization of Rosemary (*Rosmarinus officinalis* L.) Essential Oil Extraction using Solvent-Free Microwave Extraction (SFME) Method and Box–Behnken Design

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Abstract. Rosemary essential oil is widely utilized in pharmaceutical, cosmetic, and food industries due to its bioactive components. Solvent-Free Microwave Extraction (SFME) offers a green and energy-efficient alternative to conventional hydrodistillation. Previous studies on rosemary essential oil extraction using SFME or RSM have generally focused on isolated process parameters or limited optimization scopes. The novelty of this work lies in the systematic and simultaneous optimization of microwave power, feed-to-distiller ratio, and extraction time using a Box–Behnken Design, enabling a comprehensive evaluation of their individual and quadratic effects on essential oil yield. This study aims to optimize rosemary essential oil extraction using Response Surface Methodology (RSM) with a Box–Behnken Design (BBD). Three variables were evaluated : feed-to-distiller ratio (0.05–0.15 g/mL), extraction time (30–90 min), and microwave power (150–450 W). A quadratic model was successfully developed and validated through analysis of variance (ANOVA). The optimum conditions were achieved at a feed-to-distiller ratio of 0.10 g/mL, an extraction time of 60 min, and a microwave power of 300 W, resulting in a maximum essential oil yield of 3.1679%. The RSM–BBD model demonstrated strong predictive capability, confirming the significant influence of quadratic terms. The optimized SFME method provides an efficient, solvent-free extraction approach with potential applicability for industrial-scale natural product processing.

1 Introduction

Essential oils are natural products with high economic value due to their wide applications in pharmaceuticals, cosmetics, food, and aromatherapy industries[1]. Rosemary (*Rosmarinus officinalis L.*), a Mediterranean herb, is a potential source of essential oil rich in bioactive compounds such as 1,8-cineole, camphor, and α-pinene, which exhibit antioxidant, antimicrobial, and therapeutic properties[2], [3].

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Previous studies on rosemary essential oil extraction have primarily employed conventional hydrodistillation as well as microwave-assisted techniques, such as microwave-assisted hydrodistillation (MAHD) and solvent-free microwave extraction (SFME). Conventional hydrodistillation is widely used due to its simplicity; however, it often requires long extraction times and high energy consumption, which may lead to thermal degradation of volatile compounds [4]. Microwave-assisted methods have been introduced to overcome these limitations by enhancing internal heating and mass transfer, resulting in shorter extraction times and improved efficiency [5],[6]. The solvent-free microwave extraction (SFME) method is an environmentally friendly technique that increases the extraction efficiency of aromatic plants compared to traditional methods such as hydrodistillation, with a much shorter extraction time[10].

Despite these advancements, many reported studies have focused on investigating individual process parameters or have employed limited optimization strategies, which restrict a comprehensive understanding of parameter interactions and nonlinear effects. In particular, the simultaneous optimization of microwave power, feed-to-distiller ratio, and extraction time using a structured response surface methodology has not been extensively reported for rosemary essential oil extraction.

These variables play a critical role in solvent-free microwave extraction. Microwave power directly affects internal heating and rupture of oil-bearing cells, the feed-to-distiller ratio influences microwave energy absorption and vapor generation, and extraction time determines the extent of mass transfer and release of volatile compounds [5],[6]. The interaction among these parameters governs the efficiency of essential oil recovery and must therefore be evaluated simultaneously to achieve optimal extraction performance.

Response Surface Methodology (RSM) is a powerful statistical tool for process optimization, allowing the simultaneous evaluation of multiple variables and their interactions [7]. Among various RSM designs, the Box–Behnken Design (BBD) is particularly advantageous due to its efficiency and avoidance of extreme operating conditions while requiring fewer experimental runs compared to Central Composite Design (CCD) [8]. Therefore, this study addresses the existing research gap by applying RSM with a Box–Behnken Design to systematically optimize SFME conditions for rosemary essential oil extraction, providing statistical and mechanistic insights into the effects of key operating parameters on essential oil yield.

