Chemical Composition and Physical Characteristics of Orange Peel Essential Oil

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Abstract. The citrus-based food and beverage industry is limited to using fruit flesh as the main raw material. Orange peels that cannot be utilized are disposed of as waste. The potential of citrus peel is very high to be processed into essential oil. Essential oils are widely used in the pharmaceutical, food, and agricultural industries. Various citrus varieties in Indonesia have different characteristics, including the content of essential oils in peel waste. The process of making essential oils can be done by distillation. This research was conducted to find out the chemical and physical of various essential oils from citrus varieties (RGL Mandarin, Gamindo B, and Montaji Agrihorti lemon). The test results show that the yield of each essential oil of RGL Mandarin, Gamindo B, and Montaji Agrihorti lemon is 3,866%; 1,174%; and 3,615%. The volatile compound content tested using GC-MS in Gamindo B citrus essential oil contained 3 compounds, RGL Mandarin found 4 compounds, and Montaji Agrihorti lemons found 4 compounds. A sensory preference test on color and odor attributes was conducted on 30 panelists. Montaji Agrihorti Lemon essential oil is the most preferred by color and Gamindo B essential oil is the most preferred by odor.

1 Introduction

Citrus plants are annual plants originating from the continent of Asia, especially from India to China. Many species of citrus are in subtropical cultivation. Some of the plants that are native to Indonesia and plants can adapt and even become species of regional excellence. In Indonesia, many citrus plants are found in cultivation starting in the lowlands. Indonesia is a tropical country that has various varieties of citrus such as tangerines, sweet oranges, pomelo oranges, limes, and many more. The majority of citrus grown in Indonesia are tangerine oranges as much as 70%, mandarin as much as 20%, and other citrus variations as much as 10% of the highland [1,2].

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Citrus fruit peels are usually just thrown away as garbage, which is currently becoming a problem in big cities [3]. To solve the problem of waste, one of the usual efforts is to process or recycle waste into useful products or materials, such as organic waste into compost and plastic waste into household appliances [1]. Citrus peels contain essential oils that can be extracted so it has a high selling point. This essential oil is preferred by consumers, especially the medium to high, for health purposes and fragrances [4].
The potential for processing orangecitrus peel waste into essential oil is very high. The utilization of these wastes can increase the economic value of oranges. RGL mMandarin is an orange that comes from the Bengkulu province of Indonesia. RGL mMandarin has a fresh sweet and sour taste so it is widely consumed directly or for processed drinks. Gamindo B oranges are a new superior variety that was released by the Ministry of Agriculture in 2021. Gamindo B oranges come from the Kumquat orange family. Gamindo B oranges are round with characteristic sweet skin. MMontaji aAAgrihorti llLemon is a type of seedless lemon released in 2018. This lemon has a fairly high vitamin C content.
Research on the potential variety of citrus to become essential oils has not been done much, therefore, in this research, the characterization of essential oils from RGL mMandarin, Gamindo B, and mMontaji Agrihorti lemon is carried out to see their physicochemical properties and their potential use for food and non-food products.

2 Material and Method

2.1 Plant Material

RGL mandarin, mMontaji Agrihorti lemon, and Gamindo B were obtained from the waste produced at KPRI Citrus, Batu City. The collected citrusorange peels were then washed and dried using a food dehydrator for 8 hours at 500C. The dried citrusorange peels were reduced in size using a blender to form orange peel powder. The citrusorange peel powder was then processed by distillation for 2 hours to get the essential oil.

2.2 Yield

The essential oil yield was tested by measuring the weight of the starting material and the weight of the distilled oil. the yield calculation formula is as follows: [5]

$$\text{Yield: } \frac{a}{b} x100\% \frac{a}{b} x100\% \quad (1)$$

Description:
a: weight of the essential oil weight
b: material to be distilled

2.3 Acid Level

Testing the acid level iswas done by weighing the essential oil as much as 0.4 grams and adding 10 ml of ethanol solvent. The solution was stirred using a magnetic stirrer until completely mixed. Then, 5 drops of PP indicator were added. The solution was titrated using 0.01 N NaOH solution. The color changed from yellow to pink. Then, the final volume of the titration was recorded [5].

