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# Optimization of ginger (*Zingiber officinale)* oleoresin extraction using palm oil as a green solvent through response surface methodology

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**ABSTRACT**

Ginger oleoresin is a concentrated natural product containing volatile and non-volatile constituents, including gingerols and shogaols, and is widely used in food, pharmaceutical, and related applications. Conventional extraction often relies on organic solvents, which may raise safety and environmental concerns. This study aimed to optimize the ultrasound-assisted extraction (UAE) of ginger oleoresin using palm oil as a vegetable-oil-based green solvent. A Central Composite Design (CCD) within Response Surface Methodology (RSM) was used to evaluate the effects of the solvent-to-sample ratio and extraction time. The investigated solvent-to-sample ratios ranged from 1:3 to 1:5.414 g/mL, while extraction times ranged from 17.574 to 102.426 min. Extraction yield and free fatty acid (FFA) content were used as response variables. The experimental data were evaluated using analysis of variance (ANOVA), the coefficient of determination ($R^2$), adjusted $R^2$, and response-surface analysis. The RSM models were significant for extraction yield ($p = 0.0030$) and FFA content ($p = 0.04848$). Increasing the solvent-to-sample ratio and extraction time tended to increase the measured extraction yield, whereas increasing the solvent-to-sample ratio tended to decrease the FFA content. The predicted optimum was a solvent-to-sample ratio of 1:4.774 g/mL and an extraction time of 89.211 min, yielding a predicted extraction-fraction yield of 176.807% and an FFA content of 0.252%. RSM identified an operating region that simultaneously addressed the extraction yield and FFA responses. However, the yield above 100% indicates that the measured fraction contained residual palm oil and, therefore, should not be interpreted as the yield of pure ginger oleoresin. Further separation and experimental validation are required before the optimum can be regarded as a quantitative optimum for pure oleoresin recovery.

**Keywords:** ginger; oleoresin; ultrasound-assisted extraction; palm oil; green solvent; response surface methodology



## INTRODUCTION

Ginger (*Zingiber officinale Roscoe*) is an important rhizomatous plant used as a spice, food ingredient and traditional medicinal material. Ginger contains a complex mixture of phenolic compounds, terpenes, lipids and other constituents. Among its characteristic bioactive compounds, gingerols and shogaols are particularly important because they contribute to the pungency and are associated with antioxidant, anti-inflammatory, antimicrobial and other biological activities (Mao et al., 2019; Zhang et al., 2020). Ginger oleoresin provides a concentrated form of these constituents and combines volatile and non-volatile fractions, making it useful for applications in food, pharmaceutical, cosmetic and nutraceutical products (Jayanudin et al., 2017; Kamaruddin et al., 2023). The extraction process strongly determines the composition, recovery and quality of ginger oleoresin.

Conventional extraction of ginger oleoresin commonly uses ethanol or other organic solvents. Although effective, solvent-intensive processes may involve flammability, toxicity, solvent-residue and environmental issues. Consequently, the development of extraction processes based on safer and more sustainable solvents has become an important aspect of green extraction technology (Kamaruddin et al., 2023). Vegetable oils are attractive alternatives because they are non-volatile, comparatively safe, renewable and capable of dissolving lipophilic compounds. Palm oil has also been investigated as a solvent for oleo-extraction of natural products, offering the additional possibility of producing an extract that can be directly incorporated into lipid-based formulations (Yara-Varón et al., 2017; Portillo-López et al., 2021). Ultrasound-assisted extraction (UAE) can further improve the efficiency of solid–liquid extraction through acoustic cavitation. The collapse of cavitation bubbles produces local mechanical effects that can disrupt plant tissues, improve solvent penetration and enhance mass transfer. Previous work on ginger has demonstrated that ultrasound can accelerate oleoresin extraction compared with conventional extraction, supporting its use as a process-intensification technology (Supardan et al., 2012). Nevertheless, extraction performance depends on operating parameters such as solvent-to-solid ratio and extraction time. An insufficient solvent volume can limit mass transfer, while excessive solvent may provide diminishing returns. Likewise, longer extraction can increase recovery up to a certain point, but prolonged exposure may increase degradation or undesirable reactions (Kumar et al., 2021; Malau et al., 2021).

