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EXTRACTION OF ESSENTIAL OILS FROM FRESH FLOWERS USING MICROWAVE-ASSISTED AND ULTRASOUND-ASSISTED EXTRACTION METHODS

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Essential oil
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INTISARI

Minyak esensial adalah senyawa aromatik volatil alami yang diekstraksi dari berbagai bagian tumbuhan melalui metode seperti distilasi uap, pengepresan dingin, atau ekstraksi pelarut. Penelitian ini mengevaluasi pengaruh jenis bunga terhadap rendemen minyak esensial menggunakan Ekstraksi Berbantuan Gelombang Mikro (MAE) dan Ekstraksi Berbantuan Ultrasonik (UAE) dengan etanol. Bunga yang diuji adalah kenanga, melati, dan kamboja. MAE menghasilkan rendemen yang lebih tinggi untuk semua bunga, dengan bunga kenanga menghasilkan rendemen terbanyak, yaitu 27,6% (MAE) dan 18,0% (UAE). Analisis FTIR ekstrak bunga terbaik yaitu kenanga mengungkapkan gugus fungsional (–OH, C=O, C–H, C=C) yang mengindikasikan senyawa aktif seperti linalool, eugenol, dan geranil asetat. Secara fisik, kenanga menunjukkan warna cokelat gelap dan aroma kuat, yang menunjukkan kandungan minyak esensial yang tinggi serta potensi untuk parfum dan aromaterapi. Efisiensi UAE terbatas karena sistem terbuka dan suhu rendah; oleh karena itu, sistem tertutup dengan kontrol suhu yang lebih baik direkomendasikan. Secara keseluruhan, kenanga adalah bahan yang paling menjanjikan untuk ekstraksi minyak esensial melalui MAE.

Keyword:

Essential oil
Extraction yield
MAE
UAE
Yield

ABSTRACT

Essential oils are compounds produced by naturally occurring volatiles extracted from various parts of plants through methods such as steam distillation, cold pressing, or solvent extraction. This study investigated the effect of flower type on essential oil yield using Microwave-Assisted Extraction (MAE) and Ultrasonic-Assisted Extraction (UAE) with ethanol. The flowers tested were ylang-ylang, jasmine, and frangipani. MAE yielded higher yields for all flowers, with ylang-ylang yielding the highest yields, at 27.6% (MAE) and 18.0% (UAE). FTIR analysis of the best flower extract, ylang-ylang, revealed functional groups (–OH, C=O, C–H, C=C) indicating active compounds such as linalool, eugenol, and geranyl acetate. Physically, ylang-ylang exhibited a dark brown color and a strong aroma, indicating its high essential oil content and potential for perfumery and aromatherapy. UAE efficiency was limited due to the open system and low temperature; therefore, a closed system with better temperature control is recommended. Overall, ylang-ylang is the most promising material for essential oil extraction via MAE.

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1. INTRODUCTION

Essential oils have become a promising export commodity for Indonesia. The country has successfully exported essential oils to 102 countries, including the United States, India, and several European nations. This indicates that Indonesia holds a competitive advantage in the global essential oil market [1]. Indonesia, as a country rich in biodiversity, has great potential in essential oil production by utilizing various aromatic plants such as lemongrass, cinnamon, vetiver, basil, citrus, clove, jasmine, and rose [2]. Essential oils are commonly derived from roots, bark, leaves, flowers, and seeds, each possessing distinct aromas and applications in pharmaceuticals, perfumery, food, and cosmetics. Essential oils offer benefits such as modulating leukocyte activity and providing antifungal, anti-hyperalgesic, and anti-inflammatory properties [3]. The diverse sources of aromatic compound-producing plants in Indonesia make it very important to explore local plants that have not been fully utilized. One aromatic plant with great potential but still insufficiently explored is ylang-ylang.

