

EXTRACTION OF ESSENTIAL OIL FROM RED GINGER (*Zingiber officinale var rubrum*) BY SOLVENT-FREE MICROWAVE EXTRACTION METHOD

Renita Manurung✉

Department of Chemical Engineering¹
renita.manurung@usu.ac.id

Zahra Adelia Siregar

Department of Chemical Engineering¹

Anefa Baroza

Department of Chemical Engineering¹

Alwi Gery Agustan Siregar

Department of Graphic Engineering²

¹Universitas Sumatera Utara

Kampus USU Padang Bulan, Medan, Indonesia, 20155

²Politeknik Negeri Media Kreatif

6 Jl. Guru Sinumba, Helvetia Medan, Indonesia, 20124

✉ Corresponding author

Abstract

The importance of essential oils in various industries, such as pharmaceutical, food, and agriculture, has encouraged the development of production from various natural materials. One of the natural materials that produce essential oil is red ginger. Red ginger has the potential to become a local essential oil business because it has a high selling value with its content ranging from 2.58–2.72%. Extraction of essential oil by conventional methods takes longer than the microwave assisted extraction method, but to get purer results, a solventless method called solvent-free microwave extraction is used. In this study, red ginger essential oil was extracted using the solvent-free microwave extraction method with variations in microwave power (low, medium low, medium and high) and extraction time (60, 75, 90, 105 and 120 minutes). This study aims to determine the effect of microwave power and extraction time on yield and characteristics of red ginger essential oil in accordance with SNI 06-1312-1998. The best yield of essential oil was found in the treatment of Low power and 120 minutes. In this result, the yield was $1.90618 \pm 0.00807\%$ and the characteristics that have met SNI are light yellow in color and distinctive ginger aroma and density of 0.88164 ± 0.00105 g/mL. FTIR spectrogram showed the presence of O-H, C-O, and C=O functional groups and C=C double bonds. These groups were identified as terpenoid group compounds and several other derivative compounds that undergo oxidation, in this case is a carboxylic acid. Based on GC-MS analysis, the estimated oxidized zingiberene compound is 96.09%. The terpenoid compounds contained can be utilized as antibacterial with the resulting inhibition zone included in the moderate category of 7.6 mm.

Keywords: essential oil, red ginger, solvent-free microwave extraction.

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1. Introduction

Essential oils are complex combinations of volatile chemical components that have a strong aroma. These compounds can be found in various parts of plants, such as roots, stems, leaves, flowers, fruits, and seeds [1]. Essential oils are usually used as raw materials for medicines, perfumes, food flavorings, pesticides and have pharmacological effects such as antioxidants, antibacterial, anti-inflammatory, analgesic [2]. Its important role for society causes various industries and researchers to continue working to develop essential oils from various natural ingredients.

One of the essential oil-producing plants is red ginger (*Zingiber officinale var rubrum*). Red ginger is one type of spice that is commonly found in Indonesia. The distinctive flavor of red

ginger comes from the yellow and slightly thick essential oil. The essential oil content in red ginger ranges from 2.58–2.72% based on dry weight [3]. Essential oil from red ginger, also known as red ginger oil, has high potential to be used as a local business because it has a high value in the world market. The price of ginger oil can reach IDR 500,000–600,000/kg [2]. However, red ginger oil-based products are still rare in the market. Compared with fresh red ginger, red ginger essential oil has a higher economic value and is easier to circulate. Processing red ginger into essential oil is expected to fulfil the needs of the growing industry.

The development of methods in the extraction of essential oils from natural materials has been widely carried out by researchers. This is done to find out the right and effective method in obtaining high yields with quality that meets quality standards. Research that has been done in extracting red ginger essential oil is using the hydrodistillation method with a material and solvent ratio of 1:12 and obtained a yield of 1.55% with a distillation time of 4 hours [4]. However, the long heating time can result in the decomposition of compounds. Therefore, an extraction method with a short time is needed with the help of micro waves. However, due to concerns about the impact of solvent use on the environment and human body, a microwave-assisted extraction method called solvent-free microwave extraction (SFME) was developed. This method is a technology for extracting natural materials without the use of solvents and high energy [5].