Because extraction variables can interact, optimization based on a single-factor-at-a-time approach may require many experiments and may not identify the combined optimum. Response Surface Methodology (RSM) provides a statistical framework for modelling the response as a function of several independent variables and for identifying an operating region that meets predefined response criteria. Central Composite Design (CCD) is commonly used for this purpose because it estimates linear, interaction and quadratic effects with relatively few experimental runs (Montgomery, 2017). RSM has been applied successfully to optimize extraction processes for plant-derived products and can provide a more systematic basis for process development (Kuś et al., 2018; Chokwe et al., 2022). The present study therefore investigated the optimization of ginger oleoresin extraction using UAE with palm oil as the extraction medium. Solvent-to-sample ratio and extraction time were selected as the process variables, while measured extraction yield and FFA content were used as response variables. The study also explicitly considered an important limitation of oil-based extraction: because the palm oil solvent remains in the recovered fraction, a mass-based yield may exceed 100% and should not automatically be interpreted as pure oleoresin yield. This distinction is important for evaluating the feasibility of vegetable oils as green extraction media.

## METHODS

### Materials

Fresh ginger rhizomes were obtained from Pasar Wage, Purwokerto, Indonesia, and subjected to botanical identification to confirm their identity. Palm oil was used as the extraction solvent. n-Hexane, potassium hydroxide (KOH), and phenolphthalein were used for FFA analysis. All materials were handled according to standard laboratory procedures.

### Preparation of ginger simplicia

Fresh rhizomes were washed, drained, sliced into small pieces, and air-dried until sufficiently dry. The dried material was milled into powder and passed through a 40-mesh sieve to obtain a relatively uniform particle size. Size reduction was performed to facilitate solvent contact and improve mass transfer during extraction, consistent with the general principles of solid–liquid extraction.

### Experimental design and RSM

RSM was used to evaluate the effects of solvent-to-sample ratio ($X_1$) and extraction time ($X_2$) on extraction yield and FFA content. The nominal factor ranges were 1:3, 1:4 and 1:5 g/mL for solvent-to-sample ratio and 30, 60 and 90 min for extraction time. A CCD was then used to extend the experimental domain to axial points, resulting in a solvent-to-sample ratio range of 1:2.58579 to 1:5.41421 g/mL and an extraction-time range of 17.5736 to 102.426 min. The experimental combinations were randomized to reduce the influence of uncontrolled external factors. Statistical modelling was conducted using Design-Expert version 13.0.5.

**Table 1.** Central Composite Design used for optimizatiom of ginger oleoresin extraction

| Coded $X_1$ | Coded $X_2$ | Solvent:sample (g/mL) | Time (min) |
|---|---|---|---|
| -α | 0 | 1:2.58579 | 60 |
| -1 | -1 | 1:3 | 30 |
| -1 | +1 | 1:3 | 90 |
| 0 | -α | 1:4 | 17.5736 |

| | | | |
|---|---|---|---|
| 0 | 0 | 1:4 | 60 |
| 0 | 0 | 1:4 | 60 |
| 0 | 0 | 1:4 | 60 |
| 0 | 0 | 1:4 | 60 |
| 0 | 0 | 1:4 | 60 |
| 0 | +α | 1:4 | 102.426 |
| +1 | -1 | 1:5 | 30 |
| +1 | +1 | 1:5 | 90 |
| +α | 0 | 1:5.41421 | 60 |

**Ultrasound-assisted extraction**

The dried ginger powder was extracted with palm oil at the solvent-to-sample ratio specified by the experimental design. The extraction vessel was placed in an ultrasonic bath at a depth of approximately 7 cm and sonicated for the designated extraction time. After sonication, the mixture was centrifuged at 13,000 rpm for 10 min at room temperature. The supernatant was collected for subsequent analysis and determination of the extraction yield.

**Determination of extraction yield**

The measured extraction yield was calculated from the mass of the recovered extraction fraction relative to the initial mass of ginger powder. Because palm oil was intentionally used as the extraction medium and was not completely removed before weighing, the measured value represents the total recovered extract fraction rather than the mass of pure ginger oleoresin. This distinction was taken into account when interpreting the results.

**Determination of free fatty acids**

FFA content was determined by alkalimetric titration. Approximately 2.5 g of the recovered extraction fraction was accurately weighed and mixed with 25 mL of n-hexane. The mixture was warmed to approximately 40 °C to facilitate dissolution, followed by the addition of two drops of phenolphthalein. The sample was titrated with KOH until a stable pink endpoint was observed. The FFA content was expressed as a percentage. Alkalimetric titration is commonly used to determine free fatty acids in oil-containing matrices because the endpoint can be visually observed and the result can be calculated from the volume of titrant used.