Ylang-ylang (Cananga odorata), YY is a tropical plant that grows best in lowland areas with temperatures ranging from 25°C to 30°C. This plant is known to have significant antioxidant and anticancer activities [4]. There are two main varieties of ylang-ylang: Philippina Cananga (forma macrophylla) and Java Cananga (forma genuina), which differ in morphological characteristics and aroma. These differences give each variety specific uses, particularly in the fragrance and therapy industries. The unique aroma and therapeutic benefits of YY flowers come from their complex chemical composition. The main compounds in YY essential oil include linalool, which has a calming effect; germacrene D, which provides a strong and long-lasting scent; benzyl benzoate and methyl benzoate, which impart sweet and warm notes; and geranyl acetate, which adds a fresh and soft aroma. Additionally, compounds such as eugenol, farnesol, and benzyl salicylate also contribute to its therapeutic properties, making YY a highly valuable ingredient for various pharmaceutical and cosmetic applications [4]. The variation in the essential oil composition of YY flowers is greatly influenced by various factors, such as the plant variety, growing environmental conditions, and the extraction methods used.

Jasmine flowers (Jasminum sambac), JA, a member of the Oleaceae family, are shrubs that can grow either climbing or upright. This plant is widely known for its white flowers that emit a fragrant and refreshing aroma. Jasmine flowers contain various important volatile compounds, such as linalool, benzyl acetate, indole, farnesol, jasmone, and methyl anthranilate, which are the main components of its essential oil. These compounds make jasmine flowers highly potent as a natural source of essential oil [5]. The essential oil extracted from these flowers offers various biological benefits, including sedative and antidepressant effects, as well as antimicrobial and antioxidant activities. In the cosmetic industry, jasmine essential oil is widely used as a key ingredient in perfumes due to its distinctive and luxurious fragrance. Meanwhile, in aromatherapy, this oil is often used to relieve stress, improve mood, and provide deep relaxation [6].

Frangipani (FP), commonly found in various regions of Indonesia, is a plant that is relatively easy to encounter. Although often used in religious ceremonies, most of these flowers have not been utilized optimally, and many are simply left unused. This plant frequently grows around cemetery areas. The fragrance of FP flowers comes from natural aromatic compounds such as geraniol, farnesol, citronellol, phenethyl alcohol, and linalool. The oil extracted from these flowers, known as FP essential oil, has great potential for use in various products such as bath soaps, lotions, perfumes, and mosquito repellents [7].

Various extraction methods have been developed to obtain essential oils, using both conventional and non-conventional techniques. Conventional methods such as Soxhlet extraction and maceration are still widely used because they can produce extracts of good quality. However, these approaches have limitations, including high solvent consumption and relatively long processing times [8]. Therefore, non-conventional extraction methods such as Microwave-Assisted Extraction (MAE) and Ultrasound-Assisted Extraction (UAE) have been developed. Microwave-Assisted Extraction (MAE) is a non-conventional method used to extract bioactive compounds from various plants because it offers advantages such as higher drying rates, greater energy efficiency, reduced solvent usage, and faster extraction times [9].

Meanwhile, the UAE method is an extraction technique that utilizes the principle of acoustic cavitation, which is the spontaneous formation of microbubbles in a liquid phase below its boiling point. This process generates pressure and shear forces capable of disrupting the cell walls of plant materials, allowing the solvent to penetrate more easily and dissolve the active compounds inside. Compared to maceration, the UAE method offers several advantages, such as increased solvent penetration efficiency into the cell matrix and accelerated mass transfer rates. [10].

2. MATERIALS AND METHODS

Materials

The main materials used in this study are three different types of flowers: dried ylang-ylang flowers obtained from Bekasi, West Java, Indonesia; jasmine flowers; and frangipani flowers sourced from the traditional market in Bandar Lampung. The solvent used for the extraction process is 96% industrial-grade ethanol. In the extraction process, 96% ethanol is selected as the solvent due to its effectiveness in extracting a wide range of bioactive compounds. Ethanol is considered a safer and more neutral alternative compared to other organic solvents, making it more suitable for applications in pharmaceuticals and food products [11]. All other chemicals used are of technical or analytical grade as required for the analyses, and did not undergo any further purification.

