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Extraction of Essential Oils from Sweet Orange Leaves (*Citrus aurantium*) Using the Microwave Hydrodistillation (MHD) Method

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Abstract. This study aims to utilize sweet lime leaves, which are only waste that has no value, where sweet lime leaves are one of the ingredients that contain essential oils. Apart from the skin, it turns out that this sweet lime leaf can also be extracted using the Microwave Hydrodistillation (MHD) method. This study uses three variables: the F/S ratios used were 0.375 g/mL, 0.5g/mL, and 0.65 g/mL, the powers used were 150 watts, 300 watts, and 450 watts, and the times used were 30, 60, and 90 minutes. The extraction method uses fresh ingredients with a solvent volume of 200 ml. The test carried out is the GC-MS test. The results of the test method and the purpose of this study were to determine the limonene content in the extraction of sweet lime leaves using the MHD method with variations in ratio, power, and time. The optimum yield of essential oils is at a power of 300 W, 90 minutes, and a ratio of 0.625, with a yield of 0.373%. The most significant components produced in the MHD method of extracting citrus leaves were Germacrene D 29.51%, Alpha-Copaene 18.47%, CIS-CAROPHYLNE 15.42%, 9-Eicosene (E) 8.58% and Limonene 5.51%.

Keywords: *essential oil, MHD, GC-MS test.*

1. Introduction

The most essential fruit commodity worldwide is oranges, with annual production exceeding 120 million tonnes. In Indonesia, the area of land planted with oranges has reached more than 57000 hectares with a production of around 2.5 million tonnes [1]. One of the citrus producers is in Semboro Village, Jember. Citrus fruit is one of the fruits that are in great demand by the public because of its refreshing aroma and is a source of vitamin C, besides that it also contains various other essential nutrients such as carbohydrates (dietary fiber and sugars), potassium, magnesium, copper, calcium, phosphorus, folate, thiamin, niacin, riboflavin, vitamin B6, pantothenic acid, and other phytochemical compounds [2].

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Extracting essential oils from citrus plants has long been popular because they can be extracted from fruit, peel, and leaves. The most popular type of orange that extracts its essential oil is sweet orange (*Citrus aurantium*). Lime leaves contain bioactive compounds: alkaloids, phenolics, saponins, tannins, steroids, and flavonoids. These phenolic and flavonoid compounds can act as antioxidants [3]. Similar compounds are also found in citrus leaves of Citrus Aurantifolia, which contain volatile compounds belonging to the monoterpenes, sesquiterpenes, alcohols, aldehydes, esters, and others. The monoterpene group is the dominant compound found in the leaves of C. aurantifolia, such as limonene (30.11%), β-pinene (19.27%), and β-oxy (3.488%). Sesquiterpenes contained as much as 3.365%, while the alcohol group included in the leaves is citronellol (3.989%) and α-terpineol (3.061%). The aldehyde group abundant in C. aurantifoia leaves is citronellal [4].

Orange leaves were used in this research because they can produce limonene, which is obtained from the essential oil extraction process. Orange leaves are traditionally used to treat skin diseases and as an anti-inflammatory agent [5]. Decoction of orange leaves is usually used for eye drops, reducing fever, as a mouthwash, sore throat, canker sores, cardiovascular disease, treatment, and prevention of cancer. The benefits of orange leaves can reduce anxiety and nervousness, reduce stress-related disorders such as insomnia or digestive disorders of nervous origin, have anti-inflammatory potential (digestive system), antispasmodic properties of the digestive system (distension, diarrhea), and treat cardiovascular disorders. It also fights fever, headaches, and colds [6].

Limonene is a compound belonging to the monoterpene group. Limonene freezes at -40°C, boils at 176.5°C, and is colorless and odorless [7]. In nature, there are two kinds of limonene: d-limonene, which smells like oranges, and l-limonene, which smells like turpentine. Limonene can be obtained by extracting lime leaves, where, in general, limonene is used as a flavoring and aroma enhancer in food and fragrances in perfumes [8]. Limonene is very useful if extracted from the leaves as an essential oil. In addition to reducing the number of waste sources, the essential oils produced have a high selling value [9].

Extraction of essential oils from lime leaves usually uses conventional methods such as hydro distillation, steam distillation, cold pressing, and solvent extraction. However, this traditional method has drawbacks in terms of product quality, including loss of some essential volatile compounds, low extraction efficiency, high energy consumption, and too long processing time. In addition, essential compounds in the oil can be degraded due to heating and hydrolysis, and the extracts can also be contaminated with toxic solvent residues [10]. To overcome this shortcoming, a "green technique" has been applied in essential oil extraction to make the process more effective. One of the more effective methods is Microwave Hydro distillation (MHD), which uses a relatively short process, produces high yields, and minimizes the use of solvents [11].