2.4 Specific Gravity

Specific gravity was measured using a pycnometer [5]. The steps are as follows: prepare a dry empty pycnometer and weigh the empty pycnometer (m); fill the pycnometer with distilled water and avoid air bubbles; weigh the pycnometer containing the distilled water (m1); fill the pycnometer with essential oil and avoid air bubbles; and weigh the pycnometer containing the essential oil (m2).

2.5 Ester Number

Testing the ester number in essential oils was carried out using the reflux method. Ethanol and potassium hydroxide solutions were used as blanks. Reflux was carried out for 1 hour, and then the solution was added with a phenolphthalein indicator. Then, it was titrated to produce a color change [6].

2.6 Volatile Compound (GC-MS)

Testing the content of volatile compounds was done with GC-MS model Shimadzu QP-2010 with Agilent J&W DB-1 column type specification, column length 30 meters, diameter 0.25 mm column, 310oC injector temperature, 13.7 kPa pressure, helium carrier gas at a flow rate of 0.50 ml/min. The programmable column temperature with the initial temperature was set at 70oC for 5 minutes, then it was slowly increased by increasing 5oC/min to a final temperature of 260oC and maintained. The separation result by GC is directly analyzed using MS in the following way: comparing patchouli oil fragmentation spectra with standard fragmentation contained in memory [7]

2.7 Sensory Preference

Sensory preference testing was conducted on 30 untrained panelists by assessing color and odor attributes. The panelists were asked to give an assessment ranging from very like to very dislike with a score of 1-5. The higher value given indicates that the panelists highly preferred the essential oil on the attribute [1].

2.8 Experimental Design

This research applied the descriptive method. The data collection was carried out by collecting samples from 3 citrus varieties and then testing the characteristics of the essential oil.

3 Result and Discussion

3.1 Yield

The yield of essential oils is a comparison between the oil yield volatile produced with raw materials to be distilled. The yield of essential oils can be influenced by the condition of the material, the amount of solvent used, the distillation time, and the distillation temperature [8].

Table 1. Yield, Specific Gravity, and Optical Rotation of Orange Peel Essential Oil.

Essential oil type	Yield (%)	Specific gravity	Optical Rotation (^{0})
RGL Mandarin	3.866	0.839	103.364
Gamindo B	1.174	0.838	105.538
Montaji Agrihorti Lemon	3.615	0.847	83.619

Table 1 shows that RGL Mandarin has a yield of 3.866%. The results of a previous study on Mandarin Ponkan peel, with pretreatment in the form of size reduction, resulted in a 3% yield with a distillation time of 2 hours [3]. Nanfeng mandarin peels yield 0,689% [9]. Gamindo B has a yield of 1.174%. Oval Kumquat with steam distillation has a yield of 0.266% [10]. In this study, Gamindo B has a higher yield than an oval kumquat. Meanwhile, Mmontaji lemon has a yield of 3.615%. According to the previous study, the fresh lemon essential oil has a yield of 0.59% [11].