In this research, essential oil extraction from red ginger (*Zingiber officinale var rubrum*) was carried out using the solvent-free microwave extraction (SFME) method. The use of this method is a research update on red ginger raw materials. The effect of extraction time and microwave power on the yield and composition of red ginger essential oil using the SFME method and the resulting characteristics were evaluated.

2. Materials and method

2. 1. Materials and equipment

The materials used include red ginger and aquadest. The main equipment used included microwave, condenser, thermometer, separating funnel, glass funnel, and a set of other glassware, analytical balance, pycnometer and refractometer brix. Characterization instruments used include Fourier transform infra-red (FTIR) and gas chromatography-mass spectrometry (GC-MS).

2. 2. Experiment design

The experiment was designed using four microwave power variations namely low (< 100 W), medium low (100–300 W), medium (300–500 W), and high (> 500 W). The planned extraction time was 60, 75, 90, 105, and 120 minutes.

2. 3. Stages of research

In this research, sample preparation is the process of preparing red ginger samples before the extraction process. The purpose of sample preparation is to remove the epidermis and impurities such as residual soil or red ginger roots that can interfere with the extraction and analysis process. At this stage, it is also necessary to consider the sample size and sample moisture content before the extraction process.

The extraction stage is the main process in extracting red ginger essential oil. The extraction of red ginger essential oil was carried out using a microwave. The essential oil produced will be collected in a separating funnel. At this stage, the things that need to be considered are microwave power and extraction time according to predetermined variables.

Analysis of red ginger essential oil is carried out to determine the physical characteristics of essential oil, namely density, refractive index, organoleptic in the form of aroma and color, carried out to compare with the Indonesian National Standard (SNI). In addition, analysis using Fourier transform infra-red (FTIR) to determine the functional groups of essential oil and gas chromatography-mass spectrometry (GC-MS) to determine the composition of essential oil. After that, an antibacterial test was carried out to determine whether or not there was an inhibition of essential oil against *S. aureus* bacteria.

3. Results and discussion

3. 1. The effect of time and power on increasing extraction temperature

The initial experiment was designed with four variations of microwave power, namely low, medium low, medium, and high. The temperature profile for each time interval at different power levels is shown in **Fig. 1**.

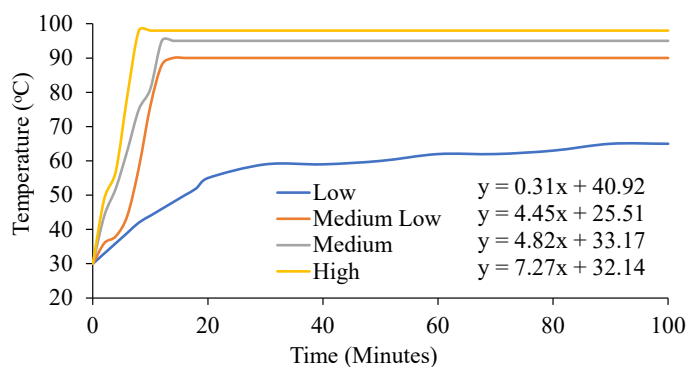


Fig. 1. Temperature profile by time for various microwave power

Fig. 1 shows the relationship of extraction time and microwave power to temperature increase. It is known that the temperature increases with the length of extraction time and will reach a constant temperature at a certain time. Low power experiences a relatively slow temperature increase compared to the other three powers, this is evident from the rate of temperature increase of $0.34^{\circ}\text{C}/\text{minute}$. The power will be constant at the 90th minute with a temperature of 65°C while medium low power is constant at the 14th minute with a temperature of 90°C , and medium and high power are constant at the 12th and 8th minutes with temperatures of 95°C and 98°C respectively. The results of this research are in accordance with the theory which states that power and time are directly proportional to the increase in temperature [6].