The Microwave Hydro Distillation (MHD) extraction method combines water distillation with microwave heating [12]. This method was chosen because the extraction process does not require further purification solvents, and the vacuum pressure and operating conditions do not need to reach a critical condition, making it easier. Water as a solvent was chosen because it has a high dielectric constant, making it more effective at absorbing microwaves [13]. Water is a cheap, environmentally friendly, non-flammable, non-toxic solvent, and allows for clean processing and pollution prevention [14]. The MHD method uses microwaves as an environmentally friendly energy source; the extraction process is fast, making it more efficient and economical, and produces less liquid waste [10]. In the MHD method, microwaves heat and evaporate water from cells so that cells swell, stretch, burst, and allow metabolic components to be extracted by solvents [15]. Various previous studies regarding the extraction of citrus fruit leaves and peels can be seen in Table 1 below.

Table 1. Previous studies on the Extraction of Leaves, Stems, and Citrus Fruits

No.	Raw Material	Methods Results	Reference
1.	Lime leaves	The process using the US-MAE method can produce a greater yield of kaffir lime leaf oil than the conventional method (0.33%) with a very short extraction time.	[16]
2.	Stem, Leaves and Peel of Kaffir lime (Citrus Hystrix Dc)	The optimum yield results in the extraction of kaffir lime leaf oil obtained by the SFME method are: - Fresh condition size (0.88 ± 0.25 cm) ratio of 0.15 g/ml is 7.23%. - Extraction of kaffir lime stem oil in fresh condition size (2.24 ± 0.15 cm) ratio of 0.3 g/ml is 0.29 %. - Optimum yield in fresh condition size (1.34 ± 0.23 cm) ratio of 0.2 g/ml is 4.19 %.	[7]
3.	Orange peel (Citrus Aurantium L.)	The MSDF method produces a yield faster than the MHG method at an optimal power of 264 W. At 20 minutes, the MSDF method produces an essential oil yield of 1.95%, while the MHG method produces a 1.93% yield at 40 minutes.	[17]

No.	Raw Material	Methods Results	Reference
4.	Orange peel (Citrus Aurantium L.)	Microwave power of 100 and 300 watts and extraction time of 60 minutes. The % yield obtained from the MHD method is 0.3% for 50 minutes of the extraction process, comparable to the % yield from the MHG method of 0.293% for 35 minutes of the process	[12]
5.	Sweet Orange Leaves	In the MAHD method, the optimal conditions obtained were a yield of 0.43% with a material and water ratio of 3.46:1 (mL/g), an extraction time of 100.47 minutes, operating power of 471.58 W. The largest compound was obtained from the test is Sabinene (30.556 %), Cis-Ocimene (10.139 %), and D-Limonene (9.682 %).	[18]

Based on the explanation above, a study was carried out to extract sweet lime leaves using the MHD method to obtain the yield and quality of essential oil, and then to test the limonene content using the GC-MS test.

2. Materials and Methods

2.1 Materials and Tools

The materials used in this study were fresh sweet lime leaves (*Citrus aurantium*) aged 2-3 years obtained from Semboro Village, Jember Regency, and the solvent used was H_2O aquadest. The tools used in this study were: Electrolux EMM-2007X type microwave, 20 L, 220 V, maximum power 800 W, wave frequency 2450 Hz, 1000 mL round bottom flask, modified Clevenger, condenser, analytical balance, scissors, bottle vials, Erlenmeyer merk Pyrex 100 ml, beaker glass merk Pyrex 500 ml, pumps, states, and clamps. PTFE-coated microwave cavity measures 46.1 x 28 x 37.3 cm. The modified Clevenger tool set can be seen in Figure 1.

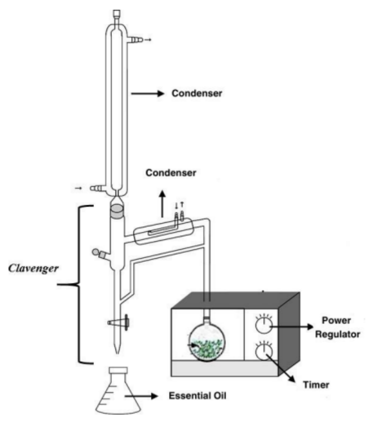

Figure 1. Clevenger Modification

2.2 Measurement of water content

Moisture content is the percentage of water content of a material, which can be expressed based on wet weight (damp premise) or dry weight (dry premise). Moisture content has a significant influence and role on the quality of a product. Water content was measured using the thermogravimetric method (oven method). The sample for which the water content will be calculated is weighed first and then dried in an oven at 100 °C for 2 hours, then allowed to stand at room temperature. Then, in the oven again at a temperature of 100 °C for 1 hour, it was weighed to obtain a constant level after being cooled to room temperature. Calculation of the water content is obtained by comparing the mass of the sample before drying and the mass lost after drying multiplied by 100% (equation 1) [7].

