

Extraction of Rose Hybrid Tea Oil with Microwave Assisted Integration of Mahd as a Diffuser

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Abstract – Rose flower have rich compound of oils that are used in many things for example in parfuem industries. Rose flower contains rich of oils fragrance such as eugenol, citronellol, geraniol, nerol, and linalool. This compounds has been used in variety of parfuem and many application has been used such as air freshener, softener, and diffusier. The objective of this research was to find out how to separate essential oils from rose flowers with Microwave Assisted Hydro Distilation and test them as diffusers and to find out the characteristics of essential oil. The extraction process takes place with two variables independent for time and power of extraction with their respective combinations of power: time (watt:minutes) namely; (300/15, 300/25, 300/35, 400/15, 400/25, 400/35, 500/15, 500/25, 500/35). The sample was prepared by make a solution with ratio of solvent (water distillation) and rose petals repeatedly 3:1 (v/m). During the research, an analysis of the yield of extract rose was carried out with a best result of 0.012% and the density produced 1,096 gr/ml. Organoleptic test was obtained with colored white limestone and typical odor of essential oil of rose flowers.

Keywords: red roses, microwave assisted extraction, essential oils

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INTRODUCTION

A diffuser is a device that converts essential oils into fragrance or aromatherapy vapour so that they spread in the air and are easy to inhale (Hasanah & Lestari, 2023). The spread of scent is a vital necessity because of its calming effect on the mind. Therefore, fragrances are also known as complementary drugs. Essential oils are also called essential oils because they resemble ether. In international use, this oil is called an essential oil because of its unique ability to give aroma/smell. Essential oils are chemically non-polar and consist of a mixture of different compounds that produce certain odor effects. Most essential oils belong to a group of organic compounds that are soluble in oils (lipofilik) (Utomo et al., 2023). Maintaining a balance between supply and demand requires the continuous expansion of the availability of essential oils.

Indonesia has a lot of tropical plants that are rich in essential oils. Nearly 80 species of plants are traded throughout Indonesia and the world. However, there are still many plants that have not been used as essential oil raw materials. Roses are one of the kinds of plants that need to be explored because of their economic value and potential essential oil content (Utomo et al., 2023). The rose is one of the plants whose leaves are suitable for the use of oil because it has an aromatic and durable aroma. Essential oil content by distillation and enfleuration of 0.9% - 5% consisting of citronelol, geraniol and methyl eugenol (Utomo et al., 2023).

Extraction methods are commonly used to extract bioactive compounds from various plants because they have advantages such as, high drying rate, high energy efficiency, little solvent used and fast extraction time. Dipole extraction between water molecules and solvents in the microwave causes the temperature and pressure to rise, resulting in a diffusion process from the sample to the solvent at a fairly high speed (Sari, 2020). After the extraction process, the material will be separated in the principle of distillation process. The microwave separation process will emit radiant heat that can be transmitted, reflected or absorbed depending on the reaction caused by the interaction on a sample. The waves are transmitted by the waveguide (a component used to emit and direct waves) and the waves are reflected off the walls of the oven chamber and absorbed by the plant sample. Plant material stored in a flask (flash), a translucent clear container will be penetrated by microwave radiation and will be absorbed by the grandular glands and vascular system of the cell wall. This event will cause heat and cell walls to break, so that the oil in the plant can come out (Effendy et al., 2020).

Available methods of extraction of essential oils from plants such as soxhlet extraction, steam distillation, ultrasonic wave extension, supercritic al CO2 extraction and microwave extraction (Souiy, 2023). Microwave Assisted Extraction becomes a method that has great yields, cheap, and environmentally friendly (Putra et al., 2023). This study will discuss the characteristic of the Microwave Assisted Extraction (MAE) method integrated with Microwaves Assisted Hydro Distillation (MAHD) in the extraction of essential oils from rose flower beetles using water as a solvent (Ghazanfari et al., 2020). The future of this scientific article could provide strategies for methods and sources of alternative essential oil materials as a diffuser product (Hakim, 2019).

METHODOLOGY

Method and Time of Implementation

The research was carried out on an ongoing basis with the health protocol in the Laboratory of Industrial Chemical Engineering Technology Studies, Department of Industrial Technology, School of Vocation, Diponegoro University in a two-month period by 2023. The design of experiments are used factorial design with two variable independent.

Material and Equipment

The equipment used in the study included, a set of Microwave Assisted Hydro Dystilation (MAHD) instruments, a round clove, a digital balancer, a picnometer, a beaker glass, a glass mixer, a gauge glass, and a Clevenger apparatus. While the materials used include, rose flowers and aquadest.