In this research, the microwave power that experienced the fastest temperature increase was High with power ≥ 500 W. However, in the SFME method, there are material characteristics that affect the extraction process so that high power does not necessarily produce the best yield. The material characteristics considered in this study are dry materials with a moisture content of 30–100%. Under these conditions, the maximum extraction time required for samples at medium and high power is 15 minutes, after which they will be damaged (burnt). Therefore, the appropriate power used in this study is low and medium low with extraction times of 60, 75, 90, 105, and 120 minutes.

3. 2. Functional groups of red ginger essential oil

The chemical bonds contained in red ginger essential oil are known through Fourier transform infra-red (FTIR) analysis. FTIR spectrogram of red ginger essential oil in the best variation of microwave power is low. Low microwave power can be seen in **Fig. 2**.

The FTIR spectrogram in **Fig. 2** implies the structure and functional groups of red ginger essential oil from the wavelength of $4000\text{--}650\text{ cm}^{-1}$ where there are 4 identified peak spectrum absorptions. There is an O–H group absorption at a wave number of 3257.7 cm^{-1} with strong intensity and wide shape. The O–H wave number interval is $3650\text{--}3250\text{ cm}^{-1}$ [7]. The absorption at wave number 1751.8 cm^{-1} describes the presence of carbonyl groups (C=O) such as ketones, aldehydes, esters, and carboxyls. This compound is in the interval of wave numbers $1850\text{--}1650\text{ cm}^{-1}$ [7]. In addition, absorption in the $1300\text{--}1050\text{ cm}^{-1}$ range also indicates the presence of carbonyl groups (C–O). The peak C–O wave number that appears on the spectrogram is 1297.1 cm^{-1} . On the other hand, the absorption at wave number 1617.6 cm^{-1} indicates the presence of alkene hydrocarbon compounds and aromatic compounds, namely in the wave number interval $1650\text{--}1600\text{ cm}^{-1}$ [7]. The presence of carbon-carbon double bonds (C=C) from the FTIR absorption results indicates that there are main compounds that make up red ginger essential oil such as zingiberene which is a class of sesquiterpene compounds [8].

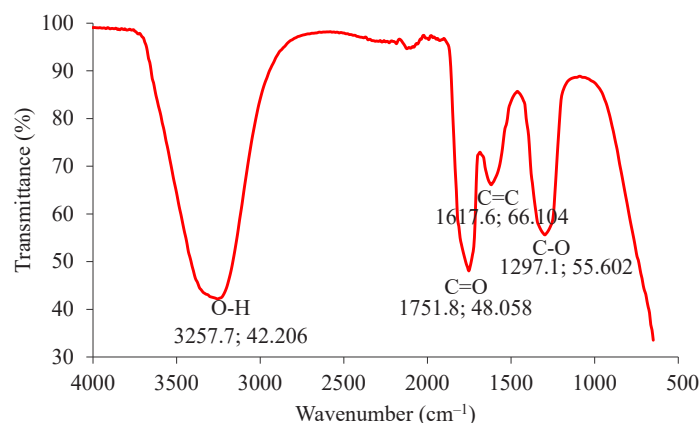


Fig. 2. Functional groups of red ginger essential oil

3. 3. The effect of time and power on red ginger essential oil yield

In this research, the highest yield was obtained at low and medium low microwave power of $1.90618 \pm 0.00807\%$ with an extraction time of 120 minutes and $1.92752 \pm 0.05736\%$ with an extraction time of 105 minutes, respectively. This yield is greater than other methods that have been studied with the same material, namely red ginger, by using the steam distillation method for 3 hours to produce a yield of 1% [7] and by using the hydrodistillation method for 4 hours to produce a yield of 1.55% [9]. The yield of red ginger essential oil at various power and time is shown in **Fig. 3**.

