Microwave Hydrodistillation of Andaliman (Zanthoxylum acanthopodium DC) Seed Essential Oil Using Box-Behnken Design

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Abstract. Andaliman (Zanthoxylum acanthopodium DC) is a unique Indonesian spice that grows in North Sumatra. It has the potential to be the material source of essential oil. Andaliman essential oil has not available as a market commodity yet or still in the research extent where even the study of it is limited. This research has carried out the extraction of essential oil through one of the advanced extraction processes, namely microwave hydrodistillation. The experiment was designed using Box-Behnken to obtain an equation model of its process extraction. In this study, the effects of extraction parameters, such as microwave power, F/S ratio, and extraction time, were analyzed. From this research, a reduced quadratic model was obtained that could represent the extraction of andaliman essential oil using microwave hydrodistillation, where all three parameters had a significant effect on the yield of andaliman oil. Besides, the optimization of the model obtained the optimum condition of the andaliman essential oil extraction process. Also, the analysis of the sample product revealed that it was miscible in alcohol and had a relative density value of 0.8861 gr/ml (20°C). The main components of the sample consisted of dl-limonene, geranyl acetate, carvone, and citronella.

1. Introduction
Essential oil consists of volatile substances, such as terpenes and oxygenated compounds [1], which can evaporate at room temperature. The essential oil gives many benefits for applying in a body not only as a medicine but also as a beauty product [2,3]. Besides, it is also used in the raw materials of home care products. Recently, the research about essential oil has increased due to the health issues that become more intense and are assumed as an effect of synthetic chemicals using [4,5]. Many researchers focus on the extraction of natural products to replace synthetic chemicals, including essential oil either from an ordinary plant or an endemic plant.

Andaliman (Zanthoxylum acanthopodium DC) is one of the endemic plants in North Sumatra, Indonesia. It grows at 0.93 miles above sea level [6]. It is a spice which commonly used in the traditional food of Batak tribe, such as naniura, naniarsik, and napinadar. Previous research showed that andaliman had antimicrobial and antioxidant activity, besides also increased cell resistance to toxicity [7,8]. Based
on the approximate analysis conducted by Parhusip (2006) [7], andaliman had 8.01% essential oil content. However, with such great potential, there are only a few studies about andaliman. Therefore, this research would conduct the extraction process of andaliman essential oil using microwave hydrodistillation. This method is one of the advanced extraction methods which can overcome the lack of conventional extraction methods because it needs less solvent, takes shorter extraction time, and produces purer product. Besides, Kusuma and Mahfud (2017) [9] mentioned that the microwave hydrodistillation method required lower energy and contributed less CO₂. In this study, the experiment would be design based on the response surface methodology, which gives advantages, including reducing the material and time requirement [10]. From this design would be obtained a model that could be used to analyze the effect of extraction parameters on the yield of essential oil and to optimize the process. The adopted type of response surface methodology was the Box-Behnken design. Because compared with the other model designs, for example, central composite design, it has a fewer run number and only divides the level of parameters into three levels [11]. Also, it does not have a star-point so that it is easy to be applied.

2. Materials and Methods

2.1. Materials

Andaliman seeds were harvested from one of the field’s farmers in Silaen, district of Toba Samosir, North Sumatra. The seeds were dried in shaded open space and then put into woven polypropylene bag, wrapped and sent to the Department of Chemical Engineering, ITS Surabaya, where the seeds were sorted and its moisture content was determined about 12.36%. Aquadest was used as a solvent.

