COMPARISON PROCESS OF SOLVENT-FREE MICROWAVE EXTRACTION AND MICROWAVE HYDRO-DISTILLATION METHODS IN ESSENTIAL OIL PRODUCTION FROM CABBAGE ROSES FLOWER (*Rose* × *centifolia*)

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Abstract

Indonesia has many diverse natural resources that still cannot be utilized optimally such as the extraction of essential oil from Cabbage Roses flowers (*Rose* × *centifolia*). This study compares the extraction amendments, extraction time, and essential oil composition of fresh rose flowers obtained by the Solvent-Free Microwave Extraction (SFME) method to those obtained using the Microwave Hydro-Distillation (MHD) extraction method. Optimum conditions using the same amount of raw material in this study for the MHD method optimum conditions of operation: time of 90 minutes; the ratio of 1 g/mL; the power of 450 W, obtained a yield of 0.152% and SFME method optimum conditions of operation: time of 90 minutes, ratio 0.2 g/mL; power 450 W, obtained a yield of 0.358%. Electricity consumption to obtain 1 g of rose essential oil extracted using the MHD method is 4.087 kWh, while for the SFME method, it is 1.073 kWh. So it can also be said that the MHD method requires electricity consumption which is 3.8 times higher when compared to the SFME method.

***Keywords**: essential oil, extraction, MHD, rose, SFME*

Introduction

Indonesia has many diverse natural resources that still cannot be utilized optimally. Among these biodiversities, plants producing essential oils that until now have not been able to be utilized optimally. More than 40 types of essential oils that are traded around the world are produced in Indonesia and some of these essential oils are Indonesia's leading products in the world market (Alighiri et al., 2017). Biologically active composites (BACs) deduced from natural sources in essential canvases have salutary impacts on mortal health (Giacometti et al., 2018).

Figure 1. Cabbage roses flowers (*Rose* × *centifolia*)

Of the many essential oils that are traded throughout the world, rose essential oil is one of the many essential oils that are in great demand and has high economic value. Roses are a type of shrub, included in the genus Rosa and the Rosaceae family. This flower is a resource that has many benefits (He et al., 2020). The benefits of rose essential oil are numerous, one of which is that it can be used to relieve a few spasmodic problems in the digestive tract, dyspepsia and relieve muscle pain, and peripheral circulation disorders (Ribeiro-Santos et al., 2015). Rose flower essential oil can also be used as an anti-diabetic, anti-inflammatory, antioxidant, and anti-cancer related to rose flower extract derived from flavonoids, triterpenes, phenolic acids, and diterpenoids. In addition, several studies have stated that rose BAC has antidepressant, anti-inflammatory, and antidiabetic effects, antifungal activity, antiulcerogenic and antithrombotic. (Giacometti et al., 2018).

The improvement of recent separation strategies for the food enterprise has these days obtained a variety of interest because of environmental restrictions, wastewater reductions, and the want to decrease electricity costs. There is likewise a steady call to enhance the excellent of vital oils as customers call for those traits of their meals, pharmaceutical, or perfumery products. Essential oil is a complicated combination of risky materials which might be usually gift at low concentrations (Kusuma and Mahfud, 2016). Today, the main method for producing essential oils, especially rose essential oil, is the traditional hydrodistillation (HD) method. This method is very time-consuming, the energy intake is large, and the output is low, ensuring an inefficient extraction (Feng et al., 2021). Using solvent extraction, it's miles not possible to obtain a solvent-free product and this procedure normally consequences withinside the lack of highly unstable components. These deficiencies have caused attention to the use of recent strategies withinside the extraction of essential oils (Kusuma and Mahfud, 2016). Therefore, another alternative method is needed to produce rose essential oil by identifying and replacing the old conventional extraction system with a more efficient and environmentally friendly rose essential oil extraction method.

The use of the rose flower extraction method using the Solvent Free Microwave Extraction (SFME) method has emerged as a method that has the opportunity to become a breakthrough in the extraction of rose essential oil because it is considered environmentally friendly and more efficient among the different methods for the process of extracting rose essential oil from plant material. The primary precept of this approach is that the warmth switch supplied with the aid of using microwave radiation affects fast growth withinside the temperature of the material, inflicting the growth of the plant's molecular shape and the rupture of the molecular wall (Idris et al., 2020). in the SFME method the extraction process is carried out by inserting the material directly into the microwave without adding the slightest solvent or water in the extraction process (Kusuma and Mahfud, 2016). Meanwhile, in the extraction process using the Microwave Hydro-Distillation (MHD) method, a solvent or water is required as an extracting agent or auxiliary in the advanced extraction process using a microwave (Moradi et al., 2018). SFME combines microwave heating with a distillation system that has a cooling system outdoors the microwave oven condenses distillate continuously. The water remaining in the extraction process can be returned or flowed back into the extraction vessel to assist the extraction process in the refining flask in the microwave. The critical oil may be carried via way of means of intrinsic water in flower cells to a separate funnel. This technique is operated at barometric pressure (He et al., 2020).