Flower Extraction

The flowers to be used are first thoroughly cleaned of dirt and other foreign matter, then dried naturally under sunlight for three days. This drying process aims to reduce the natural moisture content in the flower tissue, which can affect efficiency during the essential oil extraction process. After drying, the material is extracted using two different methods, namely MAE and UAE, using 96% ethanol as the solvent. The solvent-to-material ratio is adjusted based on the method used, with 5 grams of ylang-ylang flower powder at a 1:19 (w/v) ratio for MAE, and 10 grams at a 1:25 (w/v) ratio for UAE [9]. The solvent and material mixture was then placed in a 1000 mL three-neck flask to maintain temperature stability and ensure even solvent distribution throughout the process. The apparatus used in the MAE and UAE methods is detailed in **Fig. 1** and **Fig. 2**. [11]

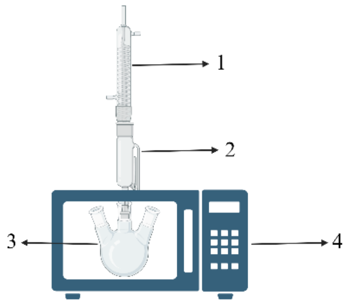


Fig 1. Microwave-Assisted Extraction (MAE) equipment series

Number 1 is a condenser that functions to condense the volatile compound vapor produced by heating so that it returns to the liquid phase. Number 2 shows a beaker used as a container for storing the extract resulting from the extraction process. The device at number 3 is a three-neck flask that functions as a place for mixing solvents and materials, while also allowing temperature and pressure control during the process. Meanwhile, device number 4 is a microwave unit used as a heat source to facilitate the release of active compounds from the flower matrix into the solvent.

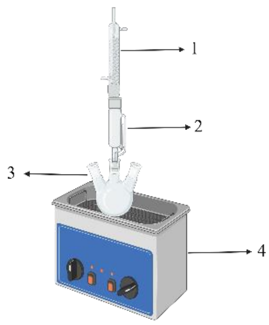


Fig 2. Ultrasonic-Assisted Extraction (UAE) equipment series

Number 1 is a condenser that functions to condense volatile compound vapors into a liquid phase. Number 2 is a beaker used to hold the extract during the process. Number 3 shows a three-neck flask used as a container for mixing ingredients and solvents while maintaining the stability of the process temperature. Number 4 is an ultrasonic unit that functions to transmit wave energy to accelerate the release of active compounds from the flower matrix into the solvent.

For the MAE method, extraction was conducted using microwave power of 375 W for 45 minutes, while for the UAE method, sonication was performed for 90 minutes to ensure maximum release of bioactive compounds [12]. After extraction, the mixture was filtered using Whatman No. 42 filter paper to separate the liquid fraction from the solid residue. The next step was to remove the bulk solvent from the oil fraction using a rotary evaporator, operated at 50°C, with a rotation speed of 80 rpm, and a vacuum pressure of 175 mbar for one hour [10].

Raw Material Preparation

Fresh ylang-ylang flowers were used as raw materials and were first dried naturally under sunlight for three days until evenly dried. The dried material was then ground using a grinder into a fine powder.

Yield Analysis (%)

The yield value is obtained by comparing the volume of essential oil produced to the mass of the dry raw material, and the result is expressed as a percentage. The calculation formula is shown in **Equation 1**:

$$\%Yield = \frac{Vm}{m_b} \times 100\ \% \qquad \text{Eq. (1)}$$

Explanation:

Vm = volume of essential oil produced (mL)
Mb = mass of the dry raw material (g)

Fourier Transform Infrared (FTIR) Analysis

The FTIR analysis results were obtained from the characterization of the ylang-ylang flower extract using a Fourier Transform Infrared Spectroscopy (FTIR) Agilent Technologies Cary 630 instrument within the wavelength range of 4000–650 cm^{-1} [13]. This process aims to identify specific functional groups that are part of the active components in the tested extract. The extracted samples were analyzed without destructive treatment, where the resulting absorption spectrum displayed characteristic bands representing the stretching or bending vibrations of specific chemical bonds. The information obtained from the FTIR spectrum was used to identify dominant compounds in the extract, which are related to its biological activity and potential further applications.

Characteristic Analysis

The characterization of essential oil includes observations of color, aroma, and Free Fatty Acid (FFA) content. Color testing is performed visually to assess the purity and quality of the oil, while aroma analysis ensures that the scent profile of the oil meets the expected standard.