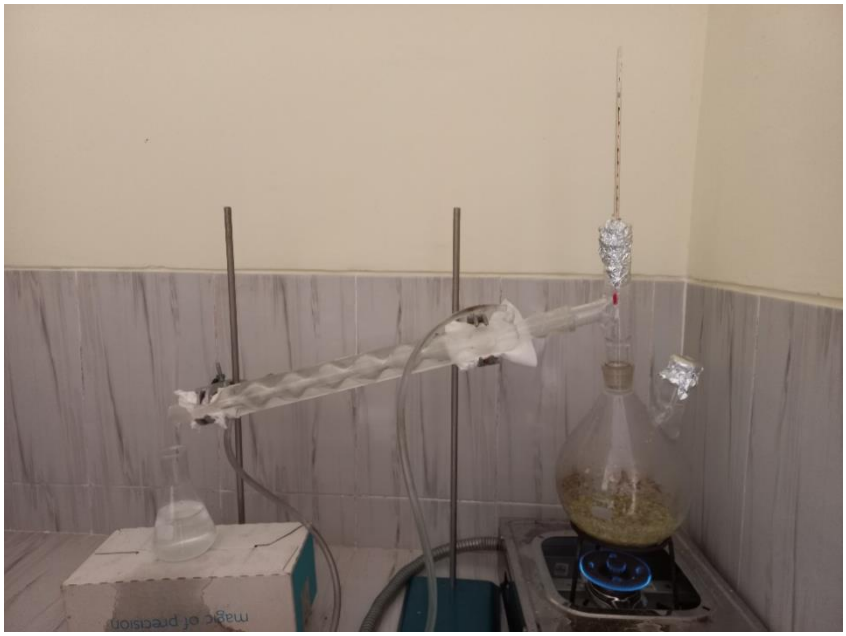

Fig.1 Essential oil distillation process

The yield of essential oils is also affected by the contact between the solvent and the raw material. In addition, the temperature can also affect the yield of essential oils. An increase in temperature causes an increase in the yield of essential oil [8]. In addition, in this study, the dried orange peel was carried out with a preliminary process in the form of size reduction. Size reduction is an attempt to reduce the size of the mechanical work material, into smaller particles. Downsizing It has a variety of styles, namely breaking, cutting, and cutting styles combination.

3.2 Specific Gravity

The specific gravity of essential oil was defined as the ratio between the weight of oil and the weight of water in the same volume as the volume of the same oil [8]. The specific gravity of essential oils was measured using a pycnometer, where the weight of the orange peel essential oil was compared with the weight of water in the same volume.
Table 1 shows that RGL Mandarin has a specific gravity of 0,839 g/ml. In the other study, the specific gravity of orange peel essential oil was 0.843 g/ml. [8]. Gamindo B had a specific gravity of 0,838 g/ml and Montaji lemon of 0,847 g/ml. The essential oil of mandarin peel on oven drying produces oil volatile with a density of 0.836 which is the highest specific gravity compared to the specific gravity of the fresh treatment with a value of 0.834 [12].
The specific gravity of essential oil is highly dependent on the components of its constituent compounds. The most important components of essential oils are compounds of the terpene group. Even though the treatment was used differently, the amount of terpene extracted remained the same, which madee the values of the specific gravity tened to be the same [13]. Based on ISO 3528:2012 regarding Citrus reticulata Blanco essential oil, it was stated that the specific gravity of citrus essential oil is in the range of 0.846 up to 0.854. This study showed that the specific gravity of orange peel essential oil iwas relatively close to ISO [14].

3.3 Optical Rotation

Optical rotation is the response of the molecular structure to the trajectory of single-wave light. The amount of optical rotation depends on the type and concentration of the compound, the length of the path traveled by the light through the compound, and the temperature at which it is measured. Optical rotation analysis is used to know the purity of the distilled oil [15].
Table 1 shows that the highest optical rotation is Gamindo b citrus with 105,538, RGL mandarin 103,364, and the smallest is Montaji lemon with 83,619. The results of the optical rotation of all treatments are positive (+) which means rotating the plane of polarization of light to the right field rotation direction This polarization results from the incorporation of various components of the oil, which is dominated by components having carbon symmetrical with the direction of rotation of the plane of polarization to the right (dextrorotatory) rather than to the left (laevorotatory), thus causing more optical rotation inclined to the right [16–18]. The optical rotation value of citrus peel essential oil was is affected by limonene compounds because limonene was is the most dominant compound in the content of orange peel essential oil [13,19]
For essential oils with completely distilled components, the value of the optical rotation becoame smaller, while for those whose components are not completely distilled, the value of the optical rotation geots higher. This wais because the rotation value optics wais a combination of the components that make up the essential oil [18].