This study compares the extraction time, extraction amendment, and essential oil composition of fresh roses obtained by the SFME method to those obtained using the MHD extraction method. In addition, an analysis of electric power consumption and CO_2 emissions is used to compare the efficiency of the rose essential oil extraction process with SFME and MHD.

Research Methods

Materials

The flower sample used was the Cabbage Roses (*Rose* × *centifolia*) which was taken from Karangpring Village, Sukorambi District, Jember Regency, East Java. N-hexane (Merck), aquades.

Instrumentation

Analytical balance (OHAUS PA224), statif and cool, blender, knife, filter paper, tissue, test tube, liebig condenser, adapter, split funnel, erlenmeyer 100 mL, measuring flask, cuvette, tape, aluminum foil, measuring cup 50 mL, 100 mL, beaker glass 100 mL, 250 mL, pipettes drip, vial bottle, the distiller used is made of 1 liter round base flask, microwave (Electrolux Microwave model EMM2308X).

Procedure

Extraction process

The extraction process uses a type of Microwave tool (Electrolux model EMM2308X) with a maximum power of 800 W. A 1000 mL distiller made from a Pyrex two-neck round base flask connected through holes to a three-way connector and a Liebig condenser (Azmir et al., 2013).

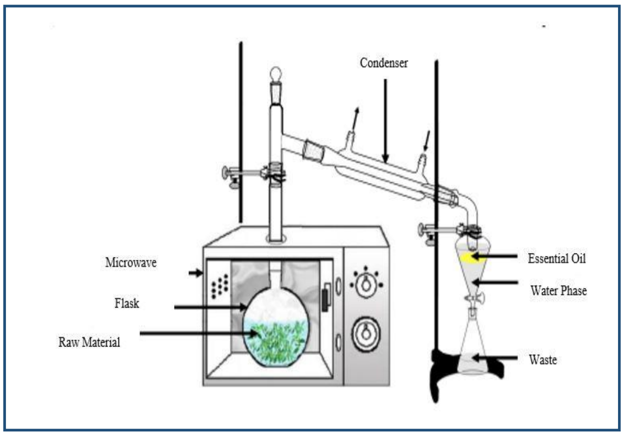

Figure 2. Extraction process using microwave (Azmir et al., 2013)

The raw materials that have been through sample preparation are weighed as much as 50, 100 and 150 grams, then put into a reaction flask that has been prepared according to a predetermined volume and heated with microwave irradiation according to a predetermined variable. During this process, the vapor condenses past the condenser outside the microwave cavity through which it flows (Azmir et al., 2013).

1) Microwave Hydro-Distillation (MHD) Extraction Method

   The raw material is weighed according to the predetermined ratio to solvent (0.75; 1; 1.25 g/mL). Then the extraction tool is assembled and put the raw materials that have been weighed in the distiller and then add a solvent (water) of 200 mL. Water is added to the Clevenger for the cohobating process. Drain water in the cooling system (Clevenger and reflux condenser). The microwave is turned on so that the distiller that has been filled with raw materials and solvents gets exposed to microwave radiation according to operating conditions and research variables. The extraction process starts from the first droplet of condensation to a predetermined time on the variable. When finished the hexane and oil are separated from the water by means of a separator funnel. To separate n-hexane from the oil used heat from hot plates. The essential oils obtained are weighed using an analytical balance sheet (Ghazanfari et al., 2020).

2) Solvent-Free Microwave Extraction (SFME) Method

The raw materials are weighed according to the predetermined ratio to the distiller flask (0.15; 0.2; 0.25 F/D). Then the extraction tool is assembled and put the weighed raw materials on the distiller. Water is added to the Clevenger for the cohobating process. Drain water in the cooling system (Clevenger and reflux condenser). The microwave is turned on so that the distiller that has been filled with raw materials and solvents gets exposed to microwave radiation according to operating conditions and research variables. The extraction process starts from the first droplet of condensation to a predetermined time on the variable. When finished the hexane and oil are separated from the water by means of a separator funnel. The essential oils obtained were weighed using an analytical balance sheet and collected in vials, n-hexane (C_6H_{14}) for the water separation process in a split funnel. The heating process is carried out to evaporate the mixture of oil and n-hexane, then weighing vials and oil bottles are carried out. The results of the extraction of essential oils obtained were analyzed (Li et al., 2012).