2.2. Extraction process using microwave hydrodistillation

Andaliman seeds were weighed according to the variable of feed/distiller ratio (0.3 - 0.5 gr/ml) using the Ohaus-analytical balance (readability = 0.0001 gr). Along with 200 ml of aquadest, it was put into a distiller 1000 ml, which next was input in the microwave oven (ELECTROLUX type of EMM2308X with a dimension of 292.5 mm x 485 mm x 370 mm, maximum power 800 W, and frequency of 2.45 GHz). The system of experimental equipment (figure 1) was turned on by setting the variable of time (20 - 140 min) and variable of microwave power (300 – 600 W). For notice, time counted of extraction was start from the first drop of condensate in Clevenger. After the process finished, the essential oil of andaliman was separated using the tap of Clevenger, and then it was collected in a vial bottle, which coated using foil to prevent the product from light degradation. Next, the yield of the product was determined using equation (1), and the physicochemical of the product was analyzed.

\[
Yield (\%) = \frac{\text{essential oil weigh}}{\text{material weigh} \times (1 - \text{moisture content of material})} \times 100 \%
\]  

(1)

Description:
1. Reflux condenser
2. Clevenger
3. Microwave oven
4. Time setting
5. Power setting
6. Distiller containing material and aquadest
7. Separating tap

**Figure 1.** A set of experimental equipment
2.3. Design of experiment using Box-Behnken method

In this study, Design-Expert, version 11 trial (Stat-Ease Inc., Minneapolis, MN), was used to design the model of Box-Behnken. The three parameters consisted of microwave power (W), feed/solvent ratio (gr/ml), and extraction time (min), at three-levels (low, moderate, and high) were set as in Table 1, where the response variable was the yield of extraction, and 17 matrix combinations of parameters, together with actual yield, were shown in Table 2.

Table 1. Parameters and levels design of Box-Behnken

| Parameter        | Unit | Low (-1) | Moderate (0) | High (+1) |
|------------------|------|----------|--------------|-----------|
| A: Microwave power | W    | 300      | 450          | 600       |
| B: Feed/solvent ratio | gr/ml | 0.3      | 0.4          | 0.5       |
| C: Extraction time | min  | 20       | 80           | 140       |

Table 2. Matrix combinations of parameters and actual results

| Run | Microwave power (W) | Feed/solvent ratio (gr/ml) | Extraction time (min) | A    | B    | C    | Actual yield (%) |
|-----|---------------------|-----------------------------|-----------------------|------|------|------|------------------|
| 1   | 600                 | 0.4                         | 20                    | 1    | 0    | -1   | 2.2607           |
| 2   | 600                 | 0.5                         | 80                    | 1    | 0    | 0    | 1.7660           |
| 3   | 300                 | 0.3                         | 80                    | -1   | -1   | 0    | 1.0427           |
| 4   | 450                 | 0.3                         | 20                    | 0    | -1   | -1   | 0.6229           |
| 5   | 600                 | 0.4                         | 140                   | 1    | 0    | 1    | 4.2313           |
| 6   | 450                 | 0.5                         | 20                    | 0    | 1    | -1   | 0.3233           |
| 7   | 450                 | 0.4                         | 80                    | 0    | 0    | 0    | 3.5494           |
| 8   | 300                 | 0.4                         | 20                    | -1   | 0    | -1   | 0.6248           |
| 9   | 450                 | 0.5                         | 140                   | 0    | 1    | 1    | 1.2325           |
| 10  | 300                 | 0.5                         | 80                    | -1   | 1    | 0    | 0.3560           |
| 11  | 450                 | 0.4                         | 80                    | 0    | 0    | 0    | 3.0481           |
| 12  | 450                 | 0.4                         | 80                    | 0    | 0    | 0    | 3.4780           |
| 13  | 450                 | 0.3                         | 140                   | 0    | -1   | 1    | 1.7743           |
| 14  | 600                 | 0.3                         | 80                    | 1    | -1   | 0    | 3.5933           |
| 15  | 300                 | 0.4                         | 140                   | -1   | 0    | 1    | 2.2944           |
| 16  | 450                 | 0.4                         | 80                    | 0    | 0    | 0    | 3.3585           |
| 17  | 450                 | 0.4                         | 80                    | 0    | 0    | 0    | 2.9032           |

