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Main Components of The Semboro Variety of Siam Orange Peel Essential Oil Prepared Using Water Distillation Method

Budi Hariono [1,✉], Risse Entikaria Rachmanita [2]

[1] Study Program of Food Engineering Technology, State Polytechnic of Jember, INDONESIA.

[2] Study Program of Renewable Energy, State Polytechnic of Jember, INDONESIA.

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Corresponding Author:
✉ budi_hariono@polije.ac.id.
(Budi Hariono)

ABSTRACT

The maximum production potential for low quality oranges is 15% or 9,057 tons/year, the potential for orange peel waste is 50% of the total weight of oranges or the equivalent of 4,500 tons of orange peel waste The average yield of orange peel essential oil is 0.6%, resulting in a potential of 27 tons/year. The aim of this research is to obtain information regarding the best heating temperature in the distillation process and to identify the main components of Siam orange peel essential oil of the Semboro variety. The research was carried out using a laboratory-scale water distillation system, with a distillation flask capacity of 1 liter, with 150 g of orange peel as raw material at a distillation process heating temperature of 250°C and 300°C. The yields of 1.9 mL and 0.9 mL of Siam orange peel essential oil were obtained 1.083% and 0.513% respectively. The specific gravity of Siam orange peel essential oil is 0.855 g/cm^3 and 0.886 g/cm^3 respectively, the main components are 29 and 18 components respectively. An important finding of this research is that at a distillation process with heating temperature of 250°C, yields a limonene content of 16.10%, whereas heating at 300°C results in 7.69% limonene content

1. INTRODUCTION

Jember Regency is known as the center of the Semboro Siam Oranges. The Siam orange plantation area is 3,500 ha out of 5,600 ha of the total area of orange peel plants with production reaching 65,145 tons. The average productivity of Semboro orange farmers is 172.93 quintals per hectare. The quality of oranges based on grain diameter is divided into 4 grades, namely: grade A (super) diameter > 73 mm, grade AB diameter 63 – 73 mm, grade C diameter 56 – 62 mm and grade D diameter 50 – 55 mm (Setiawan & Suhendra, 2014). For grade D, is known as low grade quality oranges. The maximum production potential of low grade oranges is 15% or 9,057 tons/year. In general, low grade oranges have low prices, so to increase the selling value, it is necessary to diversify into processed orange juice. The potential for orange peel waste is 50% of the total weight of oranges or the equivalent of 4,500 tons of orange peel waste (Singh *et al.*, 2020). Given the distillation provide essential oil of average 0.6%, then the potential of essential is 27 tons/year (Muhtadin *et al.*, 2013). This can potentially improve the welfare of orange peel farmers, in particular.

The yield of orange peel oil generally depends on the variety (Potter & Richard H, 1991). The yield of orange peel oil from the sweet orange variety (Siam) 0.28%; lime 0.1%; lemon 0.4%; mandarin orange 0.5%; grapefruit 0.4% and bergamot 0.5%. Some of the identification results of sweet orange (Siam) oil from previous studies have been published. Poore in Guenther (1972) reported the identification of Valencia and Washington Navel sweet orange peel essential oils showed the following results: (1) d-Limonene, which is the main constituent, is present in the oil with a content of around 90% limonene; (2) decyl aldehyde; (3) Citral; (4) Octyl Alcohol; (5) Olefinic Alcohol; (6) Linalool; (7) Formic acid; (8) Acetic acid; (9) Caprylic acid and capric acid and (10) White amorphous component.