Rose Essential Oil Extraction Method Analysis

1) Measurement of essential oil yield of rose cabbage *(Rose × centifolia)*

The resulting essential oil is put into a vial bottle to be weighed and the yield is calculated with Eq. (1) (Azmir et al., 2013).

$$Yield\ (\%) = \frac{\text{Mass of oil obtained (gr)}}{\text{Raw material mass (gr)}} \times 100 \qquad (1)$$

2) Analysis of electrical energy consumption of the essential oil extraction process

The energy intake of various extraction strategies is calculated primarily based totally at the impact of energy intake and extraction time. Such equations are referred to in-text as Eq. (2).

$$Ec = \frac{Pt}{3600000} \qquad (2)$$

Where Ec is the electronic consumption (kWh), P is the power intake of the microwace during the extraction process (W), and t is the long time of the extraction process (s). In addition, the relative electricity consumption of the extraction method can be expressed in Eq. (3).

$$E * c = \frac{Ec}{m} \qquad (3)$$

Where E * c is the relative energy consumption (kWh/g) and m is the mass of cabbage rose essential oil obtained (g) (Kharisma Putri et al., 2019a).

3) Analysis of CO_2 emissions from the essential oil extraction process

Measurement of CO_2 emissions from the extraction process of rose cabbage essential oil was carried out based on the method mentioned in the previous study: To obtain 1 kWh of power from fossil fuels or coal, 800 g of CO_2 can be released into the environment during combustion. So the CO_2 emission is described by Eq. (4).

$$ECO_2 = \frac{Ec\ 800}{1000} \quad (4)$$

Where ECO_2 is the emission (g) and Ec is the electricity consumption from the extraction process using each method (kWh). Meanwhile, the relative CO_2 emission from the different methods of extracting rose cabbage essential oil was calculated according to Eq. (5).

$$E * CO_2 = \frac{ECO_2}{m} \quad (5)$$

Where $E * CO_2$ is the relative CO_2 emission (kg/g) and m is the mass of cabbage roses essential oil obtained (g) (Kharisma Putri et al., 2019a).

Results and Discussion

Essential oil yield of rose cabbage (Rose × centifolia) using SFME and MHD

In the extraction of essential oil, the usage of the MHD method and the choice of appropriate solvents could make the extraction technique run extra efficiently. The choice of this solvent itself additionally relies upon numerous matters along with the solubility of the additives to be extracted, their penetrating cap potential, and interplay with the matrix of the pattern or material, in addition to the dielectric constant (dielectric constant) (Chen et al., 2008). In the assessment of extraction, the use of traditional methods, the extraction of important oil of rose cabbage flowers (*Rose* × *centifolia*), the use of the MHD approach of solvent choice were critical to getting the most fulfilling yield. This is due to the fact withinside the extraction of important oil of rose cabbage (*Rose* × *centifolia*) the use of the MHD approach solvent choice additionally desires to not forget the capability of the solvent to take in microwave power and its heating ability (Routray and Orsat, 2012a). Based on research that the highest yield content using MHD method is 0.152% in samples with parameters at extraction conditions time of 90 minutes, microwave power is 450 watts, and the ratio of raw materials to solvents is 1 g/mL.

Figure 3. Rose extract results using the SFME method

The operating precept of the SFME approach of microwave-assisted extraction approach is that microwave propagation hits the pattern inflicting the in-situ water withinside the plant molecular to be inspired to rotate beneath neath microwave irradiation in order that the inner consequences are direct withinside the next strain boom in the plant molecular, which results in the breakdown of the molecular wall and the discharge of goal molecules (Kusuma and Mahfud, 2016). The extraction of bioactive compounds in Cabbage Roses (*Rose* × *centifolia*) is carried out by means of which the first sample that has been weighed will be chopped by cutting into smaller sizes. Sample enumeration aims to facilitate the process of inserting material into the distiller flask and to enlarge the area of the contact area of the material. The chopped sample is then put into a 1000 mL distiller flask. After being put into the distiller flask, it is then extracted using the SFME method with predetermined process parameters. The extract results are separated using hexane, this aims to separate the active ingredients with the possibility of water in the separation of the material that has been separated from the Clevenger. Furthermore, the essential oil produced is put into a vial bottle to be weighed and the yield is calculated. Based on research that the highest yield content using SFME is 0.358% in samples with parameters at extraction conditions time of 90 minutes, microwave power of 450 watts, and the ratio of raw materials to distillers of 0.2 g/mL.