The determination of FFA content is carried out by weighing 2.5 grams of essential oil and placing it into a 250 mL Erlenmeyer flask. Then, 50 mL of 96% ethanol (previously neutralized using 0.1 N NaOH solution) is added, followed by 2 mL of phenolphthalein (PP) indicator. The solution is then titrated slowly with 0.1 N NaOH from a burette until a persistent pink color appears and remains stable for 30 seconds. The free fatty acid content in the transparent soap can be calculated using the following formula in **Equation 2** [14].

$$FFA(\%) = \frac{V \times N \times MW}{M \times 1000} \times 100\% \qquad \text{Eq. (2)}$$

Explanation:

V = Volume of NaOH titrant (mL)
N = Normality of NaOH (0.1 N)
MW = Molecular weight of the essential oil (g/mole)
M = Mass of essential oil (g)

3. RESULTS AND DISCUSSION

Yield Analysis (%)

This study aimed to evaluate the effect of flower type on the extraction yield (%) using two non-conventional extraction methods: Microwave-Assisted Extraction (MAE) and Ultrasound-Assisted Extraction (UAE), with ethanol as the solvent. The types of flowers used in this study were ylang-ylang (YY), jasmine (JA), and frangipani (FP). The results showed significant differences between each flower type as well as the effectiveness of each extraction method in terms of the amount of extract produced. The yield results are shown in **Table 1**.

Table 1. Essential Oil Yield (%) from Different Flower Types Using MAE and UAE Methods

Flower Type	Yield (%)	
	MAE	UAE
Ylang-Ylang	27.6	18.0
Jasmine	10.0	0.0
Frangipani	7.0	1.6

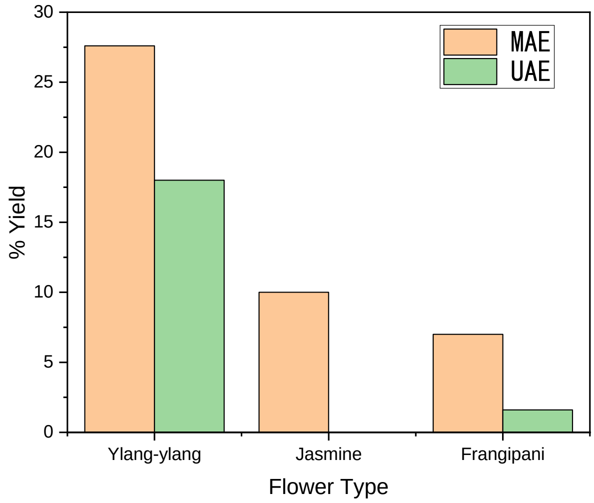


Fig 3. The Effect of Flower Type on Extraction Yield Using MAE and UAE Methods: Ylang-ylang (YY), Jasmine (JA), Frangipani (FP)

Fig. 3 shows the extraction results; the MAE method produced a higher yield for all flower types compared to the UAE method. Using MAE, YY flowers showed the highest yield at 27.6%, followed by JA flowers at 10.0%, and FP flowers at 7.0%. On the other hand, in the UAE method, the highest yield was still obtained from YY 18.0%, while JA and FP showed much lower yields, at 1.6% and 0.0%, respectively. This finding is in line with the research by Utami et al (2020), which shows that MAE provides higher yields than UAE because microwave radiation and rotating vibrations cause increased pressure on cell walls, causing cells to swell and release more active compounds [15].

The differences in extraction results can be explained by the working mechanisms of each method. MAE operates on the principle of microwave heating, which efficiently breaks down cell walls, allowing for the release of active compounds, including essential oils. Microwaves are able to heat both the solvent and sample matrix directly and rapidly, thereby supporting the effective extraction of volatile compounds. This process is particularly advantageous when using solvents such as ethanol, which has a boiling point of around 78°C, as the system can reach temperatures close to this point, facilitating the release and separation of essential oil compounds.

In contrast, the UAE method resulted in lower extraction yields overall, mainly due to technical limitations during the process. The UAE apparatus used could not be tightly sealed, resulting in a maximum temperature of only around 57°C. This temperature is far below the boiling point of ethanol, making the evaporation and release of volatile compounds suboptimal. This had a significant impact on jasmine flowers, which produced no extract at all. This failure indicates that insufficient temperature was unable to evaporate and transport the essential oil compounds from the flower tissues into the solvent. The low extraction temperature below the solvent's boiling point was found to reduce UAE efficiency, as the decreased kinetic energy of the solvent hindered penetration into the cells and reduced the cavitation intensity required to release active compounds [16].

