

Microwave Assisted Hydrodistillation Essential Oil from Lime Peel Waste as Aromatherapy Candles

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Received: March 2023

Received in revised: May 2023

Accepted: June 2023

Available online: September 2023

Abstract

Flavonoids belong to the extensive group of polyphenol compounds. They can function as antioxidants and antibacterials by denaturing bacterial cell proteins and damaging bacterial cells. One source of essential oils is lime peel, which is often discarded and goes unused. The distillation method utilizes microwaves, offering an alternative to conventional distillation techniques and resulting in enhanced effectiveness and efficiency. The Microwave Assisted Hydrodistillation (MAHD) method proves to be more energy-efficient, environmentally friendly, rapid, safe, and cost-effective. This study aims to assess the impact of the MAHD method on the quantity and quality of essential oils extracted from lime peels. Laboratory tests are conducted in accordance with Indonesian national standards. The overarching objective is to establish a process that transforms discarded lime peels into aromatherapy candles, contributing to respiratory health. The MAHD method is employed with a sample mass to distilled water solvent ratio of 1:1. The resulting essential oil undergoes analysis using the GC-MS method. According to the GC-MS test results, the compound with the highest percentage area is limonene at 98%. This is followed by trans-caryophyllene at 99% with a retention time of 12.422, and beta-selinene at 99% with a retention time of 13.191.

Keywords: Aromatherapy Candles, lime peel waste, essential oil, microwave assisted hydrodistillation

INTRODUCTION

Citrus aurantifolia, commonly known as lime, thrives in the wild across southern Asia, Japan, and Indonesia. This plant flourishes in tropical climates and is characterized by its vibrant white flowers. The resulting fruit boasts an intensely sour flavor, while its skin is thin and either green or yellow. The chemical composition of essential oils produced by Citrus aurantifolia plants from Cameroon includes Limonene (53.92%), α -pinene (0.33%), mirin (1.58%), β -pinene (0.97%), sabinene (2.06%), and sokemen (0.56%) (Irdiansyah, 2017).

Flavonoid compounds are extensively present in plants, demonstrating notable antioxidant activity. (Mulyati & Panjaitan, 2021). Natural antioxidants are sourced from plants, and they typically encompass phenolic secondary metabolites present in various plant components, including fruits, seeds, flowers, leaves, pollen, wood, and roots. Antioxidants function as inhibitors, curtailing the oxidation process by interacting with reactive free radicals to generate non-reactive free radicals. These non-reactive radicals

exhibit a certain stability, thereby offering cellular protection against the detrimental impacts of reactive oxygen free radicals (Gus Mahardika & Fajri, 2023).

Orange essential oil serves multiple purposes, finding application as an air freshener, a component in perfumes, and a flavor enhancer in food. This citrus-derived essential oil also confers health benefits, particularly in aromatherapy. The citrus fragrance holds the ability to stabilize the nervous system and evoke pleasurable sensations. This effect is attributed to the essential oil content within orange peel, which imparts an aroma capable of steadying the nervous system, thereby enabling the creation of aromatherapy products.

Aromatherapy takes various forms, encompassing essential aromatherapy oils, aromatherapy incense, aromatherapy candles, aromatherapy massage oils, aromatherapy salts, and aromatherapy soaps. Given the advantageous health properties associated with essential oils extracted from orange peel, it becomes imperative to conduct research into their practical utilization. (Lestari et al., 2020).

The preliminary test results have demonstrated that the lime peel essential oil (*Citrus aurantifolia*) from "X" brand, when utilized at a 100% concentration via the excellent diffusion method, effectively impeded the growth of *pittosporum* fungus in vitro on Sabouraud Dextrose Agar (SDA) media. This inhibition yielded an expansive inhibitory zone measuring 30 mm. Further analysis of the test results encompassed the identification of the essential oil generated through GC-MS. This analysis revealed the presence of limonene compounds bearing the molecular formula $C_{10}H_{16}$ within the orange peel essential oil. These limonene compounds were identified with a retention time of 7.01 minutes, constituting a substantial percent area of 96.79%. (Yustinah & Fanandara, 2010).