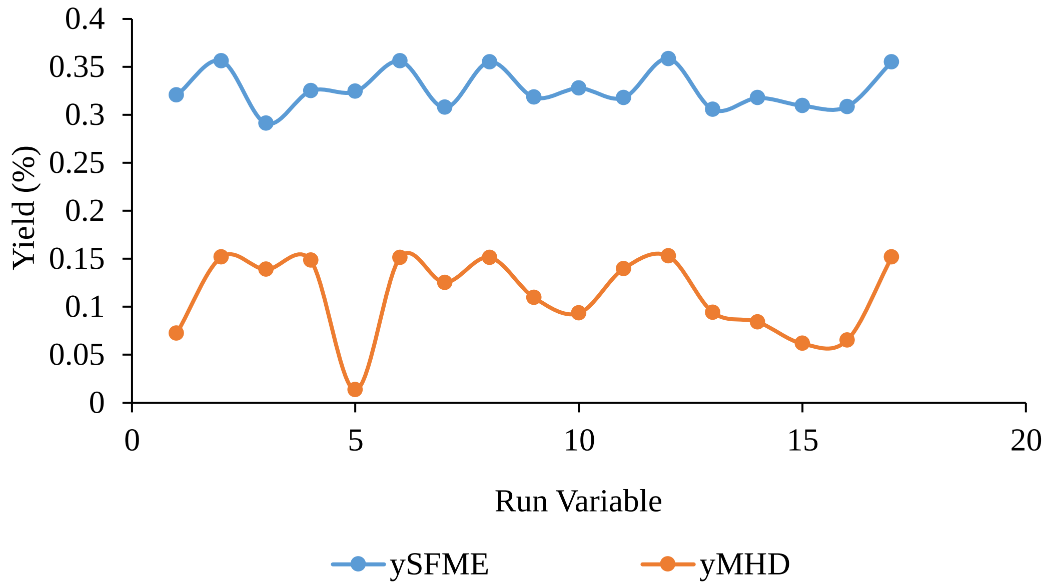

Figure 4. SFME and MHD extraction yield comparison chart

Based on Figure 4., with the same run variables as time, effort, and amounts of raw materials the yield of rose cabbage (Rose × centifolia) obtained by the SFME method achieved a higher yield compared to the MHD method. As seen in Figure 4., run variable 3 with a time of 60 minutes; 450 Watt of power, and 250 grams of raw material mass extracted by the MHD method only produces a yield of 0.013% whereas if it is extracted using the SFME method it produces a higher yield of 0.324%. The upward trend in yield in the SFME method is faster than the upward trend in yield in the MHD method. This is because microwave extraction provides a rapid heating effect because it directly heats the water in-situ efficiently and homogeneously, rapid heating of the in-situ water and also the matric on the cabbage rose flower (*Rose × centifolia*) causes internal overheating which results in the rupture of the cell wall or oil bag which ultimately facilitates the diffusion of essential oils out of the matrix (Rou tray and Orsat, 2012). In addition, in the SFME extraction process, there is a synergy between mass transfer and heat transfer from the inside out due to internal overheating so that the extraction process is faster than the MHD method because in the MHD method the heating of the material occurs from the outside to the inside due to the presence of water solvents. The presence of solvents also provides a longer heating effect due to a greater heating load than the extraction process in the absence of solvents. The yield from extraction using the SFME method is greater than the MHD method also occurred in previous studies. Based on research conducted by Ditta and Intan (2016) the extraction of essential oil from Kananga flowers using the SFME method using fresh ingredients with a mass of 100 grams and a power of 380 W for 60 minutes, a yield of 4.179% was obtained. In accordance with Figure 4., there is instability in the yield of essential oils using the SFME method, this is because if the power given is too large, it can cause oil degradation to reduce the yield value (Kusuma dan Mahfud, 2015). In addition, it was stated that the factors that affect the stability of essential oils are the presence of heat, light, and air. So, during the extraction process, oil degradation may occur due to overheating (Kusuma and Mahfud, 2017).

Analysis of electrical energy consumption and environmental impact of extraction of Rose × centifolia essential oil using SFME and MHD methods

The extraction of rose plants with the aid of using a solvent-free microwave extraction approach takes a quicker time in comparison to the microwave hydro distillation approach. This extraction time is associated with the value and hard work required. So, to discover, an evaluation of strength intake and environmental effect of essential oil extraction the use of solvent-unfastened microwave extraction approach and microwave hydro distillation approach changed into carried out.