2 Methodology

2.1 Raw Material

Fresh rosemary (*Rosmarinus officinalis L.*) leaves were obtained from a local plantation in Pasuruan and Batu City, East Java, Indonesia. The plant material was authenticated by Dreamhouse Brutal Farm and Merekaah Jaya Indonesia. After collection, the leaves were thoroughly washed with water to remove surface impurities and then air-dried at ambient temperature for approximately 30 minutes to reduce surface moisture. The prepared leaf samples were subsequently stored in airtight containers at room temperature prior to the solvent-free microwave extraction process.

The use of fresh rosemary leaves was selected to preserve the natural moisture content within the plant matrix, which plays an important role in solvent-free microwave extraction. The inherent moisture acts as an internal microwave absorber, facilitating rapid heating, cell rupture, and enhanced release of essential oil components [5],[6]. Maintaining consistent raw material characteristics prior to extraction is essential to ensure reproducibility and reliability of the extraction process.

2.2 Extraction Method

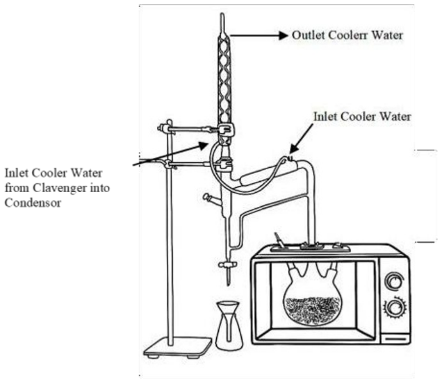


Fig 1. Microwave for Extracting Rosemary Using the SFME Method

The extraction was conducted using SFME under atmospheric pressure (1 atm) with a 1000 mL distiller volume. Independent variables included microwave, feed-to-distiller ratio (0.05, 0.10, and 0.15 g/mL), time of extraction (30, 60, and 90 min) and power (150, 300, and 450 W), extraction time up to 90 minutes. Sampling was carried out every 15 minutes, with experiments conducted in duplicate or triplicate to monitor yield development.

2.3 Experimental Design for Optimization with RSM BBD

A three-factor Box–Behnken Design was employed using Design Expert 13. Fifteen experimental runs were conducted to develop a quadratic predictive model for yield. ANOVA was applied to evaluate model significance.
A : F/D ratio : −1 = 0.05 g/mL, 0 = 0.10 g/mL, +1 = 0.15 g/mL
B : Time of Extraction : −1 = 30 min, 0 = 60 min, +1 = 90 min
C : Microwave power : −1 = 150 W, 0 = 300 W, +1 = 450 W

3 Results and Discussion

3.1 ANOVA and Model Adequacy

The quadratic model developed in this study was employed to predict the response of rosemary essential oil yield as a function of the selected extraction variables. In this section, the analysis focuses exclusively on essential oil yield as the primary response variable. Although oil quality is an important aspect of essential oil extraction, its evaluation was not included in the ANOVA model and is discussed separately.

RSM with Box–Behnken Design BBD was applied to model the relationship between process variables and responses (yield and quality of essential oil). The quadratic regression model was developed, and statistical analysis including ANOVA was performed to determine the significance of factors and their interactions[7], [8].

Table 1. Design Actual Input and Response

Std	Run	Factor 1	Factor 2	Factor 3	Response 1
		A:Ratio F/D	**B:Time**	**C:Power**	**yield**
		g/mL	**min**	**Watt**	**%**
3	1	0.05	90	300	0.663
11	2	0.1	30	450	0.2585
12	3	0.1	90	450	0.2585
6	4	0.15	60	150	0.1195
9	5	0.1	30	150	0.8662
15	6	0.1	60	300	3.1679
13	7	0.1	60	300	3.1061
1	8	0.05	30	300	0.5907
14	9	0.1	60	300	3.0224
4	10	0.15	90	300	0.3122
10	11	0.1	90	150	0.8662
2	12	0.15	30	300	0.1726
8	13	0.15	60	450	0.4127
7	14	0.05	60	450	0.6562
5	15	0.05	60	150	0.8663

P-values less than 0.05 indicate model terms are significant. The quadratic model was highly significant ($p = 0.0004$). Linear term A and quadratic terms A^2, B^2, and C^2 were dominant contributors. Non-significant interaction terms indicated minimal cross-variable effects, consistent with circular contour plots. Lack-of-fit was not significant ($p = 0.0678$), confirming model adequacy.