The Megawati study (2015) employed the GC-MS test to ascertain the chemical compound composition within lime peel essential oil. The GC-MS method offers advantages such as rapid analysis, high sensitivity, favorable precision, and a long-standing history of usage. In a separate investigation conducted by Souhoka et al. (2020), GC-MS was employed to identify the chemical constituents of patchouli oil. The researchers distilled patchouli leaves for a duration of 2 hours and subsequently subjected the obtained patchouli oil to GC-MS analysis. The outcomes of this analysis unveiled a total of 13 chemical components within patchouli oil, with notable proportions of *trans*-caryophyllene (7.96%), patchouli (2.88%), and cyclohexanone (1.10%). The process of distillation is commonly employed to extract essential oils. The utilization of microwaves presents an alternative to traditional distillation methods, offering enhanced effectiveness and efficiency. The Microwave Assisted Hydrodistillation (MAHD) method, as demonstrated by Gude et al. (2012), surpasses other extraction techniques in terms of energy efficiency, environmental friendliness, speed, safety, and cost-effectiveness.

Further research, conducted by Utomo & Wihadi (2022), delves into microwave-based extraction. This study highlights that microwave heating in the extraction process provides superior temperature control compared to conventional heating methods. This is exemplified in the extraction of chemical components from Dengen leaves (*Dillenia serrata* Thunb), where the microwave approach leads to shorter extraction times, reduced energy and solvent consumption, heightened yields, improved accuracy and precision, enhanced mass transfer, and equipment

setups that synergize both Soxhlet and microwave attributes.

Megawati et al. (2015) conducted a study titled "Microwave-Assisted Hydrodistillation for the Extraction of Essential Oil from Grapefruit Peels for Aromatherapy Candles." This research concluded that both the mass of the material and microwave power significantly influenced the volume of grapefruit peel essential oil produced. The chemical-physical tests yielded the highest essential oil yield of 0.54%. The density was determined to be 0.810 g/ml, and the essential oil was fully soluble in 95% alcohol at a 1:6 ratio. The GC-MS analysis revealed the two primary components of grapefruit peel essential oil using the Microwave Assisted Hydrodistillation (MAHD) method: limonene (93.99%) and β -pinene (3.20%). Consequently, grapefruit peel essential oil is suitable for application as aromatherapy candles.

Erliyanti et al. (2020) carried out research involving the extraction of essential oils from frangipani flowers using the microwave hydrodistillation method. The study demonstrated that increasing microwave power and solvent volume correlated with an increase in essential oil density. The highest density was achieved with a 50-gram frangipani flower mass, 600-watt microwave power, and 600 ml solvent volume, resulting in a density of 0.904 gram/ml. Aromatherapy candles offer an alternative way to experience aromatherapy through inhalation. This entails inhaling the aroma vapors generated by adding a few drops of essential oils to a container of hot water. Aromatherapy candles, when burned, emit therapeutic aromas, as highlighted by Rusli & Rerung (2018).

The peel of lime contains flavonoid compounds, namely naringin, hesperidin, naringenin, hesperetin, rutin, nobiletin, and tangerine. Flavonoids are the most extensive polyphenol compounds that can work as antioxidants and antibacterials by denaturing bacterial cell proteins and damaging bacterial cells. Flavonoids can also inhibit the Glucosyltransferase (GTF) activity from *Streptococcus mutans*.

Regarding previous studies by Erliyanti et al. (2020) and Megawati et al., (2015) in this study aims to determine the Microwave Assisted Hydrodistillation method on the quantity and quality of essential oils produced from lime peels through laboratory tests according to Indonesian National Standards (INS) to determine the processing of lime peel waste into aromatherapy candles that function for health.

METHODOLOGY

Materials and Instrumentals

The study utilized various materials and tools for its research purposes. The materials included lime peel, distilled water (Water One), paraffin (technical grade), stearic acid (technical grade), n-hexane, and anhydrous sodium sulfate. The tools employed in the study comprised a Microwave (SAMSUNG), glass beaker (IWAKI PYREX), volume pipette (IWAKI PYREX), condenser, distillation flask, Erlenmeyer flask, split funnel (IWAKI PYREX), dropper pipette, measuring pipette (IWAKI PYREX), digital scales (OHAUS), pycnometer (IWAKI PYREX), refractometer (Abbe WAY-2S refractometer), blender (COSMOS), tray, knife, aluminum foil, thermometer, stirrer, wax container (mold), wax wick, match, and scissors. The study also employed a Gas Chromatography-Mass Spectrometry (GC-MS) system, specifically the Thermo Scientific ISQ 7610 Single Quadrupole GC-MS, for analytical purposes.