$$Water\ content = \frac{initial\ sample\ weight - final\ sample\ weight}{initial\ sample\ weight} x\ 100\% \quad (1)$$

2.3 Essential Oil Extraction Process

Sample preparation is the first step that is carried out before the study [19]. Fresh sweet lime leaves were obtained from Semboro Village, Jember Regency. Fresh sweet lime leaves are cut (± 3 cm). The F⁄S ratios used are 0.375, 0.5, and 0.625 g/mL with a solvent volume of 200 mL. The extraction process was operated with 150 Watt, 300 Watt, and 450 Watt power for 30, 60, and 90 minutes. The distillate is then separated using a separatory funnel on the Clevenger and put into vials.

2.4 Measurement of Essential Oil Yield

Yield is the ratio of the amount of oil produced from the extraction of aromatic plants (equation 2). The higher the yield value, the more essential oil is obtained [20]. If water is still found when calculating the yield, anhydrous sodium sulfate (Na_2SO_4) is added. Anhydrous sodium sulfate dissolves easily in water and is hygroscopic (easily absorbs water). This addition aims to purify the desired result by binding the remaining water mixed with the essential oil. Anhydrous sodium binds water and will still precipitate and leave a layer of essential oil [21].

$$Yield = \frac{mass\ of\ esential\ oil}{mass\ of\ raw\ material} \times\ 100\% \quad (2)$$

2.5 Essential Oil Composition Analysis

The GC-MS (Gas Chromatography and Mass Spectroscopy) test was used to determine the chemical content of sweet orange essential oil. The compounds present in the mixture are separated in the chromatography column. The advantages of this method are fast identification time, high sensitivity, good separation, and the fact that the tool can be used in the long term [22]. The limonene component is in the sweet orange essential oil composition, which is analyzed more deeply.

2.6 Data Analysis

Data optimization analysis was carried out with the help of Design Expert 13 using the RSM (Response Surface Methodology) method. The RSM type used is Box-Behnken Design with three process parameters: Microwave power, F⁄S ratio, and extraction time—the results of the research formulation design with design experts.

3. Result and Discussion

3.1 Effect of Process Parameters on The Yield of Citrus Leaf Essential Oil

3.1.1 Yield Result Analysis

The Microwave Hydro Distillation (MHD) method extracted essential oils from lime leaves. The variables observed to determine the Yield obtained were the extraction time of 30-90 minutes, 200mL solvent, 150, 300, and 450W power, and F/S ratios of 0.375, 0.5, and 0.625. The extraction process is based on operating condition data using Design Expert version 13 in Table 2. The yield obtained on the extraction results for each variable is shown in Table 2 below.

Table 2. Extraction Yield Results

Run	**Time**	**Power**	**Ratio**	***Yield (%)***
1	30	300	0.625	0.185
2	30	150	0.5	0.125
3	30	450	0.5	0.162
4	60	300	0.5	0.244
5	90	300	0.375	0.265
6	90	300	0.625	0.373
7	60	150	0.625	0.212
8	60	300	0.5	0.238
9	60	300	0.5	0.25

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Run	Time	Power	Ratio	*Yield (%)*
10	60	300	0.5	0.232
11	60	450	0.375	0.19
12	90	450	0.5	0.27
13	30	300	0.375	0.141
14	90	150	0.5	0.215
15	60	450	0.625	0.285
16	60	150	0.375	0.178
17	60	300	0.5	0.256

Table 2 shows that the highest yield in this study was 0.373%, namely in sample 6 with a time variable of 90 minutes, a power of 300 W, and an F/S ratio of 0.625. The lowest yield is 12.5% in sample 2 with 30 minutes, 150 W power, and 0.5 F/S ratio. The study results were then tested by ANOVA using Design Expert 13 to determine the effect of the extraction variables. The yield results obtained in this study were lower than those of previous research conducted by Dao et al in 2019, namely 0.43%. This is influenced by the mass of the material used at 3.46 g/mL with an extraction time of 100.47 minutes using 471.58 watts of power.