Quadratic Model:

$$Y = \beta 0 + \beta 1A + \beta 2B + \beta 3C + \beta 12AB + \beta 13AC + \beta 23BC + \beta 11A^2 + \beta 22B^2 + \beta 33C^2 \tag{1}$$

$$Y = 3.12 - 0.89A - 0.08B - 0.23C - 0.02AB + 0.13AC + 0.00BC - 7.65A^2 - 7.30B^2 - 6.89C^2 \tag{2}$$

In the quadratic regression model, the coded variables represent the feed-to-distiller ratio (A), extraction time (B), and microwave power (C). The linear term of factor A was found to be statistically significant ($p = 0.0346$), and its corresponding coefficient (β_1) was negative with a relatively large magnitude. This indicates that increasing the feed-to-distiller ratio beyond the central level tends to reduce the essential oil yield. In contrast, the linear effects of factors B and C were not significant, suggesting that their primary influence on the response is not linear in nature.

The quadratic terms A^2, B^2, and C^2 exhibited extremely high significance ($p < 0.0001$), demonstrating that curvature plays a dominant role in the response surface. This strong quadratic behavior reflects the existence of an interior optimum within the experimental region, rather than at the boundaries. Such behavior is typical in microwave-assisted extraction systems, where both insufficient and excessive levels of time, biomass loading, or microwave power lead to suboptimal yields.

All interaction terms (AB, AC, and BC) were statistically insignificant, as indicated by their small coefficients and high p-values. This finding is consistent with the nearly circular contour plots, confirming that the process variables exhibit minimal synergistic or antagonistic interactions. Therefore, the yield is governed primarily by the individual effects of each factor—particularly their nonlinear components—rather than by their combined interactive influences.

Table 2. ANOVA for the Quadratic Response Surface Model of Rosemary Essential Oil Yield

Source	Sum of Squares	df	Mean Square	F-value	p-value	Remarks
Model	16.82	9	1.87	40.06	0.0004	significant
A-Ratio F/D	0.3868	1	0.3868	8.30	0.0346	
B-Time	0.0056	1	0.0056	0.1204	0.7428	
C-Power	0.1603	1	0.1603	3.44	0.1229	
AB	0.0011	1	0.0011	0.0243	0.8823	
AC	0.0633	1	0.0633	1.36	0.2965	
BC	0.0000	1	0.0000	0.0000	1.0000	
A^2	6.79	1	6.79	145.68	< 0.0001	
B^2	6.31	1	6.31	135.41	< 0.0001	
C^2	5.57	1	5.57	119.53	0.0001	
Residual	0.2332	5	0.0466			
Lack of Fit	0.2225	3	0.0742	13.91	0.0678	not significant
Pure Error	0.0107	2	0.0053			
Cor Total	17.05	14				