Figure 1. (a). Rose flower sample (b). MAHD Apparatus

Yield Level Test

The yield test is the weight determination of the extract produced after the extraction process. The greater the yield, the better the result. To test the yielt, the mass of the essential oil obtained is divided by the mass from the beginning to the beginning once a hundred percent eq. (1).

$$Yield \left(\frac{w}{w}\%\right) = \frac{mass \ essential \ oil}{initial \ mass} \ x \ 100 \quad (1)$$

Density Test

Density test is a test conducted to determine the mass of a substance per unit volume. Density tests can use a pycnometer, by weighing the mass of an empty pycnometer and a pycnometer that has been filled with essential oil. The value of density are made in equation eq. (2).

$$density\left(\frac{gr}{ml}\right) = \frac{mass \ total-mass \ of \ empty \ pycnometer}{volume \ of \ pycnometer} \quad (2)$$

Organoleptic Test

Organoleptic test is a test of a research product that uses the human senses in the form of the senses of sight, smell, hearing, touch, and taste. Organoleptic test parameters can use the method of Slovin which can be measured from several populations for sample testing eq. (3).

$$n = \frac{N}{1 + N(e^2)} \quad (3)$$

Where, N is the population, n is the sample number, and e is the tolerance error value (0.01).

If an organoleptic test cannot be known the number of populations or is unlimited and there are limitations of time and funds, then the calculation of the number of sample tests can be measured by the Lemeshow method to determine the minimum sample size in quantitative research eq. (4).

$$n = \frac{NZ^2 P.(1-P)}{L^2}$$
(4)

Where, n is the minimum sample size, Z is the standard value of the normal distribution according to the desired confidence level (for example, 90% confidence level, Z = 1.645), P is the prevalence of the outcome or the proportion of the population with certain characteristics (maximum 50%), and L is the accuracy or margin of error (for example 10%).

RESULTS AND DISCUSSION Distillate of Essential Oil from Rose Flower

The average extraction volume of essential oils from plants ranges from (0.98 - 1.99 ml/100g) (Milenković et al., 2021). In this study, the volumes of extraction of essential oils were produced with a very small range, as in Table 1 and Figure 2. Samples 1 and 2 did not contain any essential oil, this is because the microwave lengths into the particle have not been able to diffuse effectively, the wavelengths can increase if the time provided increases (Megawati et al., 2019). The sample 3 - 9 produces essential oils in small volumes. Volume increases steadily as the output power (watt) and time increase, this is due to wave length increases and conversion time increases so the extraction output will also increase. On this penalty can be said to have been in accordance with the research by (Milenković et al., 2021).



Yield Test

In this study, the extraction method used was Microwave Assisted Hydrodistillation (MAHD) followed by potentiometric yield test (%) to determine the yield of essential oil products. The difference in yield levels is caused by variations in transmitting power and time. Based on Table 1, it can be seen that there is an increase in yield levels as the wattage increases and the length of time combined. Samples 1 and 2 expressed the absence of essential oils, this is due to the length of the microwaves into the particles have not been able to diffuse effectively. While samples 3-9 can be seen in the presence of essential oils, this is because the higher the power used, it can produce more essential oils as well. Likewise, the time used. The variable that more influences the production of essential oil is power (Megawati et al., 2019).

Sample	Variable	Essential	Yield	Density	Organoleptic Test	
		Oil Volume	Test	Test	Scent	Color
		(ml)	(%)	(gr/ml)		
1	15 minute	0	0,0	-	-	-
	300 watt		00			
2	25 minute	0	0,0	-	-	-
	300 watt		00			
3	35 minute	0.1	0,0	0,840	Distinctive	Clear
	300 watt		01		Rose	white
4	15 minute	0.15	0,0	0,872	Distinctive	Clear
	400 watt		02		Rose	white
5	25 minute	0.128	0,0	0,891	Distinctive	Clear
	400 watt		01		Rose	white
6	35 minute	1	0,0	0,872	Distinctive	Clear
	400 watt		10		Rose	white

7	15 minute	1	0,0	0,947	Distinctive	Clear
	500 watt		10		Rose	white
8	25	1.1	0,0	1,040	Distinctive	Clear
	minute		11		Rose	white
	500 watt					
9	35	1.2	0,0	1,096	Distinctive	Clear
	minute		12		Rose	white
	500 watt					

Density Test

Density is related to microwave power, an increase in density indicates that the use of microwaves as a pre-treatment can increase the content of compounds obtained from the cell breakdown process (Argo & Amaliyah, 2021). We obtained density results from our research as in Table 1. Samples 1 and 2 did not obtain density values due to the absence of essential oil rose produced. For the sample 3-9, there was an increase due to the presence of other compounds resulting from cell decomposition using the microwave that followed. Cell breakdown by microwave doesn't just break the essential oil tissue (Argo & Amaliyah, 2021).