Despite the suboptimal UAE conditions, ylang-ylang flowers still yielded a relatively high amount (18%), most likely due to their higher essential oil content and a floral tissue structure that is more easily disrupted compared to the other flowers. Meanwhile, frangipani produced a very low yield (1.6%) under UAE, indicating that this method is highly dependent on adequate temperature and pressure for efficient extraction.

The unsealed UAE setup also caused the process environment to remain open, allowing evaporated solvent and volatile compounds to escape into the air. The lack of internal pressure, which is essential for optimal cavitation, further reduced the efficiency of extraction. This condition hindered the release of active compounds from the flowers, even though the polar solvent used is capable of dissolving various bioactive components.

Overall, the results indicate that ylang-ylang is the most promising raw material, as it yields a high amount of extract using both MAE and UAE. In contrast, jasmine and frangipani are better suited for extraction methods that provide high temperatures and a closed environment, such as MAE. The performance of the UAE is highly dependent on precise temperature control and a sealed system to ensure effective cavitation and evaporation of volatile compounds.

The best extract result obtained in this study was essential oil from frangipani flowers, the structure of which will be identified using FTIR analysis. **Fig. 4** shows the FTIR spectrum of the ylang-ylang flower extract, indicating the presence of several functional groups associated with the active compounds that make up essential oils.

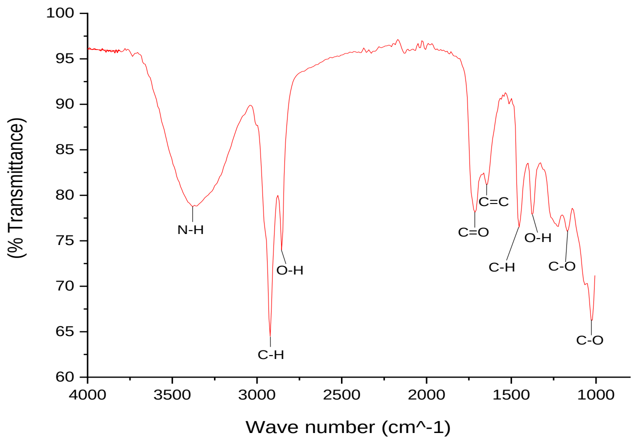


Fig 4. FTIR Analysis of Ylang-Ylang Flower Extract

Table 2 shows the results of FTIR analysis of YY essential oil showed various characteristic absorption peaks that illustrate the presence of important functional groups in its chemical composition. The first absorption appears at a wave number of 3362 cm^{-1}, which indicates the presence of an N-H group with a characteristic range of 3400-3300 cm^{-1}. The presence of this group indicates the possible presence of primary or secondary amine compounds, although nitrogen compounds are rarely found in pure essential oils. This could be from protein contamination or biological degradation during the extraction process [17].

Table 2. Range of Compounds Present in FTIR Testing of Ylang-Ylang Extract

Type of Oil	Wave Number cm^{-1}	Type of Bond	Wave Number Range cm^{-1}
YY essential oil	3362	N-H	3400-3300
	2922	C-H	3000-2840
	2855	O-H	3200-2700
	1714	C=O	1725-1705
	1654	C=C	1658-1648
	1453	C-H	1470-1440
	1379	O-H	1390-1310
	1162	C-O	1205-1124
	1028	C- O	1300-100
	894	C=C	895-885
	827	C=C	840-790

Furthermore, the absorption at 2922 cm^{-1} falls within the range of 3000-2840 $cm^{-1,}$ which is an aliphatic C-H stretch, indicating the presence of straight-chain hydrocarbon compounds such as those found in linalool or geraniol. An additional absorption at 2855 cm^{-1}, in the range of 3200-2700 cm^{-1}, indicates the presence of O-H groups, which also strengthens the notion that alcoholic compounds are the dominant components of YY oil [18].

The strong peak at 1714 cm^{-1} falls within the range 1725-1705 cm^{-1} and indicates the presence of carbonyl groups (C=O) common in ester compounds, such as benzyl acetate, a major contributor to the characteristic floral aroma. The absorption at 1654 cm^{-1} with a range of 1658-1648 cm^{-1} is characteristic of a C=C double bond, both in alkene and conjugated aromatic structures. Another absorption appears at 1453 cm^{-1}, which corresponds to the range 1470-1440 cm^{-1}, indicating bending of the C-H group in aliphatic structures. The absorption at 1379 cm^{-1} (range 1390-1310 cm^{-1}) again indicates the presence of O-H groups, possibly from different secondary alcohols [17].