Microwave Assisted Hydrodistillation (MAHD) Lime Peel Essential Oil

In the experimental process, a lime peel powder sample weighing 300 grams was carefully weighed out. This sample was then placed into a distillation flask. Subsequently, 300 ml of distilled water solvent was added to the flask, maintaining a sample-to-solvent ratio of 1:1. The assembled distillation flask was introduced into the Microwave and connected to a condenser.



Figure 1. Microwave Assisted Hydrodistillation (MAHD) lime peel essential oil

Upon initiating the experiment, the Microwave was activated, and specific power levels and heating durations were set. The variation in power and time played a crucial role in the distillation procedure

using the Microwave. Specifically, power variations of 300, 450, and 600 watts were employed in the distillation process to assess their effects. The study also involved variations in time, specifically 30, 60, and 90 minutes. After the distillation process, the resultant distillate within the split funnel was allowed to stand undisturbed for a duration of 3 hours. During this time, two distinct layers formed: the upper layer comprised lime peel essential oil, while the lower layer consisted of water. The volume of lime peel essential oil was quantified and subsequently transferred into a suitable container. Figure 1 illustrates the process of Microwave-assisted hydrodistillation for extracting lime peel essential oil.

Comparatively, the current research differs from the earlier study on Hydrodistillation (2015), which employed grapefruit peel as the raw material. In that study, the mass of raw materials was varied, specifically 50, 75, 100, 125, and 150 grams.



Figure 2. Lime Peel Essential Oil

Figure 2 illustrates the image of lime peel essential oil that was acquired through the Microwave-assisted hydrodistillation process. This essential oil is now prepared for subsequent analysis, including measurements of density, refractive index, and Gas Chromatography-Mass Spectrometry (GC-MS) analysis.

Density Measurement

Pycnometer (5 ml) is cleaned and oven-dried at 100°C for 10-20 minutes. The pycnometer is removed and put in the desiccator for 10-15 minutes. Recorded the Volume of the pycnometer used, weighed an empty pycnometer, and recorded it as a gram. Inserted the sample (lime peel essential oil) into the pycnometer to the top of the neck and put the lid on it until the sample can fill the capillary pipe to the brim. It is ensured that there are no air bubbles inside the pycnometer, dried the outside with a tissue, weighed the pycnometer containing the sample, and recorded as b gram. To calculate of density/specific gravity of lime peel essential oil was used Equation 1.

$$\rho = \frac{\text{mass}}{\text{volume}} \quad (1)$$

Refractive Index Measurement

Lime peel essential oil is dripped at the place of the sample refractometer, tightly closed, and allowed the light to pass through the solution and the prism so that the light on the screen in the device is divided into two. Digest the boundary sign by rotating the regulating knob, thus cutting the intersection point of two intersecting diagonal lines that intersect each other visible on the screen. Recorded the results obtained.

Analysis of Lime Peel Essential Oil Using GC-MS

Components of chemical compounds Lime peel essential oil was analyzed using GC-MS in the chemical laboratory of the Forensic Laboratory (Bidlabfor) of the South Sulawesi Regional Police.

Aromatherapy candle production

Weighed as much as 45 grams of stearic acid and 15 grams of paraffin (3:1), heated to dissolve. Measured the temperature of the solution to a temperature of 40°C, then added 2 ml of lime peel essential oil. Put into a wax container that has been placed a wick in the center of the container. Allowed to stand until the wax hardens. Figure 3 presents the process of making aromatherapy candles from lime peel essential oil.



Figure 3. Aromatherapy candles from lime peel essential oil

RESULTS AND DISCUSSION

Effect of Power (P) MAHD

The variation in the Power (P) of MAHD in this study was 300, 450, and 600 watts, with a ratio of sample mass and fixed solvent (1: 1) (Figure 4). Figure 4 visually presents the outcomes of the experiment, highlighting the variation in essential oil

volume at different power levels during the Microwave-assisted hydrodistillation process.