Then, using model equation (2), the predicted yield was approximated through multiple regression.

\[
Y = \beta_0 + \sum_{i=1}^{n} \beta_i x_i + \sum_{i=1}^{n} \beta_{ii} x_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} \beta_{ij} x_i x_j + e
\]  

Where \( \beta_0, \beta_i, \beta_{ii}, \beta_{ij} \) are intercept, linear, quadratic and interaction term of parameters respectively, while \( x_i \) and \( x_j \) are the independent parameters, \( n \) is parameters number, and \( e \) is the error value. The practicability of the model was analyzed using analysis of variance (ANOVA). Then the model was optimized to get the maximum yield at the range design of parameters.

3. Results and Discussion

3.1. Model selection
Box–Behnken design is one of the three-level models’ response-surface methodology that consists of a minimum 13 total run number [12]. Twelve runs of it are factorial combination, and another is the center point. This research used 17 total run numbers, which meant five of them were center points. Compared to the full factorial design, this model has reduced the combination matrix design. But even so, the model should can define the design space. Therefore, for choosing a proper model, the polynomial model fitting was analyzed, as shown in table 3. Those data showed that the cubic model noted as aliased, which meant the cubic model was inappropriate to evaluate the response surface design due to missing data points. Then the cubic model was excluded from consideration. As a notice, the highest order model with significant terms is the desired model. In this case, the quadratic model was noted as a suggested model because of having a significant effect, shown by the sequential p-value lower than 0.05. It also had an insignificant p-value of lack of fit (>0.05) and a positive R^2_pre, which represented the feasibility of the model [11]. Meanwhile, the 2FI model did not meet one of the requirements because not only it had an insignificant sequential p-value, and a significant of the lack of fit but also the value of the R^2_pre was minus. On the other hand, the linear was not a select model even its sequential p-value (0.0349) was lower than 0.05 because this model had a p-value of lack of fit =0.0059, which indicated the inappropriate of the model. Therefore, the quadratic model was the select model to be used for the next step.

| Table 3. Models fit summary statistics |
|--------------------------------------|
| Source | Sequential p-value | Lack of fit p-value | R^2_adj | R^2_pre | Note |
|---------|-------------------|---------------------|---------|---------|------|
| Linear  | 0.0349            | 0.0059              | 0.3511  | 0.1365  |       |
| 2FI     | 0.9647            | 0.0031              | 0.1783  | -0.6405 |       |
| Quadratic | 0.0002           | 0.1652              | 0.9146  | 0.5720  | Suggested |
| Cubic   | 0.1652            |                     | 0.9529  |         | Aliased |

3.2. Improving the selected model
The quadratic model equation, in the full-term, could be written as equation (3). The coefficient sign indicates the effect of the factors on the yield andaliman essential oil (Y), where the used value range of microwave power (A), F/S ratio (B), and extraction time (C) in this equation is in the coded term, (-1)–(+1).

\[
Y = 3.27 + 0.9417A - 0.4194B + 0.7126C - 0.1067A^2 - 1.47B^2 - 0.8080C^2 - 0.2852AB + 0.0752AC - 0.0605BC
\]

(3)

Further analysis of this model (table 4) showed that all the linear coefficients (A, B, and C) had a significant effect on the yield of andaliman essential oil as well as the quadratic of the F/S ratio (B^2) and extraction time (C^2) due to having p-value<0.05. While the interaction terms (AB, AC, and BC) insignificantly affected the yield of andaliman essential oil as well as the quadratic of microwave power (A^2). These insignificant terms affected the value of the regression model. There are three types of coefficients determination, including the performing coefficient (R^2), adjusted coefficient (R^2_adj), and predicted coefficient of determination (R^2_pre). R^2 and R^2_adj are fit if their values approach to one, while R^2_pre should be lower than R^2_adj, and has a different value not more than 20% to R^2_adj. Commonly, only two of them are used, especially for the process of natural material due to requiring maintenance to keep the same material specification. But it will be good if all these coefficients determination meet the requirements. The full quadratic model of andaliman essential oil extraction had an R^2 value of 0.9626 and an R^2_adj value of 0.9146, which close to one. But R^2_pre had a difference with R^2_adj about 34.26%, more than 20%, which meant not eligible. In this case, improving the full quadratic model by reducing...
insignificant factors of the model equation could improve the $R^2_{pre}$, which also improved the reliability of the model.