3.4 Volatile Compound

The GC-MS test is used to determine the content or components of chemical compounds found in essential oils. Gas Chromatography-Mass Spectrometry (GC-MS) is a method of separating organic compounds that uses two compound analysis methods, namely gas chromatography and mass spectrometry. Gas chromatography wasis used to analyze the number of compounds quantitatively and mass spectrometry is used to analyze the molecular structure of the analyte compound [20].

Table 2. Volatile components of orange peel essential oil.

No	Compound Name	Concentration Volatile Compound		
		RGL Mandarin	Gamindo B	Montaji Lemon
1	α-Pinene	1.005	-	-
2	Limonen	89.988	98.096	57.463
3	Linalool	0.956	-	-
4	beta-Myrcene	3.203	1,687	4.142
5	gamma-Terpinene	-		11.979
6	2-(2-Mmethyl phenyl) thiirane	-	-	10.917
7	Beta.-Ocimene	-	0.217	-

From the results of the GC-MS test, it is known that 4 volatile compounds were detected in the RGL citrus variety, 3 compounds were detected in Gamindo B, and 4 compounds were detected in the Montaji lemon. Limonin compounds were detected in the 3 varieties. Limonene compounds affect the aroma of essential oils. The amount of limonene compound is influenced by agro-climatic factors (16). Citrus Reticulata Blanco in the study of Chutia, et al [21] had a limonene content of 46.7%. RGL Mandarin is included in Citrus Reticulata, so it is known that the limonene content of RGL Mandarin is higher than other varieties of Citrus Reticulata in previous studies.
Gamindo B has the highest limonin content, which is 98.096% compared to RGL Mandarin and Montaji Agrihorti. Previous studies reported that limonene was the most common challenge found in orange peel essential oil. Previous studies reported that limonene was the most common challenge found in orange peel essential oil. Other hydro-monoterpenes detected by carbon are usually myrcene, -pinene, and sabinene [22–26].

3.5 Sensory Characteristic

Sensory tests were carried out to determine the panelists' preference for the color and aroma of essential oils. Color and aroma are parameters that can indicate the content of the essential oil. Usually, essential oils are not colored or yellowish. The results of sensory testing for color and odor preferences in essential oils can be seen in Table 3.
Montaji Agrihorti Lemon essential oil has the highest color preference value compared to RGL Mandarin and Gamindo B. This is presumably because lemon essential oil has a clearer color than essential oils of other varieties. The lemon essential oil has a bright yellow color while tangerine essential oil has a yellow color [8,11]. The essential oils of the three varieties of orange peel tend to have a yellow and bright yellow color, so the panelists favor this.

Table 3. Sensory evaluation of orange peel essential oil**.**

Essential oil type	Color	Odor
RGL Mandarin	4.467	4.433
Gamindo B	4.677	4.867
Montaji Agrihorti Lemon	4.767	4.633

On the odor parameter, Gamindo B essential oil has the highest preference value compared to RGL Mandarin and Montaji. This is presumably because based on the GC-MS test, Gamindo B contains the highest limonene compound. Limonene compounds affect the odor of essential oils [21]. Each citrus variety has a distinctive odor and affects the final odor of the essential oil. The distinctive smell of citrus essential oil is useful as an aroma enhancer for foods, perfumes, aromatherapy candles, and soap products [13,19].

4 Conclusion

The highest essential oil yield was obtained from RGL Mandarin, which was 3.866%. Montaji Agrihorti essential oil has the highest specific gravity of 0.847. Gamindo B essential oil has the highest optical rotation value of 105,538. The essential oils from 3 citrus varieties contain the highest volatile limonene compounds compared to other volatile compounds. Montaji Agrihorti lemon essential oil has a light yellow color so it is favored by panelists while Gamindo B essential oil has the highest limonin content so it has a strong aroma and is favored by the panelists.

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