Guenther (1972) recorded numerous essential oils extracted from peels of different orange types. For example, the main components of essential oil extracted from Florida sweet orange peel are as follows: (1) d-Limonene of 90.4%; (2)n-Desyl Aldehyde; (3) Nonil Alcohol; (4) Linaloool. Meanwhile, the the important components of essential oil from Guinea sweet orange peel were identified as follows: (1) terpene content of 95-96%; (2) Miresn; (c) d-Limonene; (3) Terpinolene; (4) Aliphatic Terpenes; (5) Aldehydes; (6) n-Nonyl Alcohol; (7) Nerol; (8) d-Linalool; (8) d-α-Terpineol; (9) Farnesol; (10) Methyl anthranilate; (11) Sesquiterpenes; (12) Phenol; (13) Residues (capric acid, palmitic, serotate in the form of esters; (14) Phytosterols; (15) Ceryl Alcohols; (16) Carotenes; (17) xanthophyllic components and (18) yellow colored substances. The components of essential oil from Italian sweet orange peel are: (1) d-Limnen by 80%; (2) Terpinolene; (3) -Terpinen; (4) n-Desyl Aldehyde; (5) Citral; (6) d-Linalool; (7) n-Nonil Alcohol; (8) d-Terpineol; (9) n-Carylic Acid; (10) Methyl anthranilate; (11) Aurapten. Switaning, *et al.* (2010) reported that in general orange peel contains terpenes, sesquiterpenes, aldehydes, esters and sterols. Tarwiyah (2001) reported the content of orange peel essential oil with details of limonene (94%), mirsen (2%), linalool (0.05%, octanal (0.5%), decanal (0.4%), citronellal (0.1%), neral (0.1%), geranial (0.1%), valence (0.05%); sinnsial (0, 02%) and sinensial (0.01%).

The purpose of the study was to determine the chemical components of the essential oil of Siam Semboro sweet orange peel produced through a distillation process at a certain heating temperature (250 °C and 300 °C) and to determine the yield of the essential oil obtained. Research related to the yield and identification of the constituent components of Siam orange peel essential oil, Semboro variety, Jember Regency has not been done much. Therefore, this research is important as initial data for potential investors to find out the potential of essential oil extracted from Siam Semboro orange peel, especially by using water distillation method.

2. METHODS

2.1. Materials and tools

The material used in this study was the fresh Siam Semboro sweet orange peel obtained from traditional market "Pasar Tanjung" in Jember. The equipment is a set of steam distillation apparatus, separating funnel, erlenmeyer, analytical balance, measuring cup, bottle containing essential oil, aluminum foil, filter paper disc, petri dish, micro pipette, electric heater, rotary evaporator, data logger, k term thermocouple and a set of Gas Chromatography-Mass Spectroscopy analyzer (GC-MS QP2010 Plus, Shimadzu).

2.2. Sample setup

Fresh orange peel was cut into 1 cm size as much as 150 grams and put into a distillation flask with a volume of 1000 mL. The set up of the distillation apparatus can be seen in Figure 1. T_1 is condenser inlet temperature, T_2 is condenser outlet temperature, T_3 is boiling flask temperature and T_4 is distillate temperature.

2.3. Essential oil distillation by steam distillation

A total of 300 g of fresh orange peel that has been cut into 1 cm size was distilled twice with a heating temperature of 250 °C and 300 °C. Steam distillation equipment includes stove, 1000 mL volume distillation flask, condenser, distillate container and separator funnel. Temperature measurements were carried out using a data logger, where the probe was placed in a distillation flask, inlet and outlet channels from the condenser, and in the distillate storage flask, then heated at a temperature of 250 °C and 300 °C. The resulting of distillate was separated using a separating funnel. The analysis was conducted descriptively.

2.4. Identification of essential oils

As much as 1 drop of essential oil is dropped on a piece of filter paper and allowed to stand for a few minutes. After a few minutes, the essential oil will evaporate completely without leaving a transparent stain (Guenther, 1972).

