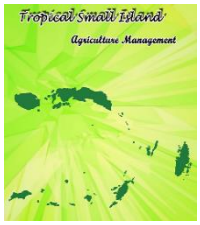

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Isolation and Identification of Essential Oil Made From Kisar Sweet Orange Peels

Gelora Helena Augustyn[1], Anthonia Matjora[2], Vita Novalin Lawalata[3], Sophia Grace Sipahelut[4*]

[1,2,3,4] *Department of Agricultural Product Technology, Faculty of Agriculture, Pattimura University, Indonesia*

Email Corresponding Author: sipahelut.grace@gmail.com

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ABSTRACT

Kisar sweet orange is an endemic fruit and primary commodity from Kisar Island, Southwest Maluku Regency - Maluku Province. Currently, Kisar sweet orangeswere only consumed as fresh fruit during this time, partially processed into juice and the peels were discarded as wastes. Kisar sweet orange peels can be processed into essential oil to increase their economic value. The essential oil made from orange peels is highly preferable, especially for the middle to upper-class consumers, for health and air-freshening purposes. This essential oil has a higher selling value. This research aims at isolating and identifying the chemical components and quality of essential oil made from Kisar sweet orange peels. This essential oil was isolated using a steam distillation method, and its chemical components were identified using GC-MS instruments.. The research results showed that the essential oil made from Kisar sweet orange peels dried with a cabinet dryer for two days utilizing the steam distillation method provides a yield of 0.53%, with its properties of specific gravity of 0.8457, refractive index of 1.4651 (20°C), acid number of 1.92, ester number of 8.05, and solubility in alcohol of 1:9. The main chemical component of the essential oil made from Kisar sweet orange peels detected by GC-MS is limonene (70.80%).

Introduction

Kisar sweet orange is one endemic fruit cultivated from generation to generation by the people of Kisar Island, Southwest Maluku Regency - Maluku Province. Kisar sweet orange

has become the local pride and a primary commodity on Kisar Island. It is nationally recognized as one of the superior orange varieties, classified as a small orange with a different shape and taste than the other sweet orange varieties outside the Maluku region. One Kisar sweet orange tree may produce up to 800-1000 oranges each season. Currently, Kisar sweet orange is only consumed as fresh fruit, partially processed into juice, and eventually, the peels are discarded as waste. The thrown-away Kisar sweet orange peels may be processed to increase their economic value as if they were processed into an essential oil with a higher economic value. Due to rapidly growing and developing industrial needs, Indonesia's essential oil demand is enormous, both in volume and type. The essential oil export demands are continuously increasing that the development opportunities for both developed and new essential oils are still widely open (Zulnely, Gusmailina, & Evi, 2015).The essential oil made from orange peels is highly preferable for health and air-freshening purposes, especially for middle to upper-class consumers. This essential oil has a higher selling value (Hidayati, 2012). Citrus essential oils (Eos) has been widely applied in a variety of products, including foods, beverages, cosmetics, and medicines, because of their flavour, fragrance, and specific properties (Liu, Chen, Liu, Zhou & Wang, 2012; Settanni et al., 2012; Torres-Alvares et al., 2017). Furthermore, Citrus EOs are used as natural preservatives due to their broad spectrum of biological activities, including antimicrobial and antioxidant effects (Mitropoulou et al., 2017; Dosoky & Setzer, 2018). Orange peel contains essential oil with various chemical compounds, such as terpene, sesquiterpene, aldehyde, ester, and sterol. Orange peel has different chemical compounds depending on varieties that result in different scents. Generally, the essential oil made from orange peels contains the following chemical compounds: 94% of limonene, 2% of missen, 0.5% of octanal, 0.5% of linalool, 0.4% of decanal, 0.1% of neral, 0.1% of citronellal, 0.1% of geranial, 0.05% of valencene, 0.02% of ß-financial and 0.01% of α-sinensial (Kartika Fitri & Proborini, 2018).

However, the dominant chemical compound is limonene (C10H16). The limonene content varies in each orange variety, ranging between 70-92% (Hidayati, 2012). This limonene compound in the orange peels makes the essential oil expensive due to its typical scent (Megawati & Kurniawan, 2015). Limonene is commonly used in cosmetics and is added to cleaning (soap) products to give them an orange scent. Besides, limonene is also known as a biofuel since it is easily burned (Hidayati, 2012). This research aims at isolating and identifying the chemical components and the quality of essential oil made from Kisar sweet orange peels.

