The effect of ginger oil extraction using Microwave Assisted Hydro-distillation (MAHD) method on zingiberene content

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Abstract. The ginger (Zingiber officinale) essential oil has been produced using microwave assisted hydro-distillation extraction process and evaluated using GC-MS. The effects of different power of microwave (450, 600, 800 Watt) and material-solvent (w/v) ratio (1:8; 1:9; and 1:10) on the yield, density, and refraction index of ginger essential oil were investigated. The results indicated that microwave power and material-solvent ratio significantly affects yield, density, and zingiberene content of ginger essential oil. The highest yield was obtained in 600 Watt with 1:8 of material-solvent ratio (w/v), i.e., 1.71% w/w. The best density of ginger oil was obtained in 600 W with 1:10 of material-solvent ratio (b/v), i.e., 0.875 g/ml. The best refractive index value of ginger oil was obtained in 800 Watt with 1:8 of material-solvent ratio (b/v), i.e., 1.494. The refractive index of essential oils is influenced by the presence of carbon chains and double bonds in oil, free fatty acid levels, oxidation processes, and temperature. The best contents of ginger oil zingiberene were obtained in 600 Watt with 1:8 of material-solvent ratio (w/v), with 27.79%.

1. Introduction
Various kinds of spice plants have been developed in Indonesia; one of them is ginger (Zingiber officinale). Ginger is known as a spice plant that is widely used for a flavor enhancer in food and also used in the health field. Ginger is cultivated in almost all regions of Indonesia, with the highest amount of production reaching 313 064 300 kg in 2015 [1]. These production results make Indonesia one of the five major ginger exporting countries in the world, with average export value reaching 32.75% of the total production produced [2]. Exports of ginger are still done in the form of raw materials (fresh ginger) and semi-finished products such as pickled and dried ginger. The export of ginger in the form of oil and oleoresin is still small, ranging from 0.4% of the total exports of essential oils from Indonesia. Ginger essential oil is a commodity that has a high economic value of US $105/kg [3].

So far, the ginger essential oil industry in Indonesia has still not been able to meet the world standard ginger essential oil. Indonesian ginger essential oil has relatively low levels of zingiberene. Low levels of zingiberene are caused by conventional distillation processes, which generally use high temperatures. While the zingiberene compound is a thermo-labile compound that will decompose at high temperatures [2]. The levels of zingiberene in ginger essential oils can indicate the level of oil purity [3]. In addition, zingiberene is a natural antioxidant and functions as an antiviral, antimicrobial, and antifertility agent. Products containing zingiberene have been widely used as natural contraceptives, cosmetics, spices, pesticides, and so on. Information about the potential and great
The benefits of ginger is still needed alternative extraction processes to be able to produce ginger oil with high zingiberene levels. Considering the extraction of essential oils, micro-wave-assisted processes are highly desirable due to their small equipment size, simplicity, and rapid controllability through mild increments of heating. The main benefits of microwave assisted extraction are the decrease in extraction time and solvents used. The purpose of this study was to determine the effect of differences in microwave power and material: solvent (w/v) ratio in the Microwave-Assisted Hydro-distillation (MAHD) extraction process on yield, density, refractive index, and zingiberene levels in the resulting ginger essential oil.

2. Materials and methods

2.1. Material preparation

The material used is ginger (Z. officinale var. Amarum) obtained from the local market in Malang, distilled water, and sodium sulfate (Na2SO4). Tools used are a knife, stove, pan, spoon, analytical scales, measuring cup, separating funnel, glass bottles, stopwatch, tray dryer, blender, 32 mesh sieves, MAHD EMM2308X, digital ATAGO Pocket Refractometer PAL-3, and GC-MS Shimadzu QP2010.

2.2. Extraction

The extraction method used was microwave assisted hydro-distillation (MAHD). Ginger was manually sliced into ±2 mm of average thickness and cleaned manually using a smooth brush then dried using a tray dryer at 35°C for 8 h. Ginger that has been dried was size reduced and sieved for homogeneity size with 32 mesh sieve. The initial moisture content of dried ginger powder then measured using a standard gravimetric method. The MAHD extraction process was carried out by a combination of 2 treatment factors, namely microwave power 450, 600, 800 Watt and material-solvent (w/v) ratio, i.e., 1:8; 1:9; and 1:10. The MAHD extraction process was carried out at 100°C of temperature until the water runs out, and the ginger powder turns into a paste. After getting the distillate in the form of oil and water, the oil then separated. Furthermore, ginger oil is purified using sodium sulfate (Na2SO4).