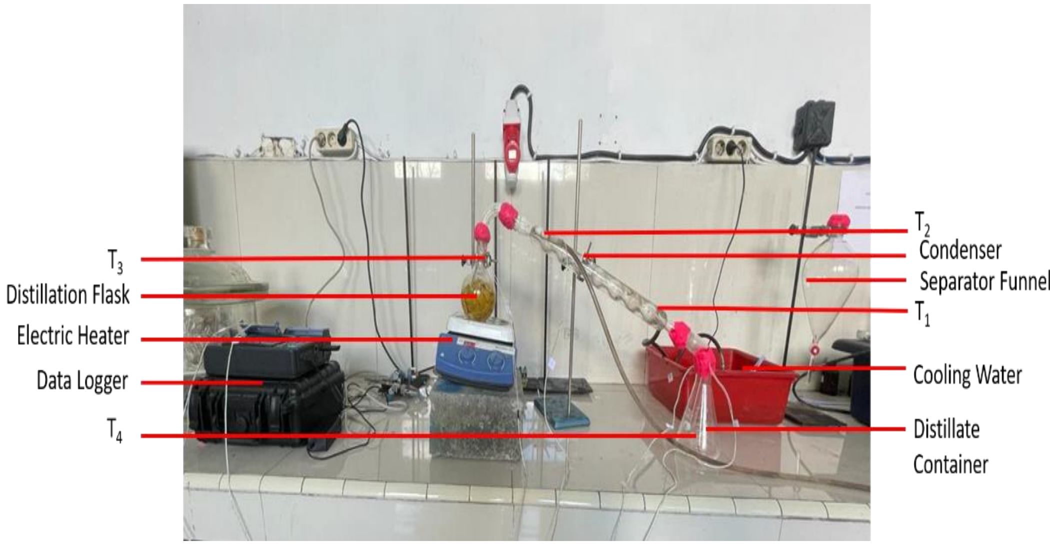

Figure 1. The set up of the distillation apparatus

2.5. GC-MS Analysis

The essential oil of Siam Semboro orange peel obtained was then analyzed using gas chromatography-mass spectroscopy (GC-MS) with column oven temperature was 80 °C, injection temperature of 250 °C with pressure of 64.1 kPa, and column flow of 0.99 mL/min. This analysis is to determine the components of the chemical groups that make up essential oils and the mass spectrum obtained is compared with the mass spectrum of the comparison compounds known in the database that has been programmed on the GC-MS tool.

3. RESULTS AND DISCUSSION

3.1. Distillation process temperature

The distillation process was carried out using a steam system employing a laboratory-scale distillation apparatus with 150 g of orange peel. The temperature distribution during the distillation process with heating temperatures of 250 °C and 300 °C, is shown in Figures 2. The distillation process with heating temperature of 250 °C starting to evaporate

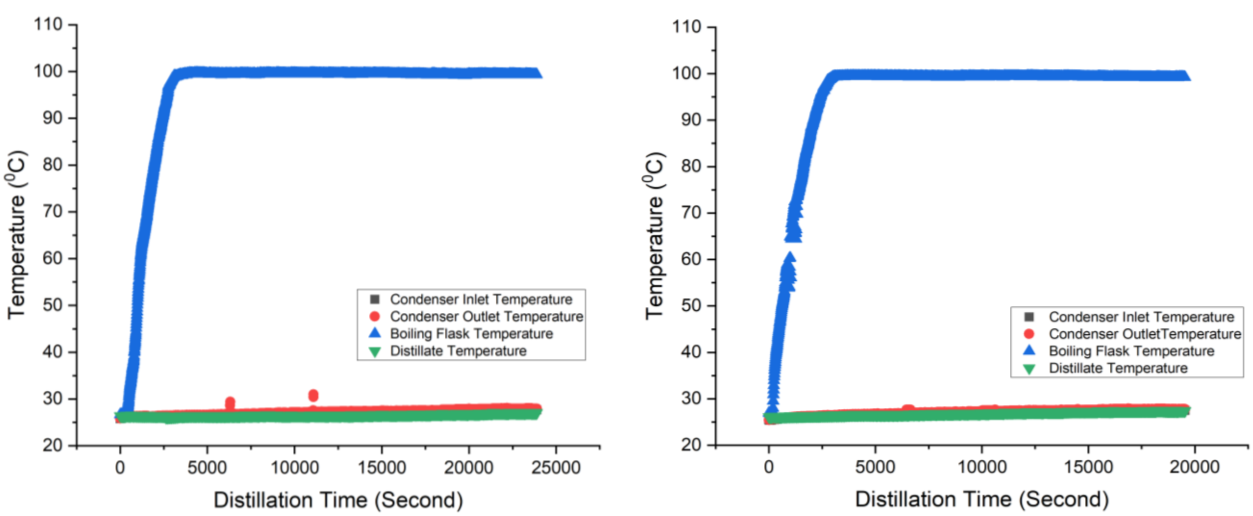

Figure 2. Temperature distribution during distillation with heating temperature at 250°C and 300°C