Furthermore, the absorption peak at 1162 $cm^{-1,}$ which is in the range of 1205-1124 cm^{-1}, as well as 1028 cm^{-1} in the range of 1300-1000 cm^{-1}, both indicate the presence of C-O groups, which are commonly found in alcohols, ethers, and esters. Finally, the absorptions at wave numbers 894 cm^{-1} (range 895-885 cm^{-1}) and 827 cm^{-1} (range 840-790 cm^{-1}) indicate the stretching of the C=C group, which corroborates the presence of aromatic compounds or terminal alkenes in the oil composition[19]. Overall, the FTIR results show that ylang-ylang essential oil is rich in alcohols, esters, saturated hydrocarbons, and aromatic compounds, all of which play an important role in giving the essential oil its characteristic aroma and functional potential.

Characteristic Analysis

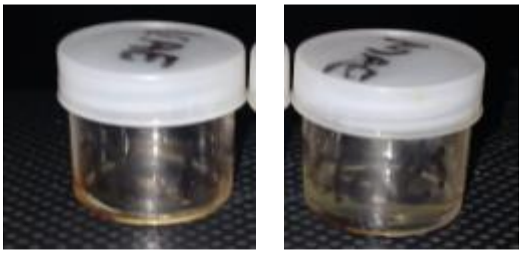
Fig. 5 shows the results of essential oil extraction from YY flowers using MAE and UAE techniques. The initial extract was then concentrated using a rotary evaporator to remove residual solvents, resulting in a final volume of 13.8 mL. The resulting color was dark brown. This indicates that ylang-ylang flowers have the highest extraction yield. This is consistent with national reports stating that extraction efficiency with microwave assistance is greatly influenced by the solvent-to-material ratio and microwave power, which affect the release of volatile compounds in plant tissue [20].

(a)

(b)

Fig 5. YY flowers were extracted using the MAE method **(a)** and the UAE method **(b)**.

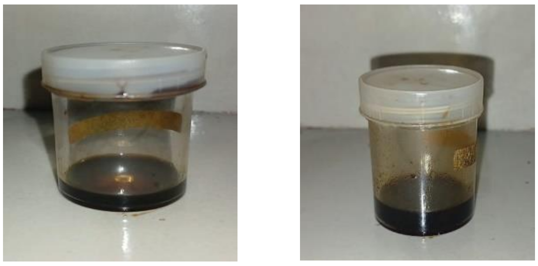
Fig. 6 shows the results of essential oil extraction from JA, where part A shows the extract obtained through the MAE method, while part B shows the results of the UAE method. The resulting color is pale yellow. All samples underwent a concentration process using a rotary evaporator to remove residual solvents. Based on these results, the MAE method produced an extract volume of 5 mL, while the UAE method did not produce any extract, 0 mL. This is due to difficulties in obtaining raw materials, the use of solvents that are not well-suited to the extract material, and an insufficient extraction time, resulting in a less optimal extraction process.



(a) ***(b)***

Fig 6. JA flowers were extracted using the MAE method **(a)** and the UAE method **(b)**.

Fig. 7 shows the results of essential oil extraction from FP flowers using two methods, namely MAE (A) and UAE (B). Both extracts are pale yellow in color, indicating the presence of volatile compounds in moderate concentrations. After undergoing solvent removal using a rotary evaporator, the yield obtained from MAE was 3.5 mL, while UAE yielded 1.6 mL. These results indicate that MAE is more efficient than UAE in extracting essential oil from FP flowers.



(a) (b)

Fig 7. FP flowers were extracted using the MAE method **(a)** and the UAE method **(b)**.

Table 3 shows the data obtained; the three types of flowers, YY, JA, and FP, have distinct physical and chemical characteristics. Frangipani and Jasmine share similarities in terms of color and aroma, both having a pale-yellow color and lacking any noticeable scent.