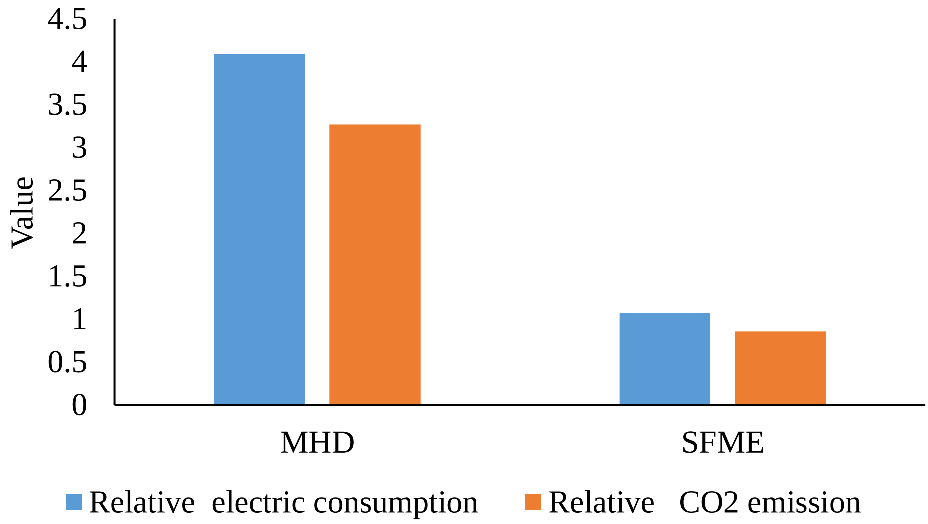

Figure 5. Comparison of electrical energy consumption (kW h g^{-1}) and CO_2 emission (kg g^{-1}) of MHD and SFME methods

In this study, the common want for relative power intake (kW h g^{-1}) for the extraction of crucial oil of Cabbage Roses with the MHD technique changed into 4.087 kW h g^{-1}, at the same time as for the SFME technique it changed to 1.073 kW h g^{-1}. So, it may be stated that the usage of the MHD technique calls for a relative power intake 3.8 instances greater in comparison to the SFME technique. The power intake to achieve 1 g of rose oil extracted from the usage of the MHD technique is 4.087 kWh, at the same time as for the SFME technique its miles are 1.073 kWh. So, it may additionally be stated that the MHD technique calls for power intake that is 3.8 instances better whilst in comparison to the SFME technique.

The amount of CO2 emissions resulting from the extraction process of rose cabbage essential oil can illustrate the environmental effects caused using the method used. If 1 kWh of electricity comes from fossil fuels or coal, 800 g of CO2 can be released into the environment at any given stage in combustion (Kharisma Putri et al., 2019). The common relative CO_2 emissions because of the extraction of vital oil from rose cabbage with the MHD technique is 3.27 kg g^{-1}, whilst the SFME technique is 0.859 kg g^{-1}. It may be stated that the extraction of Cabbage Roses essential oil through the MHD technique produces a more quantity of CO_2 emissions than the SFME technique. In the extraction process to obtain 1 gram of rose cabbage essential oil, the CO_2 emissions produced by the MHD and SFME extraction methods were 3.27 and 0.859 kg. So, it was concluded that the process of extracting Cabbage Roses essential oil using the SFME technique produces less CO_2 emissions compared to the MHD technique. Therefore, in the production of rose cabbage essential oil using the SFME method is a very environmentally friendly strategy.

Conclusions

In the process of producing Cabbage Rose essential oil (Rose × centifolia) the yield obtained by the SFME method was higher than the MHD method with the same time, power, and time variables under the t condition using the SFME method, namely 90 minutes, 0.2 g/ mL ratio; power of 450 W obtained a yield of 0.358%. Electricity consumption and emissions of CO_2 to obtain 1 g of rose flower essential oil extracted using the SFME method are more efficient and environmentally friendly with an electricity consumption value 3.8 times smaller than the MHD method and CO_2 emissions produced by 0.859 kg.

Suggestion

Further research should be carried out on the influence of parameters from the extraction of Cabbage Roses (*Rose × centifolia*) with the limitation of wider variations. The variation in the ratio of the material, time, and power is true and the yield produced has seen the difference. However, it still requires wider variation because it will influence variation more clearly.

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Conflict of Interest

The author has no conflict of interest to declare that is relevant to the contents of this article. And provide data that match the facts with the results of the research that has been done.

Author Contributions

All authors contributed throughout the research. The entire research flow from the preparation of the material to the analysis was carried out by Syah Sultan Ali Muzakhar and Lutfi Rizki Fauzi. Writing and revising the manuscript were carried out by Syah Sultan Ali Muzakhar, Lutfi Rizki Fauzi, and Ditta Kharisma Yollanda Putri. All authors read and revised the manuscript until it was approved.

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