the liquid into the gas phase at 3,090 s or 51.5 min. It means that the liquid needs 51.5 min to reach its boiling point (100 °C). The distillation process was stopped when the distillation was reached at 23,890 s or at 6 h and 38 min or the extraction process took 5 h and 50 min. Figure 3 shows that the heating temperature at 300 °C changes the liquid phase to gas at 48.5 min. The distillation process was stopped after 5 h and 25 min or the extraction process for 4 h and 37 min. The higher the heating temperature, the faster the heat transfer and mass transfer that occur between the media and the material. Thus, the faster the material reaches its boiling point for the evaporation process (Hien *et al*., 2022). Several research results related to extraction time were reported by Haryanti *et al*., (2014), reporting the process of extracting orange fern oil in 3 h; Ikarini *et al*., (2021) reported that the distillation of Siamese oranges was carried out for 2 h; Febrina & Sigiro (2021) reported that the distillation of Siam Sambas oranges was carried out for 7 h; Fachriyah *et al*., (2002), reported distillation of Siamese orange peel essential oil for 10 h; Wibaldus *et al*., (2016) reported the distillation of lime for 4 h. The distillation time varies greatly also depending on the raw material, the thicker the orange peel, the longer the distillation time is needed. General, the Semboro Siamese orange peel with an average thickness of 1 mm.

3.2. Essential Oil Extraction

As many as 150 g of fresh Siam Semboro orange peel, cut into approximately 1 cm pieces, underwent steam distillation. Following separation with a separatory funnel, yields of 1.9 mL and 0.9 mL of yellow volatile oil were obtained, corresponding to 1.083% and 0.513% yields, with specific gravities of 0.855 g/cm^3 and 0.886 g/cm^3, respectively. The extraction process at high temperatures causes heat transfer and mass transfer that occurs between the environment and the material to take place faster so that the extraction process takes place more effectively. However, if the temperature is too high, there are components in the material that are degraded, thus reducing the quality of the resulting extract (Teles *et al*., 2017). This is in line with research conducted by Hien *et al*.,(2022) on orange essential oil obtained at an extraction temperature of 130 °C resulting in a yield of 1.7 mL/g, but at a temperature of 150 °C the yield of essential oil decreased to 1.63 mL/g.

3.3. Identification of Siam Semboro orange peel essential oil

Identification is done as an effort to ensure that the oil produced is an essential oil. The identification results showed that there were no transparent stains on the filter paper, this proves that the oil obtained is an essential oil. Further analysis was performed using Gas Chromatography and Mass Spectroscopy (GC-MS). The results of the GC-MS analysis obtained two data, namely chromatograms from gas chromatography (GC) analysis and mass spectra from mass spectroscopy (MS) analysis. The essential oil chromatogram of orange peel oil is shown in Figure 4 for heating at 250 °C. The chromatograms shows 29 peaks of compounds. The suspected volatile compounds in the essential oil of Semboro orange peel chromatogram are shown in Table 1.