Materials and Methods

Research Materials and Instruments

The primary materials used in this research were Kisar sweet oranges from the Southwest Maluku Regency. The Kisar sweet orange peel type used in this research was a big and thick one. The other materials include clean water, sodium sulfate anhydrate 2-5%, distilled water, alcohol 95%, KOH solution, HCl solution, and PP indicator.This research used a set of steam distillation instruments consisting of a boiler containing pieces of Kisar sweet orange peel samples installed with a thermometer, condenser, gas stove, distillate storage bottle, aerator to pump water to the cooling pipe; digital scale; beaker glass; Erlenmeyer; measuring cup; volumetric flask, pipette; glass funnel, condenser, pycnometer, Ivymen System Abbe Refractometer, and GC MS. Research Implementation.

Preliminary Treatment

Kisar sweet orange peels were sorted before being sliced into 0.3-0.5 cm thick slices. Next, the orange peel slices were dried in a cabinet dryer for two days. During the drying process, the orange peel pieces were turned over every two hours.

Isolation of Essential Oil made from Kisar Sweet Orange Peels

Dried Kisar sweet orange peel slices were put in the boiler and distilled using the steam distillation method. The distillation temperature was 95 °C, and the distillation time was 6 hours. The essential oil obtained from Kisar sweet orange peels was collected in a clean storage bottle. Water and oil were then separated using a reparatory funnel.

Identification of Essential Oil made from Kisar Sweet Orange Peels.

The identification of the oil component was conducted using GC-MS with the following conditions: Rtxi-5MS column, length of 30 meters, ID of 0.25 mm, film of 0.25 mm, Helium lifting gas, ionization of El 70 EV, injection temperature of 300 °C, split injection mode, pressure of 13.0 kPa, total current of 79.3 Ml/min, current column of 0.55 Ml/min, linear speed of 26.8 cm/sec cleaning current of 3.0 Ml/min., Mass spectra were made based on each peak of the chromatography results and then compared with the spectra of the NIST data bank.

Determining the Physicochemical Characteristics of Essential Oil made from Kisar Sweet Orange Peels.

The criteria used in determining the quality and purity of essential oil include: color, scent, specific gravity, refractive index, acid number, ester number, and solubility in alcohol.

Results and Discussion

Preliminary treatment of Kisar Sweet Orange Peels

Some preliminary treatments for the raw materials used are critical to increasing the production and quality of essential oil.According to Nugraheni, Khasanah, Utami, & Ananditho, (2016), less precise preliminary treatments for the raw materials before distillation may result in a big loss of essential oil and decrease the quality. Those preliminary treatments include reduction of material size, drying, withering, incubation, and fermentation of microorganisms. The preliminary treatments used in producing the essential oil made from Kisar sweet orange peels were peel size reduction and the drying process. The fresh Kisar sweet orange peels (Fig 1) were cut to a smaller size of about 0.3-0.5 cm before drying (Fig 2).

Figure 1. Fresh Kisar sweet orange peels

Figure 2. Kisar sweet orange peels pieces

The size reduction was intended to widen the Kisar sweet orange peels' surface areas so that more oil glands were open (Khasanah, Kawiji, & Aji, 2015). The essential oil contained in plants is surrounded by oil glands, vessels, or glandular hairs. The oil may only be extracted if the water evaporates through the plants' tissues and is pushed to the surface through a hydro-diffusion process. If the materials remain intact, the process will run very slowly. As a result of the importance of thickness reduction in the diffusion process, the essential oil's

evaporation rate may be increased during the distillation process. (Wirasutisna, Sukrasno, Nawawi, & Marliani, 2016).

The sliced Kisar sweet orange peels were then dried in the cabinet dryer for two days. The drying process was designed to evaporate water from the materials, allowing the distillation process to run more smoothly and quickly (Sophia Grace Sipahelut, Kastanja, & Patty, 2020). The water evaporating from the materials facilitated the oil cells to break so that the oil might be taken easily during the distillation. Through the drying process, the water content in the Kisar sweet orange peel slices decreased from 77.43% to 53.31%. The dried Kisar sweet orange peel slices were then put into the boiler to be distilled (Fig. 3).

Figure 3. Dried Kisar sweet orange peel slicesare ready to be distilled

Figure 4. Distillation process of Kisar sweet orange peel

Essential Oil made from Kisar Sweet Orange Peels

The essential oil extracted from the dried Kisar sweet orange peel slices used the steam distillation method (Fig. 4). During the distillation process, a hydro-diffusion process occurred where the water evaporated through the plant cells' tissues, causing the plant cell walls to break and then the contained oil was pushed out (Musta & Nurliana, 2019).

Orange peels have oil glands between the flavedo and albedo that are easily pushed by the water vapor after the lysis of orange peels' cell walls as a result of contact between the water vapor produced by the heating process within the distillation instruments and the previously prepared orange peels. Water vapor will bring the oil particles contained in the orange peels to evaporate following the water vapor. The visual observation showed that the essential oil made from Kisar sweet orange peels was colorless (Fig. 5), and had a typical scent of orange oil that resulted from the distillation process with the yield of 0.53 %.