3.1.2 Effect of Microwave Power and Extraction Time on Yield

Microwave power functions as a driving force to break down the structure of the plant cell membrane, so that the oil can diffuse out and dissolve in the solvent. Thus, an increase in power will generally increase the yield and speed up the extraction time. In the extraction process, microwave power controls the amount of energy the material receives and converts it into heat energy. This heat energy helps release essential oils from the ingredients [23]. The greater the power used, the higher the temperature of the system during extraction, and the faster the time needed to reach the boiling point. The power of microwaves significantly affects the speed of the extraction process, so the yield of lime leaf essential oil will be even greater [24]. This is because the greater the power, the higher the operating temperature, and the greater the distillation (evaporation) rate becomes. The temperature increase results from the ability of materials and solvents to absorb energy from microwaves. The greater the power, the greater the energy the material receives to be converted into heat, so the yield of lime leaf oil is greater [25]. However, the amount of power should not be too large, because it can remove critical volatile compounds in the material [10]. This happens because the microwave treatment will break down the cell walls in the sample. The cell wall rupture occurs due to collisions and friction between particles, which generates heat. The higher the power used and the longer the extraction is carried out, the contact between the material and the solvent occurs, and the sample temperature increases, causing more solvent in the system to evaporate, and mass loss occurs [26].

This study shows that the optimal microwave power to achieve the highest yield is 300W with an extraction time of 90 minutes. Using high power for a long time can damage the compounds in the essential oil of lime leaves. Thus, the higher the power used, the greater the essential oil yield. However, please note that using 450 W of power with an extraction time of 90 minutes results in a lower yield. The decrease in yield occurs because the higher power and the longer time used will damage the material, so the material cannot be extracted. The highest yield, 0.373%, was obtained using 300 W of power for 90 minutes of extraction time.

Meanwhile, the lowest yield of 1.25% was obtained when using 150 W of power with an extraction time of 30 minutes because the combination of time and power was too low, causing the extraction to be less effective. This research also shows that yield gain can be increased with different power variations and periods, especially in the 30 to 90-minute range. The effect of microwave power and extraction time on yield is shown in Figure 2.

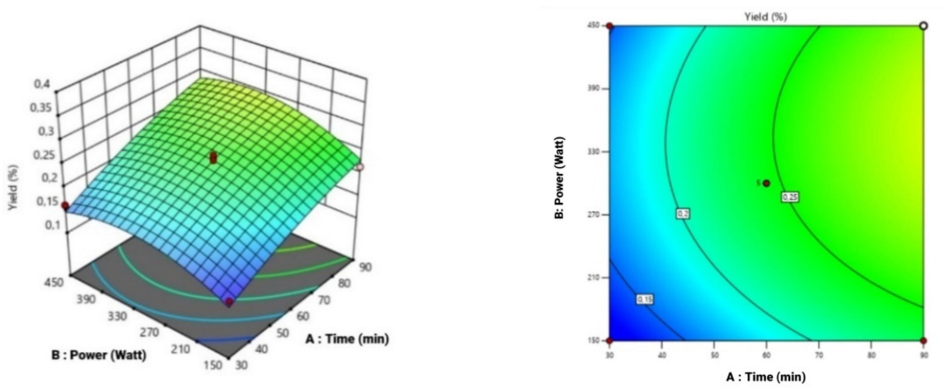

Figure 2. Effect of Microwave Power and Extraction Time on Yield

3.1.3 The Effect of Material F/S Ratio and Time on Yield

Based on this research, the extraction of lime leaf essential oil was carried out using a mass ratio variable of 0.375 g/mL, 0.05 g/mL, and 0.625 g/mL, which is equivalent to the mass of the material 75 grams, 100 grams, and 125 grams, and placed in a 1000 mL distillation flask. From the graph obtained, it can be concluded that the optimal ratio to achieve the best yield is 0.625 g/mL with an extraction time of 90 minutes. An increase in the amount of raw material used indicates an increase in yield, while using fewer materials results in a lower yield. This is because the more ingredients used, the more oil can be extracted. In this study, low yields were obtained using a ratio of 0.5 g/mL with an extraction time of 30 minutes. This is caused by the short extraction time, so the material extraction is less than optimal. The effect of the F/S ratio and extraction time on yield is shown in Figure 3.

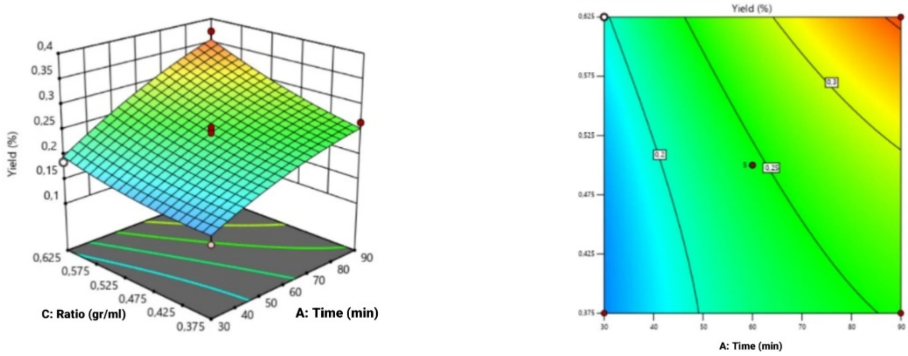

Figure 3. The Effect of Material F/S Ratio and Time on Yield

3.1.4 Effect of Material F/S Ratio and Power on Yield

Power parameters and F/S ratio (mass of extraction material) influence the yield of essential oil from lime leaves produced. Increasing the power in the extraction process will increase the yield, because the greater the power used, the more important oil can be extracted from the lime leaves. However, remember that using too much power can cause degradation of the compounds in the lime leaves, which can cause the extraction not to work optimally. Therefore, in this study, the power used was adjusted according to the literature, and it was found that the optimum power was 300 W.