### Table 4. ANOVA for fully quadratic model

| Source            | Sum of squares | df  | Mean square | F-value | p-value |
|-------------------|----------------|-----|-------------|---------|---------|
| Model             | 25.58          | 9   | 2.84        | 20.03   | 0.0003  |
| A-Microwave power | 7.09           | 1   | 7.09        | 50.00   | 0.0002  |
| B-F/S ratio       | 1.41           | 1   | 1.41        | 9.92    | 0.0162  |
| C-Extraction time | 4.06           | 1   | 4.06        | 28.63   | 0.0011  |
| AB                | 0.3252         | 1   | 0.3252      | 2.29    | 0.1738  |
| AC                | 0.0227         | 1   | 0.0227      | 0.1596  | 0.7014  |
| BC                | 0.0147         | 1   | 0.0147      | 0.1033  | 0.7573  |
| A²                | 0.0480         | 1   | 0.0480      | 0.3380  | 0.5792  |
| B²                | 9.11           | 1   | 9.11        | 64.23   | < 0.0001|
| C²                | 2.75           | 1   | 2.75        | 19.37   | 0.0032  |
| Residual          | 0.9932         | 7   | 0.1419      |         |         |
| Lack of Fit       | 0.6803         | 3   | 0.2268      | 2.90    | 0.1652  |
| Pure Error        | 0.3129         | 4   | 0.0782      |         |         |
| Cor Total         | 26.57          | 16  |              |         |         |

$R^2 = 0.9626; R^2_{adj} = 0.9146; R^2_{pre} = 0.5720; Adeq Precision = 14.42$

As the equation for the reduced quadratic model was in equation (4) in the coded term. Based on the analysis of variance (ANOVA), shown in table 5, the significance of the model has improved. It was noted for the decrease in the p-value of model, and the increase in p-value of lack of fit (0.2636). Besides, the $R^2_{pre}$ also became higher (0.8558) and got closer to $R^2_{adj}$ (0.9232) with a difference value was lower than 20 %. The $R^2$ value of 0.9472 also fitted. Furthermore, the normal probability plot of the full quadratic model (figure 2a) and reduced model (figure 2b) fitted the diagonal line, which indicated that data was normally distributed. But, these figures showed that two points potentially became outliers. The existence of these outliers was getting clearer when evaluated based on the cook's distance plot. Where in cook's distance diagnostic, a data is assumed as an outlier if the cook’s distance ($D_i$)>1 based on Cook and Weisenberg rule or if $D_i$>4/n based on Bolen and Jackman rule [13]. According to the figures, two points acted as an outlier for the fully quadratic model (figure 3a). While for the reduced model (figure 3b), none data was out of the range. Therefore, rather than using the full quadratic model, the reduced model was chosen for studying the extraction of andaliman essential oil using the microwave hydrodistillation method.

$$Y = 3.22 + 0.9417 A - 0.4194 B + 0.7126 C - 1.48 B^2 - 0.8136 C^2$$  \[(4)\]