At 450 watts of power, the most significant volume of essential oil, measuring 2 ml, is obtained. When operating at 300 watts of power, only 1 ml of essential oil is produced. This result stems from the fact that at this power level, the solution consisting of lime peel and distilled water hasn't yet reached its boiling point under a pressure of 1 atm. Conversely, when utilizing 600 watts of power, the volume of essential oil decreases slightly to 1.5 ml. This reduction is attributed to the potential for scorching in the material when excessive power is applied with a relatively small mass of distilled material. Similarly, if an extensive power supply is combined with a substantial mass of distilled material, there's a risk of overflow, which may lead to the spilling of both materials and solvents. (Anggia, Fela Tri, Yuharmen, 2014).

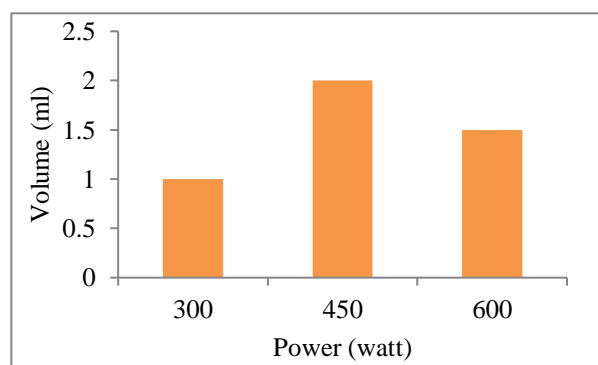


Figure 4. Effect of power (P) MAHD on the volume of lime peel essential oil

Effect of MAHD Time (t) on the Volume of Lime peel essential oil

The time variation (t) applied in this study for Microwave Assisted Hydrodistillation (MAHD) included intervals of 30, 60, and 90 minutes, maintaining a fixed sample mass to solvent ratio of 1:1 (as shown in Figure 5). In Figure 5, it's evident that at the 60-minute mark, a substantial volume of essential oil, measuring 2 ml, is generated. At the 30-minute interval, only 0.8 ml of essential oil is produced. This result stems from the fact that within this short time frame, the solution comprising lime peel and distilled water hasn't received sufficient heat from the Microwave, resulting in a limited release of oil.

However, when the duration is extended to 90 minutes, the volume of essential oil decreases to 1.3 ml. This decrease is attributed to the depletion of available oil cells that can be evaporated during the extraction process.

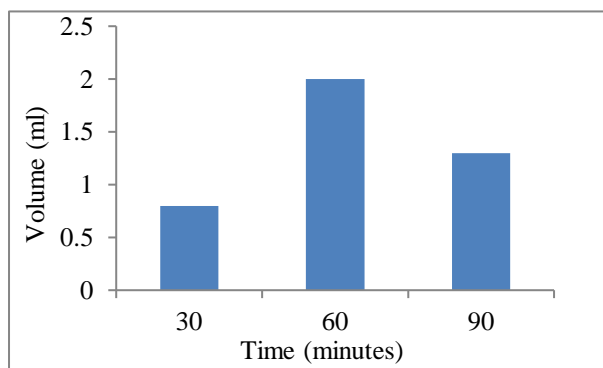


Figure 5. Effect of MAHD time (t) on the volume of lime peel essential oil

Based on these graphs (Figure 5), the Microwave-assisted hydrodistillation method produces very little essential oil. This research is the latest in the raw materials used, namely raw materials from lime peels, so researchers need to do trial and error several times to get essential oils with more Volume. The following research plan is to add research variables to raw materials and solvents, to vary the mass of raw materials and solvents, and to increase the mass of raw materials and solvents.

Yield (%) lime peel essential oil

The yield (%) of lime peel essential oil from the variation in power and time (450 watts, 60 min) obtained was 0.5378% (Table 1).

Refractive index

Refractive index testing is a valuable analysis technique used to assess the purity of oils. The refractive index value, along with the type and weight of the substance, is crucial for comparing and distinguishing between different components of compounds within the oil. This information aids in identifying the composition and characteristics of the oil sample under examination.