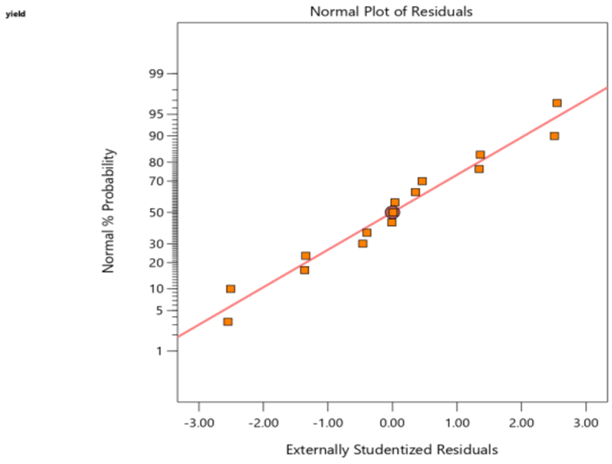


Fig 2. Plot Normal Residual of Rosemary Essential Oil

This normal probability plot of residuals shows the relationship between the expected normal values (theoretical quantiles) and the observed residuals from the RSM Box-Behnken optimization model for yield. From the plot, most data points fall close to the straight reference line, indicating that the residuals are approximately normally distributed. This suggests that the model assumptions of normality and randomness are satisfied. There are no significant outliers or deviations, meaning that the regression model used in the RSM–BBD adequately represents the experimental data. According to Montgomery[7], a normal residual pattern where data points align linearly along the reference line indicates that:

- The normality assumption is satisfied,
- The regression model is unbiased,
- The predictions are statistically reliable, and
- No significant outliers or structured error patterns are present.

Furthermore, the absence of curvature or an "S-shaped" deviation in the plot indicates that no transformation (such as Box–Cox) is required for the response variable. This confirms that the use of the untransformed quadratic model is appropriate for this study.The good agreement between the predicted and experimental values, as reflected in the distribution of residuals, also supports the ANOVA results, where the model was found to be significant and the lack-of-fit was not significant. This means that the variation between predicted and actual data is primarily due to random experimental error rather than deficiencies in the model structure.

Therefore, the yield results after optimization can be considered statistically reliable and well-fitted. The small residual variation implies that the predicted and experimental values are in good agreement, confirming that the optimization process using RSM Box-Behnken Design successfully enhanced the yield response within the studied factor range. Overall, the normal probability plot provides strong evidence that the RSM–BBD quadratic model satisfies the underlying statistical assumptions and demonstrates reliable predictive performance for rosemary essential oil yield optimization[7], [8].

3.2 Mechanistic Interpretation

The mechanistic interpretation of the optimization results provides insight into how the selected extraction variables influence rosemary essential oil yield during solvent-free microwave extraction.

3.3.1 Effect of feed-to-distiller ratio.

The feed-to-distiller ratio significantly affected the essential oil yield, as it determines the effective absorption of microwave energy by the plant matrix. An excessively low ratio limits the amount of extractable oil, whereas an excessively high ratio reduces heating efficiency due to uneven energy distribution. An optimal ratio facilitates uniform internal heating and efficient release of volatile compounds from oil-bearing cells.

3.3.2 Effect of microwave power.

Microwave power plays a crucial role in controlling the rate of internal heating and cell rupture. Increasing microwave power enhances the disruption of glandular structures, thereby improving mass transfer of essential oil. However, excessive power may lead to localized overheating and partial degradation of thermolabile compounds, resulting in a reduced yield. This trend is consistent with general microwave-assisted extraction behavior reported in the literature[5],[6].

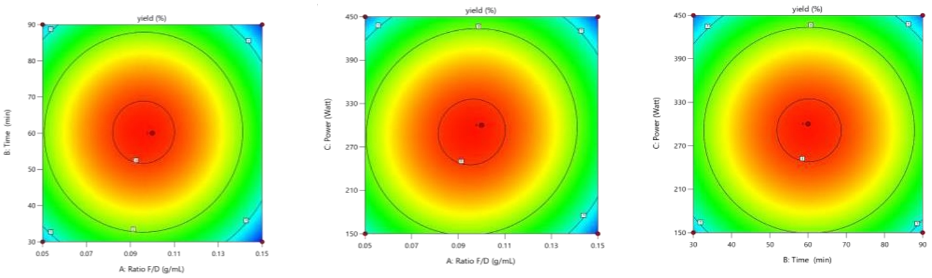
3.3.3 Effect of extraction time.

Extraction time governs the extent of microwave energy exposure and mass transfer. Insufficient extraction time results in incomplete oil release, while prolonged exposure may cause volatilization losses or degradation of essential oil components. Therefore, an intermediate extraction time was found to be optimal for maximizing essential oil yield.