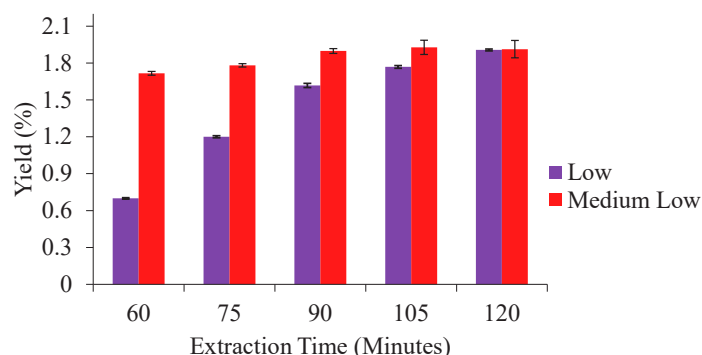


Fig. 3. The Effect of time and power on the yield of red ginger essential oil

Fig. 3 shows that medium low microwave power produces higher yields than low microwave power. Increasing the power can increase the energy received by the material in the form of heat energy, thus increasing the yield of essential oil produced. At various powers, from 60 minutes to 120 minutes, the yield increased. This is because water needs more time to take the compounds contained in red ginger. After the increase, a decrease in yield occurred at medium low power of 120th minute. This decrease is due to the fact that with the increase in extraction time at high power, the evaporation process is faster, resulting in the degradation of the extracted red ginger. This result is in line with research [10] which states that a significant decrease in yield is caused by the degradation of materials as the extraction time increases.

The superiority of the SFME method in extracting essential oils is not limited to red ginger. A study on the extraction of essential oil from fresh basil [11] also demonstrated the effectiveness of SFME. Using a power of 380 W and an extraction time of 80 minutes, the method yielded 2.373% essential oil, which is significantly higher than the yield obtained using the microwave-assisted hydrodistillation (MAHD) method (0.668%) under similar power but a longer extraction time of 180 minutes. Similarly, for red ginger, [9] reported that hydrodistillation of fresh red ginger produced the highest essential oil yield of $1.55 \pm 0.020\%$ after 4 hours of distillation. Despite using longer heating, this yield remains considerably lower than that achieved in the present study using SFME.

The results of two-way ANOVA (analysis of variance) statistical analysis using IBM SPSS Statistics 26 software showed a significance value of 0.000 for the effect of time and power on yield. Since this value is below the significance level of 0.05, it can be concluded that different extraction times and microwave power affect the yield values produced. Statistically, the extraction results in the SFME method with medium low power can provide more yield than low power.

3. 4. The effect of power on red ginger essential oil composition

Essential oils with the best yield and characteristics in each microwave power variation were tested for their composition using gas chromatography-mass spectrometry (GC–MS). Tests were carried out on red ginger essential oil with microwave power variations low 120 minutes and medium low 90 minutes with a compound content of 9 and 29 types of compounds, respectively. This is evidenced by the number of peak compounds that appear in the chromatogram **Fig. 4**.