**Statistical analysis**

The effects of $X_1$ and $X_2$ on each response were evaluated using ANOVA at a significance level of 5%. Model adequacy was assessed using the coefficient of determination ($R^2$), adjusted $R^2$, and the difference between these coefficients. Response-surface and contour analyses were used to visualize the effects of the experimental factors. The optimum condition was selected using the RSM desirability approach based on the measured responses. The predicted optimum and corresponding response values were then reported.

**RESULTS AND DISCUSSION**

**Extraction yield and interpretation of the measured fraction**

The measured extraction yield exceeded 100%. Such a value cannot be interpreted as the recovery of pure ginger oleoresin because the recovered fraction contains the palm oil used as the extraction medium. In an oil-based extraction system, the solvent is not necessarily removed during the separation step; consequently, the final weighed fraction contains both extracted ginger constituents and a substantial proportion of the carrier oil. This result is consistent with the concept of oleo-extraction, in which vegetable oil acts not only as an extraction solvent but also as a component of the final formulation (Yara-Varón et al., 2017).

This observation is important when interpreting the optimization results. A higher measured yield may indicate more efficient transfer of ginger-soluble constituents, but it may also reflect differences in the amount of palm oil retained in the recovered fraction. Therefore, the yield response in this study should be described as the measured extraction-fraction yield rather than pure oleoresin recovery. Additional separation, such as solvent exchange, selective partitioning, or another validated separation procedure, would be required to quantify the yield of pure oleoresin.

**RSM model significance**

**Table 2**. Statistical significance of the RSM models.

| Response | ANOVA p-value | Interpretation (α = 0.05) |
|---|---|---|
| Extraction yield | 0.0030 | Model significant |
| FFA content | 0.04848 | Model significant |

ANOVA indicated that the fitted models were significant for both measured responses. The p-value for extraction yield was 0.0030, whereas that for FFA content was 0.04848. Both values were below 0.05, indicating that the fitted models captured statistically significant effects of the experimental factors on the respective responses. The reported difference between $R^2$ and adjusted $R^2$ was less than 0.2 for both responses, supporting the adequacy of the models for interpretation and prediction within the investigated experimental domain. Because the exact $R^2$ values were not available in the source dataset, only the reported difference criterion is presented here.

**Table 3.** Coefficient of determination ($R^2$), adjusted $R^2$, and the differences between these coefficients for the RSM models.

| Respons | $R^2$ | Adj $R^2$ | Difference |
|---|---|---|---|
| % yied extraction | 0.6872 | 0.6346 | 0.0626 |
| % ALB | 0.4543 | 0.3452 | 0.1091 |

**Effects of solvent-to-sample ratio and extraction time**

Response-surface analysis indicated that increasing the solvent-to-sample ratio and extraction time tended to increase the measured extraction yield. Increasing the amount of palm oil may improve contact between the solvent and plant matrix and increase the capacity of the liquid phase to solubilize lipophilic constituents. However, this relationship should not be interpreted as a simple linear increase in pure oleoresin recovery because the measured mass also contains palm oil. Previous extraction studies have similarly shown that solvent volume and extraction time can influence mass transfer, although excessive solvent use may provide limited additional recovery once favourable mass-transfer conditions have been reached (Chokwe et al., 2022; Nde and Foncha, 2020). These trends are illustrated in Figure 2.

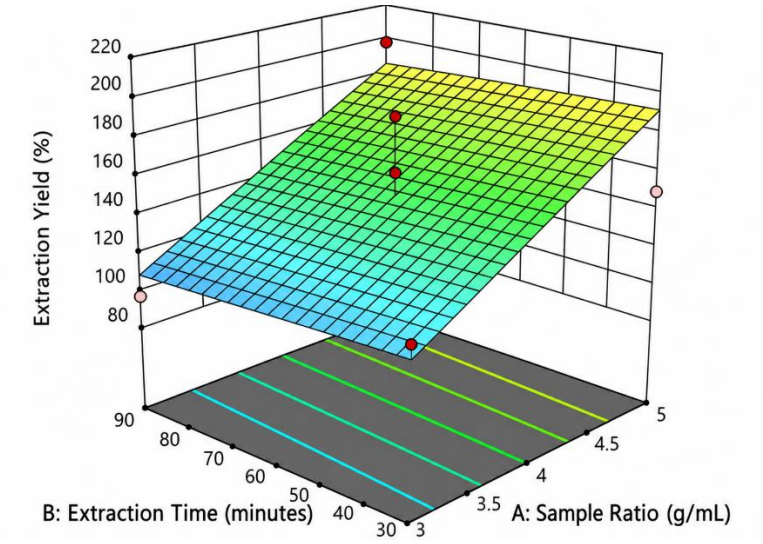


**Figure 1.** Three-dimensional response surface showing the effects of the solvent-to-sample ratio and extraction time on the measured extraction yield.