F/D ratio effect at low F/D ratios, insufficient mass leads to low oil diffusion. At high ratios, overpacking reduces microwave penetration. Thus, 0.10 g/mL provides optimal internal heating and vapor release. Microwave power at Moderate power (300 W) allows effective in-situ heating of intracellular water, causing rapid oil release without overheating. Excessive power (>450 W) causes local overheating and degradation of heat-sensitive compounds, reducing yield. Athough Extraction time, yield increases up to 60 minutes before reaching equilibrium due to depletion of volatile constituents. These mechanisms align with microwave heating theory and prior observations in similar plants[5][6].

3.4 Contour and 3D Surface Analysis

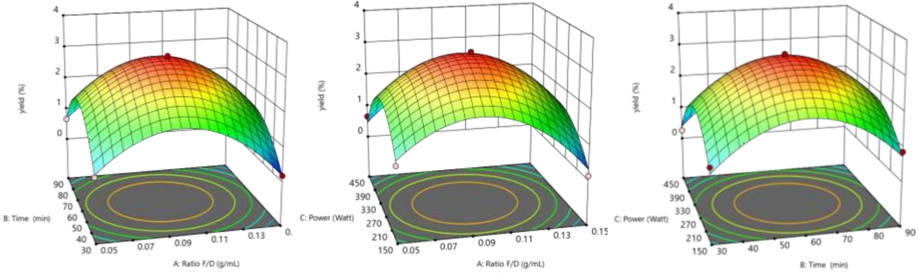
To facilitate a clearer interpretation of the interaction among the extraction variables and the location of the optimum conditions, contour and three-dimensional response surface plots were constructed using the RSM–BBD model.



(a) (b) (c)

Fig 3. Contour Plot Optimization of Rosemary Essential Oil (a) A Vs B = Ratio F/D – Time of Extraction; (b) A Vs C = Ratio F/D – Power ; (c) B Vs C = Time of Extraction – Power

The nearly circular and elliptical contou0 patterns suggest minimal interaction effects among the extraction variables. This observation is consistent with the ANOVA results, which indicate that interaction terms are statistically insignificant, while quadratic effects dominate the response behavior. The contour and surface plots therefore confirm the suitability of the RSM–BBD model in identifying the optimal operating region.



(a) (b) (c)

Fig 4. Model Graph 3D Surface Optimization of Rosemary Essential Oil (a) A Vs B = Ratio F/D – Time of Extraction; (b) A Vs C = Ratio F/D – Power ; (c) B Vs C = Time of Extraction – Power

The contour and three-dimensional surface plots (Fig. 3 and Fig. 4) illustrate the combined effects of feed-to-distiller ratio, microwave power, and extraction time on the yield of rosemary essential oil. The response surfaces exhibit a convex shape, indicating the presence of a single, well-defined optimum within the studied experimental range. The highest yield is observed at a feed-to-distiller ratio of approximately 0.10 $g \cdot mL^{-1}$, moderate microwave power around 300 W, and an extraction time of 60 min.

Under the optimized SFME conditions, a maximum essential oil yield of 3.1679% was achieved. This value compares favorably with reported yields from microwave-assisted extraction methods and is higher than those commonly obtained using conventional hydrodistillation under similar conditions[5][6]. The results demonstrate that the optimized SFME process provides an efficient and energy-effective alternative for rosemary essential oil extraction[9].

4 Conclusions

The RSM-BBD model successfully optimized rosemary SFME, identifying optimum conditions of 0.10 g/mL feed-to-distiller ratio, 60 minutes extraction duration, and 300 W microwave power, yielding 3.1679% essential oil. The quadratic model showed strong statistical performance, and mechanistic analysis confirmed that moderate internal heating promotes efficient oil release. Based on the statistical validation and optimization results obtained in this study, The optimized SFME process is a green and reproducible extraction approach, as it operates without organic solvents and achieves high yield within a relatively short extraction time, making it suitable for potential scale-up.

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