### Table 5. ANOVA for reduced model

| Source            | Sum of squares | df  | Mean square | F-value | p-value |
|-------------------|----------------|-----|-------------|---------|---------|
| Model             | 25.17          | 5   | 5.03        | 39.45   | < 0.0001|
| A-Microwave power | 7.09           | 1   | 7.09        | 55.59   | < 0.0001|
| B-F/S ratio       | 1.41           | 1   | 1.41        | 11.03   | 0.0068  |
| C-Extraction time | 4.06           | 1   | 4.06        | 31.83   | 0.0002  |
| B²                | 9.11           | 1   | 9.11        | 64.23   | < 0.0001|
| C²                | 2.75           | 1   | 2.75        | 19.37   | 0.0032  |
| Residual          | 0.9932         | 7   | 0.1419      |         |         |
| Lack of Fit       | 0.6803         | 3   | 0.2268      | 2.90    | 0.1652  |
| Pure Error        | 0.3129         | 4   | 0.0782      |         |         |
| Cor Total         | 26.57          | 16  |              |         |         |
\( R^2 = 0.9472; \ R^2_{\text{adj}} = 0.9232; \ R^2_{\text{Pre}} = 0.8558; \ \text{Adeq Precision} = 20.0879 \)

![Graph](image1)

**Figure 2.** Normal probability of fully quadratic model (a), and reduced model (b)

![Graph](image2)

**Figure 3.** Plot cook’s distance for fully quadratic model (a), and reduced model (b)

### 3.3. Effect of parameters process on the yield extraction

The extraction using microwave hydrodistillation is a unique process. Not only uses aquadest as a solvent which can not dissolve the solute, andaliman essential oil, but also it heats the materials using the micro energy which can directly be absorbed by the system. Aquadest in this process acts to help break the material matrix and to carry the solute. Andaliman essential oil and aquadest together make an immiscible mixture system so that it boils at a lower temperature than the pure boiling point of each component. On the other hand, the electric compound of the system absorbs the microwave radiation without firstly heat the distiller flask like in the conventional process. Therefore, aquadest, the most
constituent in the system with a high dielectric constant, and the other electric compound in the matrix material can directly change the microwave energy into heat energy, so that the heating process become getting faster [14]. But this process is also affected by the other parameters to get a high yield extraction. In this study, the effect of the parameters process (microwave power, F/S ratio, and extraction time) was analyzed on the yield of extraction at one factor through the graphs in figure 4.

Figure 4a displayed a positive gradient of the plot microwave power versus the yield of andaliman oil at the range of 300 W - 600 W. It meant the increase in microwave power would increase the yield of andaliman essential oil because an increasing microwave power could accelerate the increase in temperature and the internal pressure of the system due to the acceleration of molecular rotation [15], which hastened the opening of the material cell wall that facilitated penetration and diffusion process.

The effect of the F/S ratio on the yield of andaliman essential oil, as shown in figure 4b, drew a curve pattern. It indicated that increased the F/S ratio would increase the yield of andaliman essential oil for the range of 0.3 gr/ml - 0.4 gr/ml, but then the yield decreased due to increasing the F/S ratio more than 0.4 gr/ml. That increasing phenomenon could be affected by the material increasing at a constant of solvent volume, which also meant the increase in the availability of extraction components. But the increasing of material mass not always increase the yield of extraction because it can come to the point where the material mass is too much, which impede the transport phenomenon like penetration of microwave into the material. This correlated with the research that was conducted by Lee et al. (2020) [16]. In that research, it was mentioned that penetration of microwave in water only about ±14 mm. Sure it could increase due to the increase of the temperature, but with the flask sized of ±131 mm, much and centralized material would hinder that penetration. Besides, too much material also interferes with component diffusion. Those were the reason why on this research, increased the F/S ratio of more than 0.4 gr/ml caused the decrease in yield.