This research also shows that the greater the ratio of ingredients in the extraction bottle, the higher the yield. That is, when more materials are used for extraction, the amount of essential oil extracted from kaffir lime leaves will increase. However, using a huge ingredient ratio can also slow the evaporation rate in the extraction process because too much material can cover the space in the distillation flask. It can also affect the efficiency and effectiveness of the extraction process. Using the power of 300W and the appropriate F/S ratio can optimize the yield from lime leaf essential oil extraction without destroying its crucial compounds. The effect of the f/s ratio and power is shown in Figure 4.

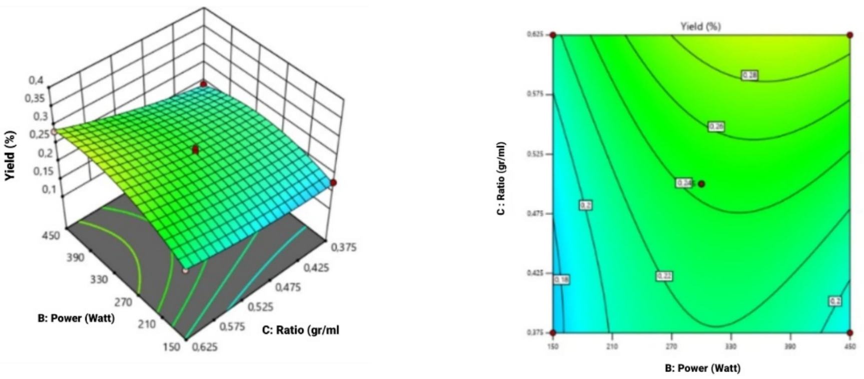

Figure 4. Effect of Material F/S Ratio and Power on Yield

3.2 Analysis of Orange Leaf Essential Oil

The components contained in lime leaf essential oil can be identified by Gas Chromatography-Mass Spectrometry (GC-MS) analysis. The results of the study of essential oils tested using the extraction time of 90 minutes, power of 300W, and a ratio of 0.625 show that essential oils contain 30 chemical components. The GC-MS test was conducted using an oven temperature of 60 °C and an injection temperature of 260 °C with split injection mode, using the 2010 GC-MS test kit. The results of the analysis can be seen in Figure 5.

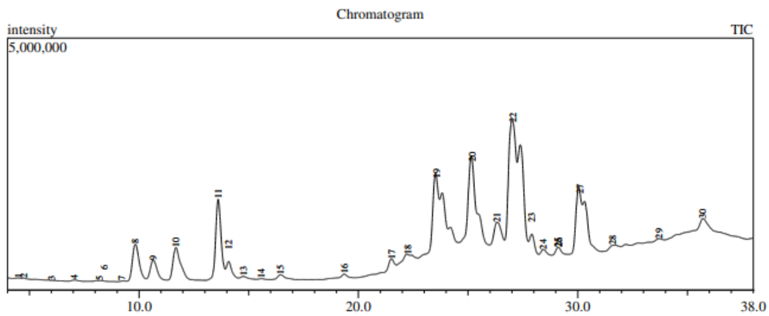

Figure 5. Chromatogram of GC-MS test results

The results from a GC-MS test are recorded as a series of peaks, where each peak represents one compound in the mixture that passes through the detector. If there are many compounds in the sample, the number of peaks in the GC spectrum reflects the amount of those compounds. By referring to the retention time known from the literature, it is possible to identify the compounds in the sample. Various factors influence GC-MS results, including the sample type and the GC conditions used. Therefore, no standard rule states that limonene will always appear as a peak in GC-MS results. In general, GC-MS results reflect the composition of the compounds in the sample. If limonene is the main compound in the sample, it will usually appear as a significant peak in the GC-MS chromatogram. However, suppose the concentration of limonene is low in the sample or there are technical problems in the analysis, such as inappropriate GC settings or interference in the mass spectrometry. Limonene may not be detected or appear at the expected peak positions [27].