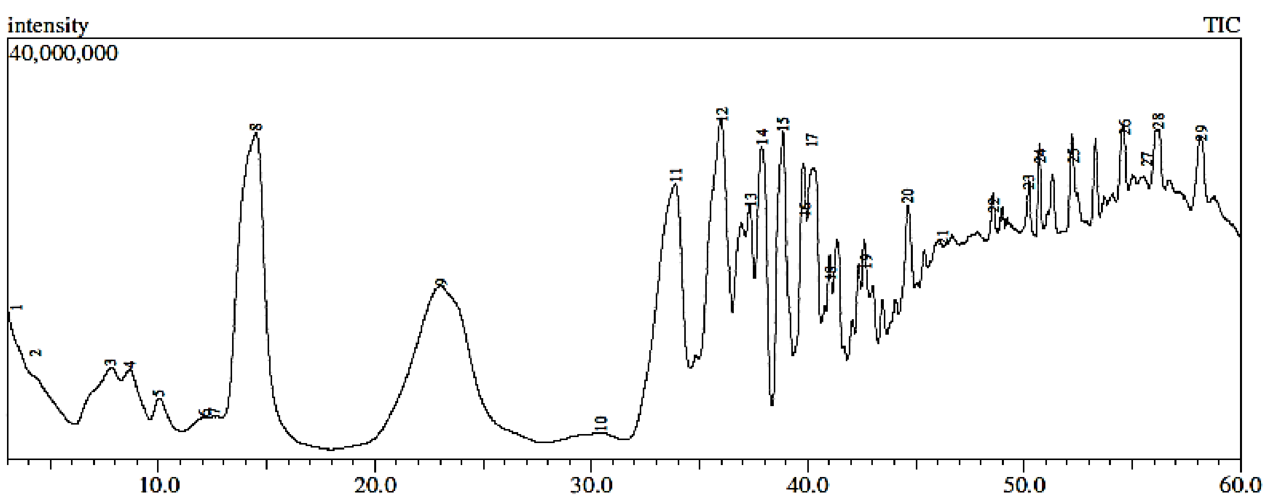

Figure 4. Chromatogram of Semboro orange peel essential oil extracted using water distillation at 250 °C

Table 1. Alleged essential compounds in the chromatogram of essential oils of Siam Semboro orange peel based on database

Peak of compound	Retention time (minutes)	Area	Peak (% Area)	Suspected Compound
1	3.04	400300096	1.79	Linalool
2	4.30	189509576	0.85	Limonene oxide
3	7.79	317472955	1.42	Trans-Para-2,8-Menthadien-1-Ol
4	8.69	294849977	1.32	Citronella
5	10.02	121145235	0.54	beta.- Myrcene
6	12.12	65870480	0.29	Trans-Para-2,8-Menthadien-1-Ol
7	12.57	74893839	0.33	Alpha.-D2-3-Phenylpropylnitrite
8	14.53	2475355384	11.05	dl-Limonene
9	23.09	2904651398	12.96	3-Cyclohexen-1-ol, 4-methyl-1-(1-methylethyl)- (CAS) 4-Terpineol
10	30.44	88601021	0.40	Bicyclo[3.3.0]Oct-2-En-7-One, 6-Methyl-
11	33.90	1938019997	8.65	3-Cyclohexene-1-methanol, alpha, alpha.,4-trimethyl-, (S)- (CAS) p-Menth-1-en-8-ol
12	36.04	1754313871	7.83	2,6-Octadien-1-Ol, 3,7-Dimethyl- (CAS) 3,7-Dimethyl 2,6-Octadiene-1-Ol
13	37.37	1662763295	7.42	2-Cyclohexen-1-one, 2-methyl-5-(1-methylethenyl)- (CAS) 2-Methyl-5-isopropenyl-2-cyclohexenone
14	37.89	446770569	1.99	Z-Citral
15	38.89	761544337	3.40	Methyl benzoate
16	39.93	1130932369	5.05	dl-Limonene
17	40.22	225456821	1.01	Beta Elemene
18	41.10	479964480	2.14	Bicyclo[4.1.0]heptane, 7-butyl- (CAS) Norcarane, 7-butyl
19	42.71	521959280 2	2,33	Z)6,(Z)9-Pentadecadien-1-ol
20	44.64	127262460	0,57	1,5,9-Cyclotetradecatriene, 1,5,9-trimethyl-12-(1-methylethenyl
21	46.23	1160311332	5.18	Epizonaren
22	48.64	1646535683	7.35	Hexadecanoic acid, methyl ester (CAS) Methyl palmitate
23	50.27	297441388	1.33	Dodecanamide, N,N-bis(2-hydroxyethyl)
24	50.77	540115582	2.41	13,16-Octadecadienoic acid, methyl ester (CAS) Methyl
25	52.31	550984291	2.46	Oxirane, tetradecyl
26	54.65	1050730581	4.69	Heptadecane (CAS) n-Heptadecane
27	55.73	198525347	0.89	1b,4a-Epoxy-2H-cyclopenta [3,4]cyclopropa [8,9]cycloundec [1,2-b]oxiren-5(6H)-one, 7-(acetyloxy
28	56.24	643637870	2.87	Dodecane, 2,7,10-trimethyl- (CAS)
29	58.24	337061324	1.50	Heptadecane (CAS) n-Heptadecane