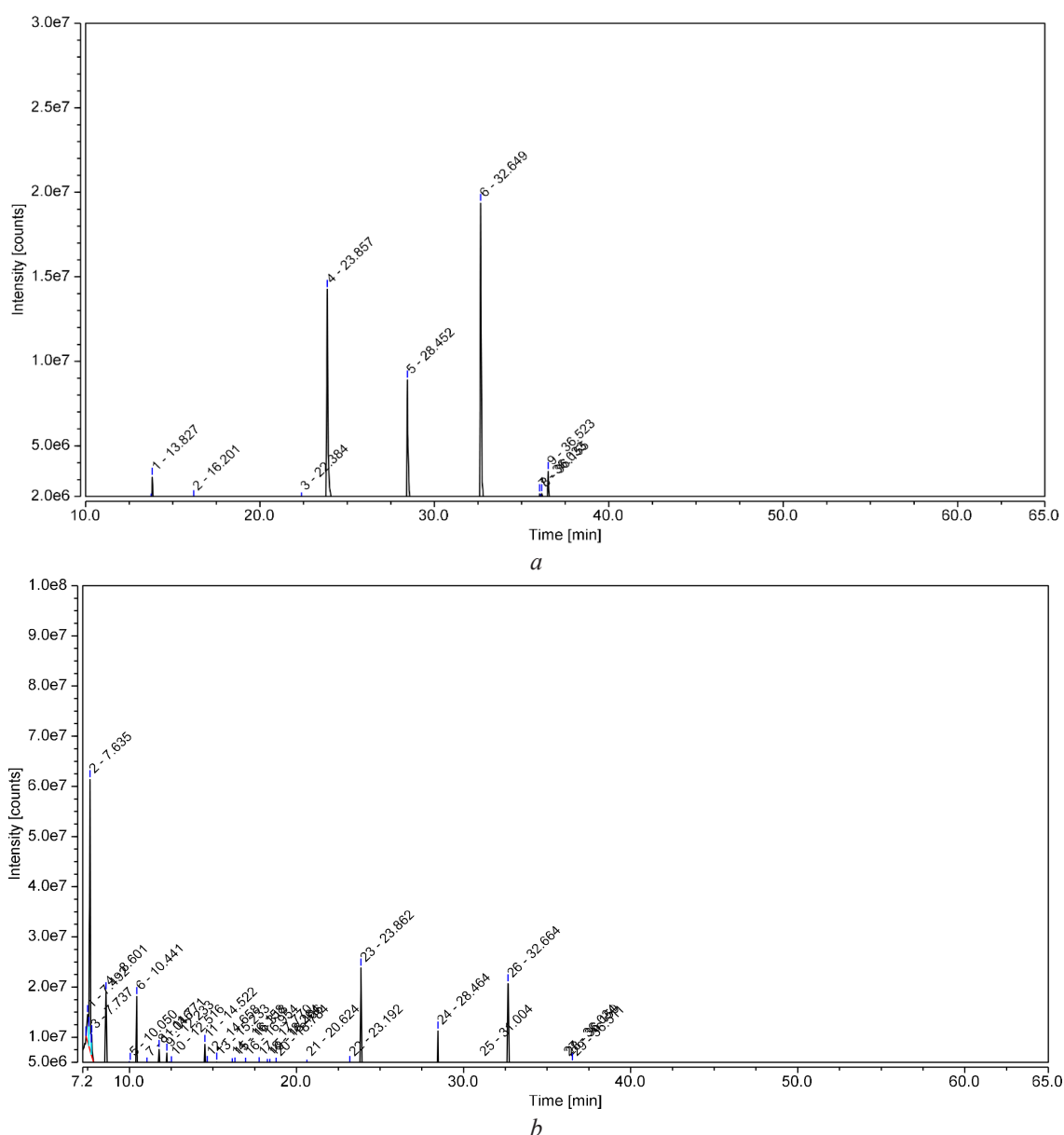


Fig. 4. Red ginger essential oil composition: *a* – low power; *b* – medium low power

One of the main components of red ginger essential oil is a compound from the terpenoid group, especially sesquiterpenes, called zingiberene. This compound is prone to decomposition

to form new compounds. This is due to its thermolabile nature that tends to decompose on simple or ordinary heating [12]. The presence of new compounds is influenced by several factors including temperature and pressure, as well as the water content in essential oils [13]. In this research, the extraction process used low (< 100 Watt) and medium low (100–300 Watt) microwave power with a maximum temperature at each microwave power of 65°C and 90°C, respectively. Meanwhile, zingiberene has a boiling point at 34°C at a pressure of 1 atm [12].