Essential oils of high quality are characterized by having a substantial refractive index value (Chandra Kartika Fitri & Kartika Widyastuti, 2020). The obtained refractive index of lime peel essential oil was recorded as 1.463 at a temperature of 20°C. This refractive index value aligns with the quality criteria outlined in INS 06-3953-1995, where the specified range for essential oil refractive index is 1.466 to

1.475. This indicates that the lime peel essential oil meets the required standards for refractive index, indicating a level of quality that aligns with established benchmarks.

Table 1. Observation Yield (%) essential oil

| The yield of essential oils with variation of power (P) t=60 minutes | | | | |
|--|------------------|-----------------|---------------|-----------|
| Power (Watt) | Sample mass (gr) | Oil volume (ml) | Oil mass (gr) | Yield (%) |
| 300 | 300 | 1 | 0.5938 | 0.1979 |
| 450 | 300 | 2 | 1.6135 | 0.5378 |
| 600 | 300 | 1.5 | 1.0225 | 0.3408 |
| The yield of essential oils with time variation (t) with P=450 watts | | | | |
| Time (minutes) | Sample mass (gr) | Oil volume (ml) | Oil mass (gr) | Yield (%) |
| 30 | 300 | 0.8 | 0.2781 | 0.0927 |
| 60 | 300 | 2 | 1.6135 | 0.5378 |
| 90 | 300 | 1.3 | 0.8023 | 0.2674 |

Density/Specific Gravity

The specific gravity of the obtained lime peel essential oil was measured at 0.845 g/ml at a temperature of 25°C. This specific gravity value falls within the range specified by INS 06-3953-1995 quality standards for essential oils, which is 0.875 to 0.893 g/ml. This suggests that the specific gravity of the lime peel essential oil is consistent with the quality criteria outlined in the standard, indicating that the essential oil aligns with the required specifications for specific gravity.

Aromatherapy candle

Aromatherapy candles crafted from the essential oil of lime peel offer an alternative means of utilizing aromatherapy through inhalation. This method can serve as a substitute for analgesic, antihistamine, antitussive, antipyretic, decongestant, and expectorant medications, particularly for individuals dealing with colds and coughs a respiratory infection primarily affecting the upper respiratory tract, including the nose.

Aromatherapy, as explained by Maftuchah et al. (2020), represents a non-pharmacological therapeutic approach that harnesses the aromatic properties of essential oils distilled or extracted from various plant parts. These aromas are employed to promote healing and well-being. In the context of your study, the use of aromatherapy candles made from lime peel essential oil provides an avenue for individuals to

experience the potential benefits of aromatherapy as an aid for respiratory health.



Figure 7. Aromatherapy candle

Subsequent steps in the research process will involve assessing the efficacy of the lime peel essential oil aromatherapy candle and refining the packaging system to prepare the product for the market. These follow-up evaluations will help determine the effectiveness of the aromatherapy candle in delivering the desired therapeutic benefits, as well as ensuring that the packaging meets market standards and consumer expectations. Figure 7 visually illustrates the outcomes and findings derived from the process of creating aromatherapy candles using lime peel essential oil. It likely encapsulates the various stages, components, and results of the study in a graphical representation.

Assisted Hydrodistillation (MAHD) method, was subjected to analysis using Gas Chromatography-Mass Spectrometry (GC-MS). The quantity of chemical components is represented in terms of % area, which is determined based on the identified compounds from the GC-MS results. The graphical representation of the GC-MS test outcomes for lime peel essential oil utilizing the MAHD technique is depicted in Figure 8.

The GC-MS test outcomes reveal the presence of numerous terpenoid compound components, with some of the highest percentage areas corresponding to specific retention times. For instance, at a retention time of 6.045, the limonene compound constitutes 98% of the area. At retention time 12.422, the trans-caryophyllene compound constitutes 99% of the area, while at retention time 13.191, the beta-selinene compound also constitutes 99% of the area. Many terpenes, which are essential oil constituents, are naturally synthesized by plants, including lime peel. Notably, limonene is identified as a hydrocarbon compound classified within the terpene group. This compound, known for its citrus-like scent, is typically a liquid at room temperature.