2.3. Yield measurement

The measurement of the yield of ginger essential oil was measured using quantitative analysis, i.e., the mass of obtained essential oil per mass of dried ginger powder as raw material [4].

2.4. Density

The density of essential oil was measured using the standard method, i.e., measuring the mass of the oil at certain known volumes, according to Sutan et al. [5].

2.5. Gas chromatography-mass spectrometry (GC-MS)

Gas Chromatography-Mass Spectrometry (GC-MS) is one of the chromatographic techniques that can be used to detect volatile chemical compounds. The GC-MS test was carried out with the Shimadzu QP2010S GC-MS apparatus in the Analysis and Measurement Unit of the Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Brawijaya.

3. Results and discussions

3.1. Effect of extraction condition to extraction time

Based on data in table 1, it can be shown that the fastest extraction time is obtained at the 800 Watt with a ratio of 1:10 for 19 minutes 35 seconds. The higher microwave power implicates to shorter extraction time required due to heating. This condition occurs due to heat flow in the microwave more efficiently and evenly so that all samples can be heated simultaneously [6]. The longer extraction time will also affect the yield of essential oils. The longer extraction time can cause degradation of components in the material and can cause undesired evaporation of volatile components in oil. The microwave that operates at 450 Watt requires a longer extraction time. This can be attributed to the
effect of microwaves on polar (water) solvents, which have high dielectric constants. Therefore, low microwave power tends to extend induction time because the wave density is also low at that power [7].

| Power (Watt) | Material: solvent ratio (b/v) | Solvent Volume (mL) | Temperature (°C) | First Drop Time (min) | Extraction time (min) |
|-------------|-------------------------------|---------------------|------------------|------------------------|-----------------------|
| 450         | 1:8                           | 400                 | 100              | 9′58″                  | 31′07″                |
|             | 1:9                           | 400                 | 100              | 10′08″                 | 36′32″                |
|             | 1:10                          | 400                 | 100              | 10′11″                 | 39′13″                |
| 600         | 1:9                           | 360                 | 100              | 6′50″                  | 25′20″                |
|             | 1:10                          | 400                 | 100              | 7′42″                  | 27′32″                |
| 800         | 1:9                           | 400                 | 100              | 5′05″                  | 20′22″                |
|             | 1:10                          | 400                 | 100              | 5′04″                  | 19′35″                |

3.2. Yield of essential oil

The yield of essential oil data is presented in figure 1, which shows that the yield of a ginger essential oil varies in each treatment. The highest yield was obtained for the 600 Watt of microwave power with a material-solvent (w/v) ratio of 1:8, i.e., 1.71% w/w. The low yield is most likely due to the use of too long extraction, causing the components in the material to degrade. The yield of ginger oil can also be influenced by several factors, including the process of handling raw materials (slicing, powder fineness, drying process), extraction time, type of solvent, extraction temperature, and method of extraction process [8]. The irregular yield of essential oils in this study was also caused by the different mass factor of the material in each material-solvent ratio.

![Figure 1. Yield of ginger essential oil as affected by MAHD extraction condition](image-url)
The results of essential ginger oil obtained in each treatment as a whole are still above from previous studies 0.4 w/w Anca Racotia [14]. The combination of the amount of microwave power and the ratio of the mass of ginger and solvent can increase the yield of ginger essential oil produced.

3.3. Density of essential oil
Determination of density ginger oil is one of the major properties of the essential oil quality. According to the International Standards of Essential Oil Association (EOA), the density of ginger oil (at 20°C) ranges from 0.870 to 0.882 g/ml. The results of measurement indicated that the density of ginger oil from various treatments was ranged between 0.481 to 0.975 g/ml (figure 2). Based on the mentioned standard of ginger oil, the density value of ginger essential oil that meets the criteria was produced with 600 Watt of microwave power with 1:10 of material-solvent ratio (b/v), i.e., 0.875 g/ml. The difference in the value of the density of essential oils can be caused by several factors, e.g., the type and number of components contained in oil [8]. In addition, the more double bonds in oil, the lower density of oil. The decrease in density due to the number of double bonds can be caused by heating at high temperatures [5].