Based on the chromatogram data, a limonene compound is obtained with the molecular formula $C_{10}H_{16}$ with a molecular weight of 136. The limonene compound is number 11, which indicates the compound is less dominant with an area percentage of 5.51% in a retention time of 13.611 minutes. A comparison of target data from the spectrum shows the limonene mass in Figure 6, which shows the possibility of a compound with close to 95% similarity according to the WILEY7.LIB data library.

The amount of limonene obtained in this study was less than that obtained in the previous research conducted by Dao et al. in 2019. Dao's research yielded 9.682% limonene. This might have happened because the compounds in the two materials were different; the sweet lime leaves used in this study came from Semboro Village, Jember, and in Dao's study came from Vietnam. Research conducted by Tita Syarifah in 2017 produced 0.17% limonene in fresh ingredients with a retention time of 8.48 minutes using kaffir lime leaves, using the solvent-free microwave extraction method. Meanwhile, sweet orange peel produces more limonene than the leaves. This was proven in a previous study conducted by Yerizem et al in 2022, which stated that the limonene produced was present at a retention time of 6.09 minutes with an area of 98.70% using the Soxhlet extraction method [28]. The results of the GC-MS analysis can be seen in Table 3, and the mass spectrum of limonene compounds can be seen in Figure 6.

Table 3. GC-MS analysis results

Peak	Compound	*Retention Time*	*%Area*
1	*Pentanal*	4.513	0.03
2	*Carbamic acid, methyl ester*	4.750	0.02
3	*Piperazine*	6.021	0.02
4	*Hexanal (CAS) n-Hexanal*	7.053	0.05
5	*1-PENTEN, 4,4-DIMETHYL-1,3-DIPHENYL-1-(TRIMETHYLILLXY)*	8.207	0.05
6	*3,4-dibrom-1,1,1-trifluor-2-(trifluormethyl)-3-buten-2-ol*	8.420	0.01
7	*2-Hexenal, (E)- (CAS) (E)-2-Hexenal*	9.210	0.02
8	*.ALPHA.-PINENE, (-)-*	9.833	3.00
9	*Camphene*	10.655	1.77
10	*2-.BETA.-PINENE*	11.682	3.08
11	*l-Limonene*	13.611	5.51
12	*1,3,6-Octatriene, 3,7-dimethyl-, (E)- (CAS). BETA.OCIMENE Y*	14.091	1.31

Peak	Compound	*Retention Time*	*%Area*
13	*1-Hexanol, 2-ethyl- (CAS) 2-Ethylhexanol*	14.768	0.39
14	*delta-2-carene*	15.573	0.10
15	*Nonanal (CAS) n-Nonanal*	16.451	0.50
16	*Decanal (CAS) n-Decanal*	19.373	0.39
17	*ACETIC ACID 1,7,7-TRIMETHYL-BICYC[2.2.1]HEPT-2-YL ESTER*	21.527	1.79
18	*Bicycloelemene*	22.263	3.09
19	*alpha.-Copaene*	23.569	18.47
20	*CIS-CARYOPHYLLENE*	25.197	15.42
21	*.alpha.-Humulene*	26.329	1.75
22	*Germacrene D*	27.061	29.51
23	*.delta.-Cadinene*	27.906	0.98
24	*sesquisabinene hydrate*	28.427	0.35
25	*Nerolidol*	29.103	0.53
26	*Nerolidol*	29.155	0.97
27	*9-Eicosene, (E)-*	30.105	8.58
28	*01297107001 TETRANEURIN - A - DIOL*	31.595	0.75
29	*9-Octadecenoic acid (Z)- (CAS) Oleic acid*	33.718	0.37
30	*Tetradecanoic acid (CAS) Myristic acid*	35.706	1.19