The test outcomes align with prior research conducted by Megawati & Murniyawati (2015), where grapefruit peel oil exhibited the most prominent content of Limonene, comprising 93.99%

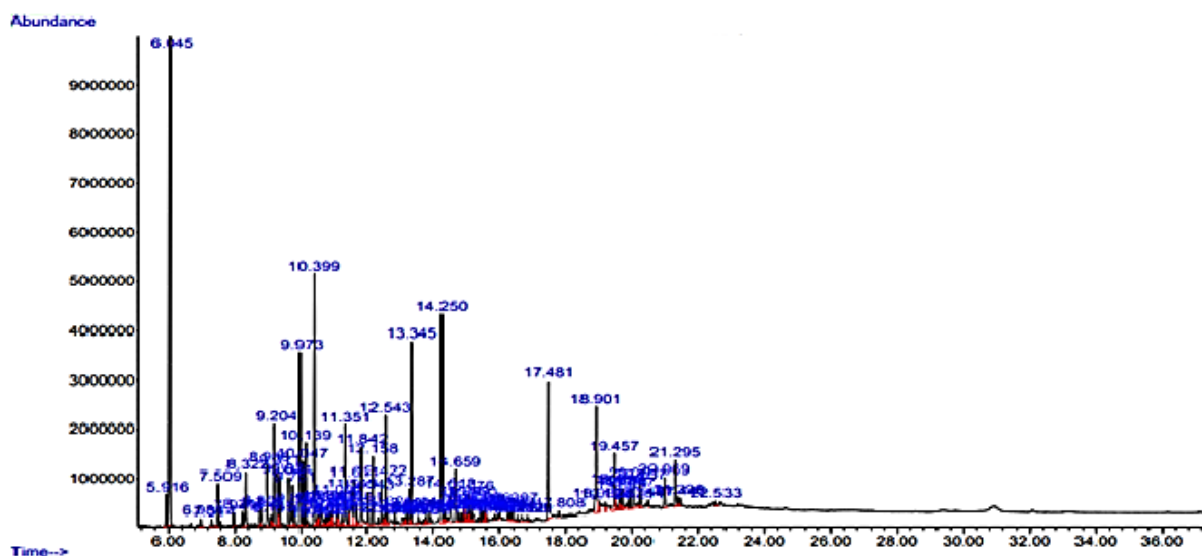


Figure 8. Chromatography of the results of the analysis of GC-MS essential oil from lime peel

GC-MS Analysis

The chemical composition of the essential oil derived from lime peel, utilizing the Microwave

of the total. It's worth noting that the presence of several peaks in the graph could be attributed to impurities within the sample during the analysis.

CONCLUSION

Variations in the distillation conditions involving microwave power and time have a direct impact on the quality characteristics of essential oils. When employing a power variation of 450 watts and a time duration of 60 minutes, the quantity of lime peel essential oil obtained is 2 ml, resulting in a % Yield of 0.5378%. This indicates the efficiency of the extraction process under these specific conditions. The quality assessment of essential oils extends to the results of physical tests. Notably, the refractive index of the lime peel essential oil is measured at 1.463 (at a temperature of 20°C), while the density of the essential oil is recorded as 0.845 g/ml. These physical properties provide insights into the composition and characteristics of the essential oil. Chemical analysis using GC-MS identifies Terpenoid compounds in the lime peel essential oil. Notably, Limonene constitutes 98% of the composition at a retention time of 6.045. Similarly, Trans-caryophyllene comprises 99% at a retention time of 12.422, and beta-selinene accounts for 99% at a retention time of 13.191. Collectively, these findings indicate that lime peel essential oil, produced using the Microwave Assisted Hydrodistillation method, possesses the necessary qualities to be utilized as an aromatherapy candle for promoting health and well-being.

ACKNOWLEDGMENT

The researchers extend their gratitude to the Ministry of Education and Culture for generously funding the Basic Research Grant (PDP) in the year 2022. Special appreciation is also extended to the Rector of Sulawesi University of Technology for their unwavering support and valuable guidance throughout the course of this research endeavor. Heartfelt thanks are due to fellow colleagues, lecturers, and students for their collaborative efforts in conducting this research and contributing to the compilation of this scientific article.

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