![Figure 2. Density of ginger essential oil as affected by MAHD extraction condition](image)

3.4. Refractive index of essential oil
Refractive index is the value of the ratio between the speed of light in vacuum and the speed of light propagation in a medium. The refractive index of a liquid is determined by the density of its molecules. Temperature affects the density of the liquid, so the temperature affects the refractive index of the liquid [9]. According to the International Standards of Essential Oil Association (EOA), the refractive index value of ginger essential oils is in the range of 1.480-1.494 at 20°C. The results of the refractive index value of ginger essential oils from various MAHD treatments can be seen in figure 3.

Based on the standard refractive index of ginger essential oils, there were several treatments whose refractive index values meet the standards, i.e. 450 W with 1:10 of material-solvent ratio (b/v) (1.492); 600 W with 1:8; 1:9; and 1:10 of material-solvent ratio (b/v) (1.491; 1.492 and 1.491, respectively); and 800 W with 1:8 of material-solvent ratio (b/v) (1.494). The refractive index of oil greatly affects the quality of the essential oils. The refractive index of essential oils is closely related to the components in the resulting essential oils. The more long-chain components are distilled, the density of essential oils will increase, and the light coming will be more difficult to refract. These facts result in a greater refractive index for oil. In addition, the refractive index value is also influenced by the
presence of water in the oil content. The more water content, the smaller the refractive index value. This is because of the nature of water that easily refracts the incoming light. Thus, essential oils with a greater refractive index value will have better quality than essential oils with a small refractive index value [10].

![Figure 3. Refractive of ginger essential oil as affected by MAHD extraction condition](image)

3.5. GC-MS results
Analysis of the components of chemical compounds in essential oils is an important part of determining the quality of the essential oils. The best GC-MS results were obtained in 600 W with 1:8 of material-solvent ratio (b/v), which can be seen in figure 4. Components of ginger essential oil chemical compounds in the mentioned treatment include curcumene (38.42%), zingiberene (27.79 %), ethylene-D4 (-1.89%), β-bisabolene (13.38%), and β-sesquiphellandrene (22.30%).

![Figure 4. GC-MS results of ginger essential oil in 600 W with 1:8 of material-solvent ratio (b/v)](image)
Based on the GC-MS test results, it can be seen that the higher the power, the more damaged the cell wall so that the compounds that come out are not only oil but also the dyes contained therein. Extraction efficiency with microwave energy increases with increasing power [11]. Radiation from microwaves in the material creates heat with water molecules, which speeds up many reactions, especially reactions through carbocation as an intermediate and breaks down essential oils. Microwaves help shorten the extraction process time, but without causing significant effects on the composition of essential oils [12]. The composition (quantity and quality) of the essential oil chemical compounds produced can be influenced by various factors, including harvest time, different climatic and geographical conditions, and the state of the rhizome (fresh or dry). In addition, the extraction factor also influences the yield of essential oils, namely solute: solvent ratio and extraction time [13].

4. Conclusions

Microwave power and material-solvent ratio significantly affect yield, density, and zingiberene content of ginger essential oil. The highest yield was obtained in 600 W with 1:8 of material-solvent ratio (b/v), i.e., 1.71% w/w. The best density of ginger oil was obtained in 600 W with 1:10 of material-solvent ratio (b/v), i.e., 0.875 g/ ml. The best refractive index value of ginger oil is obtained in 800 W with 1:8 of material-solvent ratio (b/v), i.e., 1.494. The refractive index of essential oils is influenced by the presence of carbon chains and double bonds in oil, free fatty acid levels, oxidation processes, and temperature. The best contents of ginger oil zingiberene were obtained in 600 W with 1:8 of material-solvent ratio (b/v), with 27.79%. Based on these results, it can be stated that microwave assisted hydro-distillation (MAHD) is the right method for processing ginger into essential oil.

5. References

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