The identification results of Siam Semboro orange peel essential oil with heating at 250°C produced 29 components with 10 main components (area >2.50%), including: (1) 3-Cyclohexen-1-ol, 4-methyl-1-(1-methylethyl)- (CAS) 4-Terpineol; (2) dl-Limonene; (3) Hexadecanoic acid, methyl ester (CAS) Methyl palmitate; (4) 3-Cyclohexene-1-methanol, alpha, alpha.,4-trimethyl-, (S)- (CAS) p-Menth-1-en-8-ol; (5) 2,6-Octadiene-1-Ol, 3,7-Dimethyl- (CAS) 3,7-Dimethyl 2,6-Octadiene-1-Ol; (6) 2-Cyclohexen-1-one, 2-methyl-5-(1-methylethenyl)- (CAS) 2-Methyl-5-isopropenyl-2-cyclohexenone; (7) Epizonaren; (8) Heptadecane (CAS) n-Heptadecane; (9) Methyl benzoate; (10) Dodecane, 2,7,10-trimethyl- (CAS.

The results of GC MS from oil prepared with heating at 300 °C and extraction time of 4 h 37 min obtained chromatograms from analysis by gas chromatography showing 19 peaks of compounds. The essential oil chromatogram of orange peel oil is shown in Figure 5. The suspected volatile compounds in Siam Semboro Orange Peel Essential Oil Chromatogram are shown in Table 2.

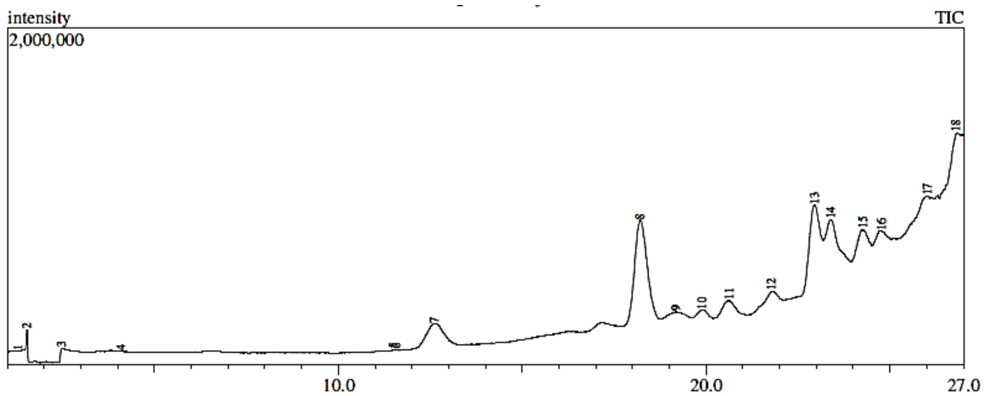

Figure 5. Chromatogram of Semboro Orange Peel Essential Oil on heating at 300°C

Table 2. Alleged essential compounds in the chromatogram of essential oils of Siam Semboro orange peel based on database