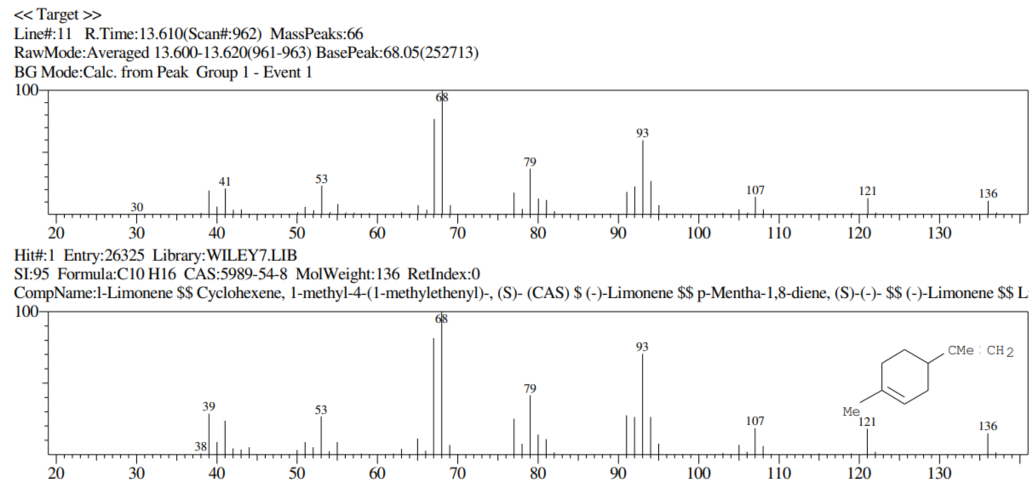

Figure 6. Mass spectrum of limonene compounds

Based on GC-MS analysis, the components contained in lime leaf essential oil consist of five groups of compounds, namely monoterpenes (14.77%), sesquiterpenes (69.22%), oxygenated monoterpenes (0.39%), oxygenated sesquiterpenes (2.6%), other oxygenated compounds (4.37%), and other compounds (8.64%). Sesquiterpene compounds influence the aroma of essential oils more than other components. The sesquiterpenes compound is one of the compounds capable of binding aroma [29]; therefore, it has a strong and fragrant aroma in research conducted on sweet lime leaves. Among the 30 components contained in essential oils, five components have the highest area, namely Germacrene D 29.51%, alpha-Copaene 18.42%, cis-Carophyllene 15.42%, 9-Eicosene (E) 8.58 % and Limonene 5.51%. Germacrene D is a monocyclic sesquiterpene product of high value due to its structural variability and insecticidal activity. Germacrene D has repellent activity against ticks, mosquitoes, and aphids [30].

3.3 Analysis of Variance (ANOVA)

Analysis of Variance (ANOVA) is a statistical test used to estimate which variable from the data is more dominant based on the relationship between other variables [31]. The purpose of analysis using ANOVA is to test statistical hypotheses and to determine data optimization. A parameter can be significant if the analysis results produce a probability value ≤ 0.05 or 5% for the p-value and a p-value ≥ 0.05 [32]. High f-values and small p-values indicate significant values significantly affecting the tests' results [33]. Other parameters are the R2 value greater than 0.7 and the precision value greater than 4 [34]. The research model produces a p-value of 0.0003, so the research analysis model significantly affects results extraction. The results of the ANOVA analysis are listed in Table 4 below.

Table 4. ANOVA results

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	0.0556	9	0.0062	21.01	0.0003	Significant
A-Time	0.0325	1	0.0325	110.65	< 0.0001	
B-Power	0.0039	1	0.0039	13.33	0.0082	
C-Ratio	0.0099	1	0.0099	33.59	0.0007	
AB	0.0001	1	0.0001	0.2757	0.6158	
AC	0.0010	1	0.0010	3.49	0.1042	
BC	0.0009	1	0.0009	3.17	0.1184	
A^2	0.0007	1	0.0007	2.47	0.1601	
B^2	0.0060	1	0.0060	20.56	0.0027	
C^2	0.0004	1	0.0004	1.47	0.2648	
Residual	0.0021	7	0.0003			
Lack of Fit	0.0017	3	0.0006	6.28	0.0540	not significant
Pure Error	0.0004	4	0.0001			

The analysis results show that the model formed is significant, where the f-value is 21.01 and the p-value is 0.0003 < 0.05. The lack of fit formed is 0.0540 > 0.05. An insignificant lack of fit states that the test model used is appropriate so that it can explain the problems being studied. Time, power, and ratio variables have values less than the significance level of 0.05, meaning they substantially affect the yield.

R-Squared (R^2) is the coefficient of determination with a value between 0 and 1. If R^2 is close to 1, the relationship between one variable and the other is stronger. Conversely, if R^2 gets smaller, the relationship between one variable and another weakens [35]. The coefficient of determination (R^2) describes the percentage of influence of variables. The results of the fit statistics that have been carried out are shown in Table 5.

Table 5. Fit Statistic

Std. Dev.	0.0171	**R^2**	0.9643
Mean	0.2248	**Adjusted R^2**	0.9184
C.V. %	7.63	**Predicted R^2**	0.5191
		Adeq Precision	18.5788

The R^2 value from the analysis results obtained is 0.9643, so it can be declared appropriate because it is more than 0.75 [36]. The adjusted R^2obtained is 0.9184, while the predicted R^2 is 0.5191. The difference between the adjusted R^2 value and the predicted R^2 value is not good because it exceeds 0.2, namely 0.3993, which indicates a problem with the model or data used. The problem with model deviation is that some variables are not significant, so the ability of these variables to explain responses is minimal [37]. The total yield of essential oils as a response to the extraction parameters in the ANOVA model can be modeled using the following quadratic equation:

Yield = 0.157750+0.001442A+0.000691B-0.867000C+1.00000E06AB
+0.004267AC+0.000813BC-0.000015A^2-1.68333E -06B^2+0.648000C^2

where the value of A is the time to solvent, B is the microwave power, and C is the ratio of ingredients. The regression equation can determine the response value of the total yield concentration obtained when the ingredients, extraction time, and microwave power ratio differ.