Figure 5. Essential oil made from Kisar sweet orange peels

The chemical components contained in the essential oil made from Kisar sweet orange peels were eventually revealed through an analysis using a Gas Chromatography-Mass Spectrometry method (GC-MS). The GC-MS principle involvels using gas chromatography to separate the components contained in the compounds, so that the mass spectrum of each component can be made with a higher accuracy. The separation using gas chromatography resulted in a chromatogram, while the examination resulted from the mass spectrometry of each compound was called a spectrum (Sophia Grace Sipahelut, Patty, Patty, Kastanja, & Lekahena, 2019). The results of GC-MS were shown in the form of a peak in the GC-MS spectrum (Fig. 6).

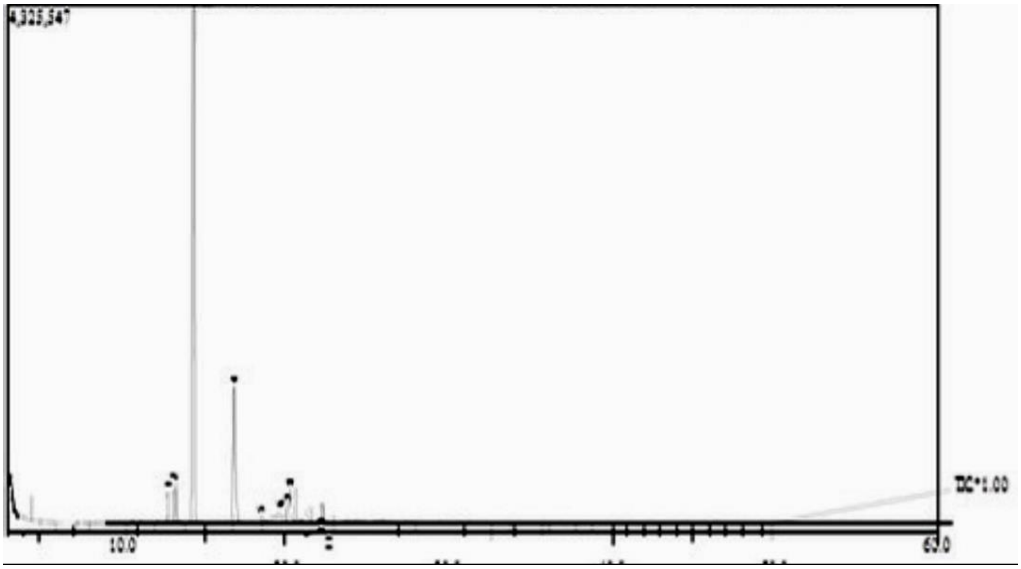

Figure 6. GC-MS Spectrum of Essential Oil made from Kisar Sweet Orange Peels

Eleven chemical components detected in the essential oil made from Kisar sweet orange peels included two major components of limonene (70.80%) and linalool (17.32%). The chemical components of essential oil made from Kisar sweet orange peels are shown in Table 1.

Table 1. Percentage of Components contained in the Essential Oil made from Kisar Sweet Orange Peels

Peak Number	Name of Component	Retention Time	Amount (%)
1	B-Myrcene	12.814	1.90
2	Octanal	13.250	2.61
3	Limonene	14.233	70.80
4	Linalool	16.792	17.32
5	Citronellal	18.542	0.34
6	Terpinene-4-ol	19.592	0.45
7	α-Terpineol	20.117	2.14
8	Decanal	20.300	2.18
9	2.6-Octadienal	21.308	1.05
10	Citral	22.217	1.23
11	1-cyclohexene-1carboxaldehyde	22.567	0.47

Source: Primary data, 2010

The most dominant component contained in the essential oil made from Kisar sweet orange peels is limonene. It is also known as limonene oil. The limonene contained in this essential oil is the major cyclic monoterpene compound. Limonene is a colored liquid at room temperature with a strong scent of orange (Hidayati, 2012). Limonene, as a major cyclic monoterpene compound, has an important role in the characteristics of this essential oil. The content of limonene has a significant impact on the scent of essential oils.. Major cyclic monoterpene compound has a simple ring and is more stable with a stronger scent when compared to the acyclic terpene component. Limonene is a major cyclic monoterpene compound (Cahyati, Kurniasih, & Khery, 2016).