Peak of compound	Retention time (minutes)	Area	Peak (% Area)	Suspected Compound
1	1.29	344720	0.61	Propane, 2,2'-oxybis[1-chloro- (CAS) Bis-(2-Chloro
2	1.53	491540	0.87	2-Octanamine (CAS) 2-Octylamine
3	2.49	131084	0.23	Carveol, dihydro-, cis-
4	4.08	31147	0.06	4-Methyl-4-(2',3'-Dimethyl-Cyclopent-2'-En-1'-Yl)-Pentanal
5	11.52	20720	0.04	-(2-Chloroethyl)Aziridine
6	11.57	12393	0.02	2,3-Dimethyl-Aziridine
7	12.62	4346970	7.69	dl-Limonene
8	18.22	15224347	26.93	Linalool
9	19.20	2049669	3.63	3-Cyclohexene-1-methanol, .alpha.,.alpha.,4-trimethyl- (CAS) Cyclohexene
10	19.89	1120304	1.98	p-mentha-E-2,8(9)-dien-1-ol
11	20.63	2397134	4.24	Z-Citral
12	21.79	2027415	3.59	3-Cyclohexen-1-ol, 4-methyl-1-(1-methylethyl)- (CAS) 4-Terpineol
13	22.96	10126099	17.91	3-Cyclohexene-1-methanol, .alpha.,.alpha.,4-trimethyl- (CAS) Cyclohexene, 1-Methyl-
14	23.41	8576631	15.17	2,6-Octadiene, 4,5-dimethyl- (CAS)
15	24.28	3501998	6.20	Trans-(+)-Carveol
16	24.76	1827410	3.23	Cis-(+)-Carveol
17	26.03	1866123	3.30	3,6-Nonadien-1-ol, (E,Z)- (CAS) Trans,Cis-3,6-Nonadien-1-Ol
18	26.83	2428517	4.30	Perilla alcohol

Treatment at 300 °C heating produced 18 components with the main components (% area above 2.5%) including: (1) Linalool; (2) 3-Cyclohexene-1-methanol; (3) 2,6-Octadiene, 4,5-dimethyl- (CAS); (4) dl-Limonen; (5) Trans-(+)-Carveol; (6) Z-Citral; (7) 3-Cyclohexene-1-methanol, .alpha.,.alpha.,4-trimethyl- (CAS) Cyclohexene; (8) 3-Cyclohexen-1-ol, 4-methyl-1-(1-methylethyl)- (CAS) 4-Terpineol; (9) Perilla alcohol; (10) 3,6-Nonadiene-1-ol, (E,Z)-

(CAS) Trans,Cis-3,6-Nonadiene-1-Ol. The heating process at high temperatures and prolonged extraction time causes oxidation and degradation reactions in the compounds contained in orange peel oil. This caused the number of compounds identified on GC-MS for the 300°C heating temperature to be lower (Sulaiman *et al*., 2017). This is in line with research conducted by Yusoff *et al*., (2013) which showed that at an evaporation temperature of 95 °C the number of compounds contained in kaffir lime oil was 19 compounds while at an evaporation temperature of 90 °C, 24 compounds were found. In steam distillation, the yield of chemical compounds in the orange peel oil is still higher than cold press-assisted aqueous extraction that was conducted by Labrath & Gaikar (2020). The research found that the total yield of orange peel oil was 0.79% which contained 7 types of chemical compounds including Tangeritin, Sinensetin, Nobiletin, Tocotrienol, Tocopherol acetate, Xanthophyll, and 3-hydroxyl α-carotene.

4. CONCLUSIONS

The 250 °C and 300 °C heating temperatures resulted in yields of 1.083% and 0.513%, respectively, the specific gravity at 18°C was 0.855 g/cm^3 and 0.886 g/cm^3, and the main components were 29 and 18 components, respectively. Treatment with heating at a temperature of 250 °C resulted in higher Siam orange peel essential oil yields, while the specific gravity value was smaller and the number of chemical compounds was greater than heating at a temperature of 300 °C. The 250 °C treatment was chosen to be used as a reference for further research related to the initial treatment of materials with High Pulsed Electric Field (HPEF) treatment and application to vacuum distillation.

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