Organoleptic Test

Microwave power, time, and temperature in the process of extracting rose flower oil using Microwave Assisted Hydro Dystilation (MAHD) have a significant influence on the results of rose flower essential oil. Variables of power, time, and temperature can affect the amount of rose essential oil produced. In the practicum that has been done the most optimum essential oil is the variable power of 500 watts. and time 35 minutes. This can be seen from the % yield produced which is 0.012% and the resulting density of 1.096 gr / ml is clear white and smells typical of rose essential oil, but the density results are not in accordance with SNI 06-3735-1998 because the value is above 0.088-1.02 gr / ml, while for color and odor it is appropriate clear white and smells typical of rose essential oil.

Characteristics of Essential Oil with ANOVA

In the research we did, using the design design design of the factorial experimental design 3², then to find out the influence of each effect, data processing was done using the ANOVA (Analysis of Variance) method. In the study we did not do replication or repetition, so the analysis can only be done to determine the single effect of each variable.

Fable 2. Resu	lts of calcu	lation of	f primary	power	and
1	time effects	s on viel	d rates		

Source of Valiation	SS	Df	MS	F	P-value	F crit
Time	0,000193	2	9,65E-05	22,25797	0,006798	6,944272
Power	2,79E-05	2	1,4E-05	3,219195	0,146843	6,944272
Eror	1,73E-05	4	4,34E-6			
Total	0,000238	8				

The results of the analysis with ANOVA found that the F value, which is 22.25797 on the time variable has

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a value greater than F Crit, which is 6.944272. From the calculation results, it is proven that there is an influence between the power and time variables on the yield levels produced.

Density Test

The results of the analysis with ANOVA show that the F Value value is 6.595537 on the time variable and the power variable provides a value below the F Crit, which is 6.944272 so it can be seen that the amount of power and the length of time in the extraction process does not affect the density of the extraction results.



Figure 3. Relationship of 3% Rose Oil yields to time

From Figure 3 above we can conclude the influence of the force on the rendition, i.e. the greater the power, the effect of the vibration of the microwave produces a greater frequency of the wave, which occurs on the load of the components of the material. The speed of movement (vibration) between these molecules which then produces the heat effect, thus affecting the process of the exit of essential oil from the raw material, as a result the rate of evaporation of oil becomes faster (Effendy et al., 2020).

At the influence of time the efficiency of the device will work optimally from 30 minutes to 90 minutes and experience a decrease at 120 - 150 minutes. This is because when exceeding the optimal time the increase in yields is not very significant and tends to experience decreases due to the decreased content of essential oils in raw materials (Pane & Purba, 2020). The power used by the microwave has a strong influence on the amount of oil produced. This suggests that the greater the power or energy used in the extraction process, the weight of the product produced will keep increasing and increasing (Abed, 2020).

Effect of Microwave Power on Rose Flower Oil Yield Levels

Factors that greatly affect the quality of essential oil results are power, time and temperature. The greater the power, the greater the heat transfer rate will be which causes the rose flowers to evaporate faster so that more essential oil is produced (Effendy et al., 2020). Time affects efficiency, the tool will work optimally at 30 minutes to 90 minutes and experience a decrease at 120 - 150 minutes. This is because when exceeding the optimal time the increase in yield is not too significant and tends to decrease because the essential oil content in the raw material has decreased (Effendy et al., 2020). As for the temperature, it is directly proportional to the % yield because the higher the temperature, the faster the evaporation that occurs to remove the oil from the raw material (Yuniati et al., 2021).



Figure 4. Overall Extraction Result of Essential Oil from Rose Flower

CONCLUSION

Microwave power, time, and temperature in the process of extracting rose flower oil using Microwave Assisted Hydro Dystilation (MAHD) have a significant influence on the results of rose flower essential oil. Variables of power, time, and temperature can affect the amount of rose essential oil produced. In the practicum that has been done the most optimum essential oil is the variable power of 500 watts. and time 35 minutes. This can be seen from the % yield produced which is 0.012% and the resulting density of 1.096 gr / ml is clear white and smells typical of rose essential oil, but the density results are not in accordance with SNI 06-3735-1998 because the value is above 0.088-1.02 gr / ml, while for color and odor it is appropriate clear white and smells typical of rose essential oil.

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