The results of the ANOVA form a fit relationship between the data obtained from the model formed and the experimental data alluded to by linear regression, as shown in the parity plot in Figure 7. The line in the parity plot is straight because the results of the model formed with experimental data have an R^2 value close to 100%. The trend of this line indicates that the resulting model is a good predictor of the extraction rate in the experiment. The distance between the data position and the trendline shows the accuracy of the data, and the closer the data is to the trendline, the more accurate the data [38].

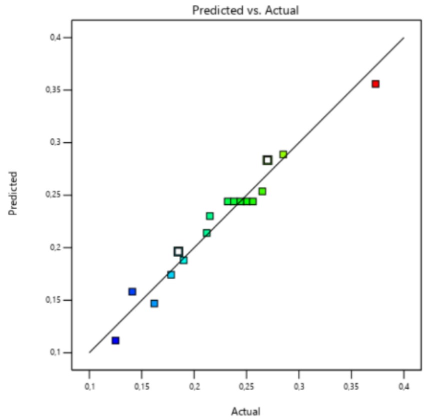

Figure 7. Parity Plot (Predicted vs Actual)

3.4 Comparison of MHD and Conventional Methods

The choice of the MHD method over conventional methods is because this method can shorten the extraction time. This is because the MHD method uses a microwave to heat ingredients and water more efficiently than traditional methods, which rely only on conventional heaters. In addition, the more efficient heating in the MHD method helps extract essential oils from orange leaves better. The result is an increased yield, meaning more essential oil can be extracted from the same material. Because the extraction time is shorter and the temperature can be controlled well in the MHD method, it can prevent the degradation of heat-sensitive compounds in essential oils so that the resulting oil can be of better quality. The MHD method is more energy efficient than conventional extraction methods. This is because microwaves only generate heat within the material, while in traditional methods, it is necessary to heat the entire system, including water or extraction solvent [16].

The yield produced by extraction using MHD was 0.373% at 300 W power, 90 minutes, and a ratio of 0.625. Meanwhile, the yield produced by the conventional ethanol solvent method was 0.4384% in 5.438 hours because the contact time between the material and the solvent was greater. When the extraction time was increased, the yield level would decrease because the solution might have reached the saturation point [39]. According to Moestofa (1981), extraction is faster at high temperatures, but this will cause some components contained in spices to be damaged. In addition, high temperatures will cause some of the ethanol to evaporate, so the amount of solvent is reduced and insufficient to extract the material. The yield is reduced [40].

3.5 Comparison of Essential Oils of Leaves and Orange Peel

Research conducted by Ayu Chandra K. F, Fikka Kartika W., to extract orange peels using the MHD method, uses a time variable of 50 minutes with a power of 300 W to produce a yield of 0.3%. This result is almost the same as the extraction of lime leaves, which produces a yield of 0.373% obtained at 300 W power with 90 minutes of extraction time. The relationship between microwave power and temperature is that high power increases the operating temperature above the solvent's boiling point, increasing the extraction yield. The cause of this increase is the heat that arises from the material and solvent, which is influenced by the value of the dielectric constant and dielectric loss factor of the material and solvent itself. The dielectric constant indicates the ability of the molecules in a material to be polarized by an external electric field.

Meanwhile, the dielectric loss factor measures the efficiency of microwave energy that can be absorbed to produce heat. Aquadest is used as a solvent because it is a polar compound that is easily soluble in water. The choice of this polar solvent occurs because the method of separating essential oils utilizes the principle of distillation, thus enabling its use in separating essential oils. Oil extracted with the aquadest solvent is browner in color due to chemical components, namely neral, geranial, β-myrcene, and citronellal extracted by the solvent because these compounds have a characteristic yellow to brownish color [41].

4. Conclusions

Extraction of essential oil from sweet orange leaves using the MHD method yielded 0.373%. Extraction time, microwave power, and ingredient ratio significantly affect the yield obtained. To obtain the maximum % yield, the maximum conditions used are 300 W power, 90 minutes, and a ratio of 0.625. The limonene obtained was 5.51% with an SI value of 95. Based on the results of the data normality test, the significant value of the independent variable data for sweet orange leaf essential oil concentration was obtained with an f-value of 21.01 and a p-value of 0.0003 < 0, 05 which means it has a significant effect on the yield results. The test results with ANOVA form a fit relationship between the data obtained from the model formed and the experimental data.

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