As for the effect of extraction time on the yield of extraction was proportional, figure 4c showed the longer extraction time was, the higher yield obtained. But it should be noticed that the graph became flatter with the increase of extraction time because that increasing became more insignificant due to reaching the limit of diffusion [17,18]. Besides, the longer extraction time could also induce the degradation of volatile compounds [19,20]. So that, in a condition where the difference of yield at a short time is not too significant compared with a long time, it is better to choose the short time condition.
3.4. Optimization

Optimization is a technique to get the optimum condition of the process parameters where the value of variable response is high by finding the critical point of the model equation [12]. Based on the reduced equation (equation 4) using the Design-Expert version 11 trial, this condition was at microwave power 590.653 W, F/S ratio 0.3741, and extraction time 98.6496 minutes where the predicted yield was 4.25787. It was one of the hundred solutions which preserved in that software. At the plot contour of two-dimension (figure 5a) and three-dimension (figure 5b), it was the point in the red color contour. The red color in figure 5 displayed a condition with a high yield of extraction. It meant that as long as a condition in the red area, there was no significant difference in yield of extraction. But it should be considered to pick the point that related either in the same or near to the condition of actual experiment. So for this case, actual condition that near to the optimum point condition has been run at microwave power 600 W, F/S ratio 0.4, and extraction time 100 minute where the actual yield was 4.1579%. This value was close to the predicted value with the difference of 0.1%. Therefore the modeling for andaliman essential oil extraction using microwave hydrodistillation could represent the process and it was feasible for use.
3.5. Physicochemical of andaliman essential oil

Physicochemical analysis of essential oil is useful for knowing its quality, and become a resource for comparison with the same type of essential oil but in a different extraction method or material plantation sourcing. Commonly, an essential oil that has already been market has a standard qualification product, determined by the standardization agency like the International Organization for Standardization (ISO) and the Standard Nasional Indonesia (SNI). But as andaliman essential oil still a new kind in essential oil or even still on the research area, it does not have the standard spesification either by ISO or SNI. Therefore the analysis of andaliman essential oil in this research is useful for quality identification rather than comparing it with another study. In this research, the physicochemical analysis was consisted of solubility in alcohol, relative density, and component analysis. For the solubility analysis, to get enough sample volume for analyzed, the andaliman essential oil samples from any process condition were combined. The result analysis for solubility in alcohol showed that andaliman essential oil was very soluble in ethanol 95% at any tested ratio or called as miscible. But the condition with a clear solution was obtained when andaliman essential oil volume higher than ethanol volume. This solubility indicated that the sample contained a high concentration of oxygenated compounds. While the relative density analysis of the product, where water was the reference, showed a value of 0.8861±0.0000 gr/ml (20°C). It meant that andaliman essential oil was less dense than water, and the dominant compound of it was the light fraction. The component analysis through GC-MS confirmed that statement. Based on that result, the components of andaliman essential oil were monoterpenes (54.7%), oxygenated (36.8%), and sesquiterpenes (1.08%). Furthermore, the GC-MS analysis showed the main components of andaliman essential oil consisted of dl-limonene, geranyl acetate, carvone, and citronella (table 6).

| No | Component      | RT  | % Normalisation area | Qual |
|----|----------------|-----|----------------------|------|
| 1  | dl-Limonene    | 4.29| 52.33%               | 99   |
| 2  | Geranyl acetate| 11.42| 25.1%                | 91   |
| 3  | Carvone        | 9.07| 3.61%                | 97   |
| 4  | Citronella     | 7.44| 3.2%                 | 91   |

4. Conclusion

The reduced quadratic model of Box-Behnken design could represent the extraction of andaliman essential oil using microwave hydrodistillation. In this process, the extraction parameters, including microwave power, F/S ratio, and extraction time had a significant effect on the yield of extraction. The appropriate of the model was confirmed by the three coefficients determination value of $R^2 = 0.9472$; $R^2_{adj} = 0.9232$; $R^2_{pre} = 0.8558$. Where optimization of the model resulted in optimum condition at microwave power 590.653 W, F/S ratio 0.3741, and extraction time 98.6496 minutes with the predicted yield was 4.25787%. As for the analysis of andaliman essential oil showed that the sample was miscible in alcohol and had a relative density value of 0.8861 gr/ml (20°C). Then GC-MS analysis showed that the main components consisted of dl-limonene, geranyl acetate, carvone, and citronella.

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