The essential oil resulting from the Citrus genus plants could potentially become a natural insecticide as a mosquito repellent. The chemical compounds produced by these plants have an important role in stopping the spread of diseases caused by mosquitos. Orange peels oil may also be used as an air freshener, a perfume material, and a food flavoring agent. Besides, orange peel oil is also beneficial for health purposes, including for aromatherapy. The scent of orange peel oil may stabilize the nervous system, create happiness and peace as well as increase the appetite and cure diseases (Cahyati et al., 2016). These advantages are due to the components found in orange peel oil, such as imonene, which improves blood circulation, relieves sore throats and coughing, and inhibits the growth of cancer cells. Linalool, on the other hand, has sedative properties (Cahyati et al., 2016).

Physicochemical Characteristics of Essential Oil made from Kisar Sweet Orange Peels

Specific Gravity

The specific gravity resulting from the essential oil made from Kisar sweet orange peels was 0.8457 g/ml. Specific gravity is one important criterion in determining the purity of

essential oil. According to Nugraheni *et al.*, (2016), “the specific gravity of essential oil at the temperature of 25 °C generally ranges from 0.696-1.188 g/ml”. The value of specific gravity is influenced by the chemical components contained in an essential oil. Specific gravity also indicates the ratio between the mass fraction and the mole fraction contained in the essential oil. The higher the mass fraction, the higher the specific gravity will be(Slamet, Ulyarti, & Rahmi, 2019). Besides, specific gravity is also frequently associated with the oxidation process occurring in essential oils. Generally, the higher the value of specific gravity is possibly caused by the oxidation process. The drying process may result in oil oxidation where a new molecule is formed and results in a higher value of specific gravity.

Refractive Index

The refractive index is defined as the ratio of the speed of light in air to the speed of light in a substance at a given temperature. (Sophia G Sipahelut & Telussa, 2011). The refractive index resulting from the essential oil made from Kisar sweet orange peels was 1.4651. index of essential oil is closely related to the components contained in the essential oil. The more components with longer chains, such as sesquiterpene or oxygen-cluster components extracted, the greater the density of essential oil medium, resulting in essential oil with a higher refractive index (Harimurti, Soerawidjaja, H, Djajeng, & Risfaheri, 2012).

Acid Number

The acid number of essential oil made from Kisar sweet orange peels was 1.92. The acid number shows the level of free fatty acid in the essential oil. The greater the acid number, the greater its influence on the essential oil quality. Acid compounds in the essential oil will influence the oil’s typical scent since they quickly react with the air. The smaller the acid number, the better the actual oil acid number of less than five c (Daryono, Pursitta, & Isnaini, 2014). The contained in the essential oil made from Kisar sweet orange peels has met the Indonesia National Standard. The smaller the acid number contained in the essential oil, the better the essential oil quality. Acid is not commonly found in essential oils because it is easily changed by the oxidation reaction in the air, which can result in scent changes (Sophia Grace Sipahelut, 2012).

Ester Number

The ester number of essential oils made from Kisar sweet orange peels was 8.05. Ester number is one component determining essential oil quality regarding its scent (Hidayati & Khaerunisa, 2018). Esters are always present in essential oils in varying concentrations. The ester number indicates that the related oil has a good scent. According to Sipahelut (2012),

the oil's ester number determines the quality of the essential oil regarding its scent. Ester is one valuable component contained in an essential oil due to its preferable scent. Esters always exist in most essential oils at different concentrations (Ma'mun, 2006; Rialita *et al.*, 2015). According to the Indonesian National Standard (SNI), the maximum ester number is 10.

Solubility in Alcohol

The solubility test in alcohol describes whether the oil is soluble or not. The higher the oil's polar compound content, the easier it is to dissolve in alcohol solubility alcohol, which is critical in examining essential oil quality. The essential oil's solubility in alcohol is determined by the chemical types contained in the essential oil. The higher the content of terpene, the lower the solubility since the un-oxygenated terpene is considered a non-polar compound with no functional clusters (Sophia G Sipahelut & Telussa, 2011). The solubility ratio of essential oil made from Kisar sweet orange peels was 1:9, indicating that the resulting essential oil only had a small amount of oxygenated hydrocarbon compound. The oxygenated hydrocarbon compound is more accessible to dissolve in alcohol than the un-oxygenated hydrocarbon component. Like terpene, the oxygenated hydrocarbon compound is relatively more polar than the terpene cluster.

Conclusion

The isolation of essential oil made from Kisar sweet orange peels dried in a cabinet dryer using a steam distillation method resulted in a non-pale yellow-coloured, a typical orange oil smell, with a yield of 0.53%, the specific gravity of 0.8457, the refractive index of 1.4651 (20oC), an acid number of 1.92, ester number of 8.05, and solubility in alcohol of 1:9. The identification of essential oil made from the Kisar sweet orange peels conducted using GC-MS shows 11 chemical components with two major components consisting of limonene and linalool compounds. These results indicate that the essential oil made from Kisar sweet orange peels has potential as an antioxidant and an antibacterial agent.

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