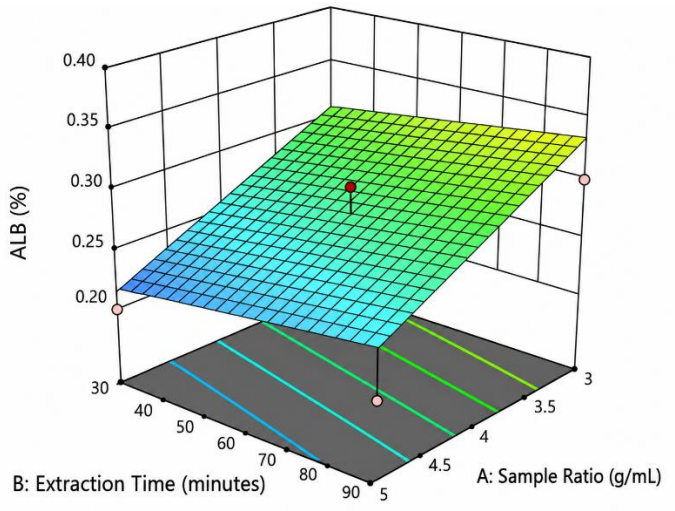


**Figure 2.** Three-dimensional response surface showing the effects of the solvent-to-sample ratio and extraction time on FFA (ALB) content.

For FFA content, increasing the solvent-to-sample ratio tended to reduce the measured percentage. A plausible explanation is a dilution effect: when a larger amount of palm oil is present relative to the extracted ginger material, the concentration of free fatty acids in the final oil-containing fraction may decrease. In contrast, longer extraction tended to increase FFA content. Prolonged exposure to the extraction conditions may increase the potential for lipid hydrolysis and the release of free fatty acids. This interpretation is consistent with the established relationship between processing conditions and FFA formation in palm-oil-containing systems (Nurfiqih et al., 2021). The opposing trends of the two responses illustrate the value of simultaneous optimization. Conditions that increase the measured extraction fraction are not necessarily those that minimize FFA. RSM provides a practical means of identifying a compromise region rather than optimizing each response independently. This approach is particularly relevant to oil-based extraction because the carrier oil is an integral component of the recovered fraction, and its quality characteristics influence the overall product.

**Predicted optimum condition**

**Table 4.** Predicted optimum condition obtained from the RSM model.

| Parameter | Predicted optimum | Predicted response |
|---|---|---|
| Solvent-to-sample ratio | 1:4.774 g/mL | Extraction yield = 176.807% |
| Extraction time | 89.211 min | FFA = 0.252% |

The predicted optimum condition was a solvent-to-sample ratio of 1:4.774 g/mL and an extraction time of 89.211 min. Under these conditions, the model predicted a measured extraction yield of 176.807% and an FFA content of 0.252%. The predicted yield is useful as a process response within the model; however, it should not be described as pure oleoresin yield because the recovered fraction contains palm oil. Therefore, the optimum represents an optimized condition for the measured oil-containing extraction fraction and its FFA response. The desirability and contour plots supporting this solution are shown in Figure 4, and the predicted optimum values are summarized in Figure 5.