Several carboxylic acid compounds were identified as zingiberene derivatives from the GC-MS analysis, include hexadecanoic acid, dodecanoic acid, tetradecanoate acid and others. Furthermore, the group of carboxylic acid compounds (RCOOH) underwent substitution of hydroxyl (OH) groups into alkoxy (OR) caused by the use of methanol solvents in GC-MS analysis [14]. Similar compound types have also been observed in GC-MS profiles of red ginger essential oil. For instance, in paper [15] found n-hexadecanoic (64%), palmitic acid (8.36%), oleic acid (5.86%), and linoleic acid (4.32%) among the major components. The reaction scheme of ester formation from carboxylic acid and alcohol is shown in Fig. 5.

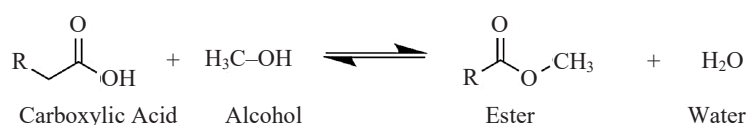


Fig. 5. Ester formation reaction scheme [16]

The alkyl group in methanol solvent directly bonds with the hydroxyl (OH) group found in carboxylic acids. This reaction results in the formation of methyl ester compounds in the GC-MS analysis. The ester compounds identified include hexadecanoic acid, methyl ester; dodecanoic acid, methyl ester; methyl tetradecanoate, and others. As a result, it is believed that oxidized zingiberene forms other compounds (carboxylic acids) is 96.09% at low power and 45.2% at medium low power. The compound thought to be zingiberene is classified as a terpenoid, known for its antibacterial properties [17]. Additionally, other sesquiterpene compounds were confirmed in the Low microwave power chromatogram (Fig. 4, a), such as cubenol, a sesquiterpene alcohol. In the medium low microwave power chromatogram (Fig. 4, b), the sesquiterpene hydrocarbon compound trans-Z-a-Bisabolene epoxide was also detected. These sesquiterpenes are known for their antibiotic and antimicrobial activities [4]. Furthermore, monoterpenes like limonene oxide, geranyl vinyl ether, and oxidized monoterpenes such as endo-borneol, cis-linalool oxide, and trans-linalool oxide were also identified [8]. These compounds serve as antiseptics and are used as flavoring agents in food and perfumes [4].

3. 5. Organoleptic properties of red ginger essential oil

The organoleptic properties of red ginger essential oil are known through visual observation, specifically its color and aroma. This observation is conducted to determine whether there are any abnormalities in the essential oil by comparing it to SNI 06-1312-1998. According to the SNI, red ginger essential oil has a light yellow to yellow color and a distinct ginger aroma. In this research, the color and aroma of the produced red ginger essential oil were light yellow and had the characteristic ginger aroma, which was in accordance with SNI 06-1312-1998. However, a change in color was observed when the extraction time increased with higher power, specifically at the medium low power setting. The resulting essential oil exhibited a yellow-brown color and a stronger aroma. In paper [18] reported that increasing power and distillation time results to a darker essential oil color and a more intense aroma, due to the extraction of a greater number of volatile components. Similarly, in paper [19] highlighted that beyond operational parameters, the oil's color and aroma are also influenced by the physical quality and moisture content of the raw material. Therefore, it can be concluded that the best treatment for producing red ginger essential oil that meets the quality standard is using low power.

3. 6. Density of red ginger essential oil

The density of red ginger essential oil according to SNI 06-1312-1998 is 0.877 to 0.882 g/ml. The density of the red ginger essential oil produced at various powers and times is shown in Fig. 6.