Table 3. Characteristic Analysis Flower

Parameter	Unit	YY	JA	FP
Color	-	Brownish	Pale yellow	Pale yellow
Odor/Aroma	-	Strong YY flower aroma	Odorless	Odorless
FFA	%	0.40	0.34	1.45
pH	-	4	4.3	5
SNI	-	SNI-8028-1-2014 Minyak Atsiri [21]	Extraction of Jasmine Essential Oil[22]	SNI-8028-1-2014 Minyak Atsiri[21]

The pale-yellow color indicates a low level of oxidation, while the absence of aroma suggests a low content of essential oils or volatile compounds in both flowers. In contrast, YY has more prominent characteristics, with a dark brownish color and a strong ylang-ylang fragrance. The dark color may indicate the degradation of active compounds or a more intense extraction process, while the strong aroma signifies a high essential oil content, making it potentially valuable for applications in the perfume or aromatherapy industry [17].

Free Fatty Acid Analysis

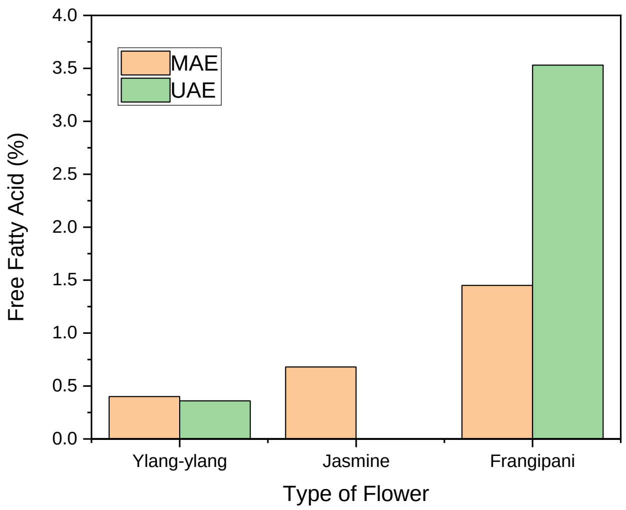


Fig 8. The Effect of Flower Type on %FFA Using the MAE and UAE Methods: Ylang-ylang (YY), Jasmine (JA), Frangipani (FP)

Fig. 8 shows that FFA are weak acids that are produced through the hydrolysis of fats, resulting in the formation of glycerol and free fatty acids [23]. The type of ylang-ylang flower has the smallest FFA percentage value. Based on the titration results, the volume of NaOH required to reach the endpoint was 2 mL. By applying this value to the calculation formula, the FFA content was determined to be 0.40%. This value indicates that the extracted essential oil contains a low level of FFA, reflecting good oil quality and the absence of lipid degradation due to hydrolysis or oxidation during storage or processing. An increase in FFA levels can occur due to lipase enzyme activity triggered by uncontrolled temperatures, high humidity, or microbial contamination [24]. The low FFA level also supports the potential use of this essential oil in cosmetic or aromatherapy applications, where compound stability and purity are critical.

Moreover, the low FFA content suggests that the essential oil is relatively stable against oxidation, which is an important factor in preserving the aroma, color, and bioactive efficacy of the final product. Increased FFA levels have been shown to affect aroma quality due to a decrease in aroma-forming aldehyde compounds, such as hexanal and (E)-2-hexenal. Furthermore, phenolic compounds associated with oxidative stability and FFA levels also play a role in determining the color and sensory properties of the oil [25]. This enhances the suitability of the essential oil for commercial formulations that require extended shelf life and consistent quality [26].

4. CONCLUSION

Based on the results of essential oil extraction from ylang-ylang, jasmine, and frangipani flowers using Microwave-Assisted Extraction (MAE) and Ultrasonic-Assisted Extraction (UAE) methods, it was found that the type of flower and extraction method significantly affected the extract yield obtained. Among the three samples, ylang-ylang flowers yielded the highest results, particularly through the MAE method, with a yield of 27.6%. FTIR spectrum analysis of the ylang-ylang flower extract revealed the presence of functional groups such as hydroxyl (–OH), carbonyl (C=O), alkyl (C–H), and double bonds (C=C), indicating the presence of bioactive compounds such as linalool, eugenol, and geranyl acetate as the main components of the essential oil. Meanwhile, the UAE method is also capable of extracting essential oil from ylang-ylang flowers, but its efficiency is highly dependent on the technical settings of the equipment. Therefore, to enhance the effectiveness of the UAE, it is recommended to use a closed system with higher temperature control to create internal pressure that supports the cavitation process while preventing the loss of volatile compounds to the environment.

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