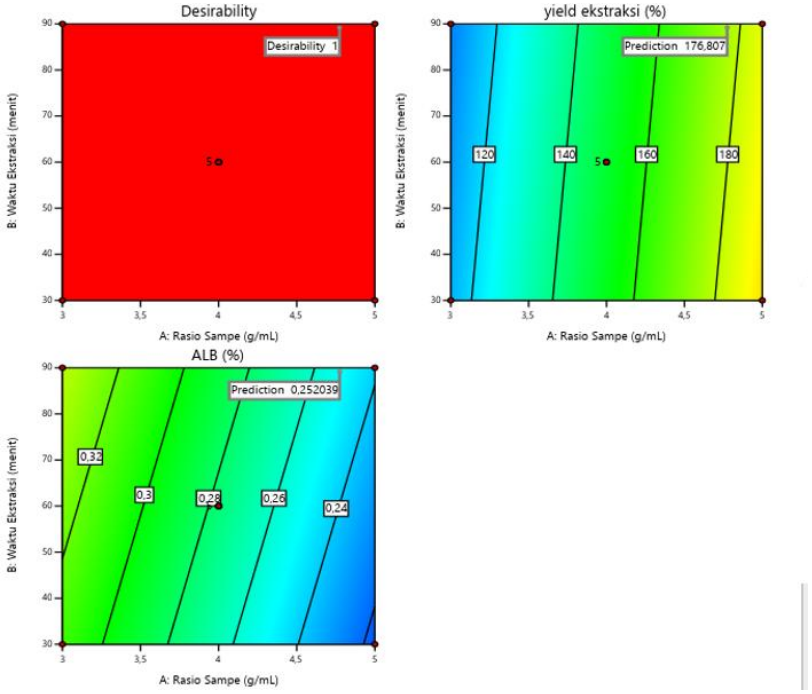


**Figure 4.** Desirability and contour plots used to identify the simultaneous optimum for extraction yield and FFA content.

**Table 5.** Predited optimum condition and corresponding response values from the RSM model.

| Ratio P:S (g/mL) | Time extraction (second) | % yield extract | % ALB |
|---|---|---|---|
| 1:4.774 | 89,211 | 176,809 | 0,252 |

**Discussion**

The findings support the feasibility of combining UAE with palm oil as an alternative extraction approach for ginger-derived lipophilic constituents. Vegetable oils have several characteristics that make them attractive green solvents: low volatility, relatively low toxicity, availability and compatibility with food and natural-product applications. Their use can reduce dependence on petroleum-derived organic solvents and may facilitate direct formulation of the extract in an oil-based product (Yara-Varón et al., 2017; Portillo-López et al., 2021). The present study extends this concept to ginger oleoresin by applying RSM to process variables that are operationally important for ultrasound-assisted extraction. The effect of extraction time is also consistent with the mechanism of ultrasound-assisted extraction. Acoustic cavitation can disrupt plant tissue and enhance solvent penetration, thereby accelerating mass transfer. Earlier work on ginger oleoresin reported faster extraction under ultrasound than with a conventional Soxhlet process and demonstrated structural disruption of plant cells after ultrasonic treatment (Supardan et al., 2012). However, the present study used palm oil rather than ethanol, and the behaviour of an oil-based medium may differ because of differences in viscosity and solubilization properties. Therefore, the optimum identified in this study should be considered specific to the investigated material, solvent, and process configuration. The FFA response provides an additional quality-related perspective. In an oil-based extraction system, the carrier oil contributes substantially to the chemical matrix, and its FFA level may change during processing. The increase in FFA with longer extraction time observed in the response surface is therefore relevant to product quality. Minimizing FFA may be desirable for maintaining oil stability and sensory quality, although the appropriate acceptance criteria depend on the intended application. Future studies should evaluate additional quality indicators, such as peroxide value, oxidative stability, fatty-acid composition, and markers of ginger bioactive compounds. A key limitation of this study is that the separation procedure did not completely remove palm oil before the extraction yield was determined. Consequently, the yield above 100% reflects measurement of the total recovered fraction. This limitation does not invalidate the statistical optimization of the measured response, but it limits interpretation of the response as a direct measure of oleoresin recovery. A stronger follow-up study should include quantitative determination of gingerol, shogaol, or other relevant marker compounds, together with a validated method for distinguishing extracted ginger constituents from the palm-oil carrier. Experimental validation at the predicted optimum would also be necessary to compare observed and predicted

responses and to establish the reproducibility of the model. Despite this limitation, the study provides a useful process-development framework. The combination of a green solvent, ultrasound-assisted extraction, and RSM reduces the number of experiments required to investigate interacting process factors and identifies a practical operating point. The results can serve as a basis for subsequent optimization focused on chemical markers and functional activity rather than mass-based yield alone.

## CONCLUSION

Response Surface Methodology successfully identified significant models for optimizing ginger oleoresin extraction using UAE with palm oil. The solvent-to-sample ratio and extraction time significantly influenced the measured extraction yield and FFA content, with model p-values of 0.0030 and 0.04848, respectively. The predicted optimum condition was a solvent-to-sample ratio of 1:4.774 g/mL and an extraction time of 89.211 min, with predicted responses of 176.807% measured extraction yield and 0.252% FFA. However, the yield above 100% indicates that the recovered fraction contained residual palm oil and therefore cannot be interpreted as pure oleoresin yield. Future studies should experimentally validate the predicted optimum, improve separation of the oil carrier from the extracted constituents, and quantify ginger-specific chemical markers to establish true oleoresin recovery and product quality.

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## CONFLICT OF INTEREST

The authors declare that they have no conflict of interest. The funder had no role in the study design, data collection, data analysis, interpretation of the results, manuscript preparation, or the decision to submit the manuscript for publication.

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