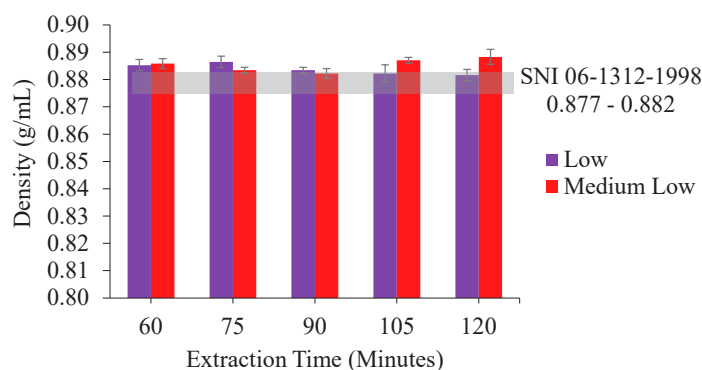


Fig. 6. Density of red ginger essential oil at various powers and times

Based on **Fig. 6**, the essential oil produced at Medium Low power for 90 minutes met the quality standard of SNI 06-1312-1998, with a density of 0.88225 ± 0.00182 g/ml. In addition, essential oils produced at low microwave power for 105 minutes and 120 minutes also met the standard, with densities of 0.88225 ± 0.00314 g/ml and 0.88164 ± 0.00209 g/ml, respectively. However, other treatments did not meet the SNI, as they contained heavier fractions. This is due to the specific gravity of a substance depends on the types and amounts of compounds it contains. Essential oils with a higher proportion of long-chain or oxygen groups tend to exhibit greater density, as reported by [20].

The results of the two-way ANOVA statistical test, conducted using IBM SPSS Statistics 26 software, showed a significance value of 0.078 for the effect of power on density. Since this value is above the 0.05 significance level, it indicates that microwave power does not have a significant effect on density. Similarly, the effect of extraction time on density showed a significance value of 0.066, also above 0.05, meaning there is no significant difference between extraction time and density. Therefore, it can be concluded that variations in extraction time and microwave power do not significantly affect the density of the essential oil. From an economic perspective, it is recommended to use lower power and shorter extraction times, given the results obtained in relation to the quality standard. In this research, the best extraction method that resulted in the optimal density of the essential oil was low power with an extraction time of 120 minutes.

3. 7. Refractive index of red ginger essential oil

The refractive index of red ginger essential oil according to the quality standard SNI 06-1312-1998, should be between 1.4853 and 1.4920. The refractive index of the red ginger essential oil produced at various powers and times can be seen in **Fig. 7**.

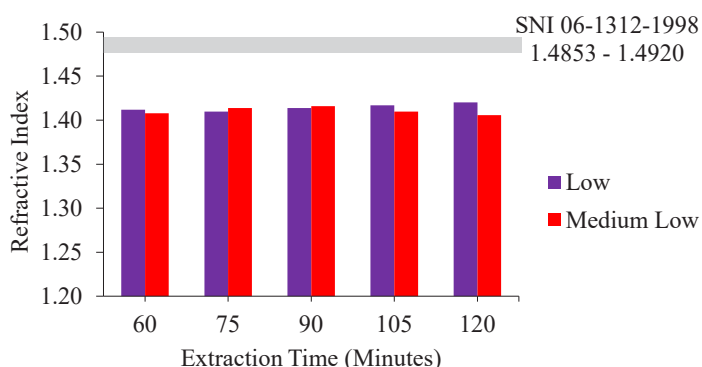


Fig. 7. Refractive index of red ginger essential oil at various powers and times

In this research, the refractive index values for all treatments did not meet the quality standard. The low refractive index values were due to the presence of water in the essential oil. These

findings are consistent with the research [21], which reported refractive index values for red ginger oil ranging from 1.372 to 1.421. This is further supported in paper [22], who observed that the essential oils with higher water content tends to have lower refractive index.

The results of the one-way ANOVA statistical test, using the IBM SPSS Statistics 26 software to determine the effect of power on the refractive index, showed a significance value of 0.178. Since this value is above the 0.05 significance level, it indicates that microwave power does not significantly affect the refractive index. Another one-way ANOVA test was conducted to determine the effect of extraction time on the refractive index. The significance value obtained was also above 0.05, at 0.901. This means that there is no significant difference between extraction time and refractive index. Therefore, it can be concluded that variations in extraction time and microwave power do not significantly affect the refractive index of the essential oil.

3. 8. Antibacterial activity of red ginger essential oil to *staphylococcus aureus*

The antibacterial activity was tested using the disk diffusion method, with the test sample consisting of red ginger essential oil extracted using Low power for 120 minutes and Medium Low power for 90 minutes. Based on the test results, the inhibition zones were similar for both low and medium low power treatments, measuring 7.6 mm and 7.4 mm, respectively. In comparison, in paper [23] reported that red ginger extracts obtained using ethyl acetate, 96% ethanol, and n-hexane exhibited inhibition zones against *Staphylococcus aureus* of 3.38 mm, 3.17 mm, and 3.70 mm, respectively. Interestingly, despite using organic solvents, the observed antibacterial activity was weaker, while the present study employed a solvent-free method achieved higher inhibition zones, indicating that SFME can yield more potent bioactive components even without solvent assistance. The antibacterial activity is classified as weak if the inhibition zone is < 5 mm, moderate if between 5–10 mm, strong between 10–20 mm, and very strong if > 20 mm. The inhibition zones of the red ginger extract to *Staphylococcus aureus* at low and medium low power are in the moderate category. The antibacterial activity is attributed to the presence of antibacterial compounds in the extract, which belong to the terpenoid class. Terpenoid compounds can inhibit bacterial growth by disrupting the formation of membranes or cell walls, preventing their proper formation or compromising their integrity [15].

3. 9. Limitations of the study and future directions

Several limitations must be acknowledged in this study. The extraction was conducted on a laboratory scale, which may not fully represent the performance or scalability of the process under industrial conditions. The optimization was limited to two parameters (extraction time and microwave power), while other factors such as particle size, raw material moisture content, and sample loading were not examined. Moreover, the red ginger samples were obtained from a single source, without considering potential variations in essential oil composition caused by geographical origin or seasonal factors. Lastly, the analysis focused only on the chemical composition of the selected extract, without assessing all extraction variations, which may have resulted in certain components remaining undetected.

Future research should focus on the integration of SFME with emerging green extraction technologies, such as ultrasonic assistance, to enhance the yield and selectivity of bioactive compounds from red ginger. In addition, investigating the scalability, stability, and shelf-life of the SFME process may offer insights into industrial-scale application. Finally, comprehensive profiling of volatile and non-volatile components using GC-MS/MS and LC-HRMS would further deepen understanding of compositional variations under different operating conditions.

4. Conclusions

The low microwave power (< 100 W) and medium low power (100–300 W), as well as extraction times (60, 75, 90, 105, 120 minutes), in this research had no statistically significant effect on the characteristics of red ginger essential oil, including density, refractive index, color, and aroma. The red ginger extraction using the SFME method in this research gave the best results in the Low power for 120 minutes. The composition of carboxylic acids, which are suspected to

come from zingiberene compounds, was 96.09%. The antibacterial activity test to *Staphylococcus aureus* showed that the red ginger essential oil obtained from low microwave power is included in the medium category, with an inhibition zone of 7.6 mm.

Conflict of interest

The authors declare that there is no conflict of interest in relation to this paper, as well as the published research results, including the financial aspects of conducting the research, obtaining and using its results, as well as any non-financial personal relationships.

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The study was performed without financial support.

Data availability

Manuscript has no associated data.

Use of artificial intelligence

The authors confirm that they did not use artificial intelligence technologies when creating the current work.

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Authors' contributions

Renita Manurung: Conceptualization, Methodology, Supervision, Formal analysis, Writing – review & editing.

Zahra Adelia Siregar: Investigation, Data curation, Resources.

Anefa Baroza: Investigation, Data curation, Formal analysis.

Alwi Gery Agustan Siregar: Writing – original draft, Formal analysis, Visualization.

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