Comparison The Use of n-hexane and Water Solvents in The Extraction of Semboro Orange Peel Powder Essential Oil using The Maceration Method

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ABSTRACT

Semboroe orange fruit production in Jember Regency in 2020 was first rank compared to the production of other fruit crops, which amounted to 2,221 tons. The weight of orange peel is about 25-30% so that the potential for orange peel is 555 tons to 666 tons of orange peel which can be extracted to produce essential oils. The method used is maceration so that it is easily adopted by the people of Semboro Village. The solvents used were water and n-hexane with the ratio of Semboro orange peel powder and solvents 1:10, 1:15 and 1:20. The test results show that only 1:10 ratio of essential oil can be produced with water as a solvent. The three compounds with the highest % area of essential oil by using GCMS testing are 3-Cyclohexen-1-ol, 4-methyl-1-(1-methylethyl)- (CAS) 4-Terpineol (12.96%), dl-Limonene (11.05%), 3-Cyclohexene-1-methanol, alpha, alpha.,4-trimethyl-, (S)- (CAS) p-Menth-1-en-8-ol (8.65%) belonging to the sesquiterpene group

Keywords: *Essential oil, maseration, n-hexane, Semboro orange peel, water.*

INTRODUCTION

Semboro orange fruit production in Jember Regency in 2020 was first rank compared to the production of other fruit crops, which amounted to 2,221 tons which were the result of citrus plantations in Semboro (1). Rahmanda et al. (2021) stated that about 25-30% is the weight of orange peel so that the potential for orange peel is 555 tons to 666 tons of orange peel (2). Ahmad et al. (2013) conducted a study with the condition of fresh orange peel oil, dried for 12 hours, and dried for 24 hours, obtained an average yield of 0.6%; 0.82%; 0.76% with pre-treatment oven obtained limonene content in fresh orange peel oil, dried for 12 hours, and dried for 24 hours obtained 93.39% respectively; 97.57%; 95.32% (3).

According to Julianto (2016), essential oils are a mixture of volatile compounds (4). The content of complex compounds belongs to essential oil. Through the process of breaking down plant tissue and opening sebaceous glands as much as possible, obtained essential oils from natural ingredients (5). According to Kurniawan (2015), essential oils are useful as raw materials for air freshener, perfume, changing the taste of food to make it more attractive, and can be used as aromatherapy (6). Orange aroma is one of the aromas that are widely used as raw material for air fresheners and perfumes. The essential oil contained in orange peel consists of several compounds such as terpenes, sesquiterpenes, esters, sterols, and aldehydes. Different citrus fruits also affect the content of different volatile oil compounds. However, each variety has one main compound, limonene ($C_{10}H_{16}$) which is found in orange peels (7).

According to Qorriaina et al. (2015) extraction is the activity of withdrawing soluble chemical content so that it is separated from insoluble materials by using a liquid solvent. The extraction process can generally be carried out by maceration, percolation and reflux (8). The advantages of the maceration method are that the equipment used is very simple, the working technique is relatively simple and easy to do, the operational costs are relatively low and can be used to extract thermolabile compounds because maceration is carried out without heating so that it can easily be applied by the people of Semboro Village.

Solvent is very influential in the process of essential oil extraction. The solvent used during the extraction process was n-hexane and water as a comparison. Hexane is an alkane hydrocarbon compound with the chemical formula C_6H_{14} which has stable and volatile properties so that the solvent is very good to use during the extraction process. Orange peel essential oil is non-polar so it is more soluble in non-polar solvents such as hexane (9).

Based on the description above, a research was conducted on extracting the essential oil of Semboro orange peel powder using n-hexane and water as solvent and maceration method.

MATERIAL AND METHODS

The study was conducted at the Energy Laboratory and Bioscience Laboratory in Politeknik Negeri Jember for 4 months from June to September 2022.

The material that will be used in this study is Semboro Siamese orange peel obtained from Semboro Village, Semboro District, Jember Regency. The solvents used were n-hexane and water. The tools used in this study included an oven, digital scale, 1000 ml pyrex extraction tube, connecting glass pipe, 50cm transfer condenser, separating funnel, thermometer, heating stove, dropper pipette, stirring rod, funnel, measuring cup, sieve, magnetic stirrer, glass bottle, measuring cup, aluminum foil, plastic wrap, plasticine, blender, stative, fine filter paper, GC-MS.

This research method is a type of quantitative experimental research, namely a research method that serves to determine the causal effect of the treatment on the results under controlled conditions. In this study, experimental variations were carried out with the ratio of material to solvent (1:10; 1:15; 1:20) so as to get the best ratio with a temperature of 55°C and an optimal time in the extraction process using the Maceration method. The characteristic parameters of essential oils tested were water content, yield and content of essential oils produced. Testing the characteristics of essential oils was repeated three times for each sample with the same composition. The design of this research can be seen in Table 1.

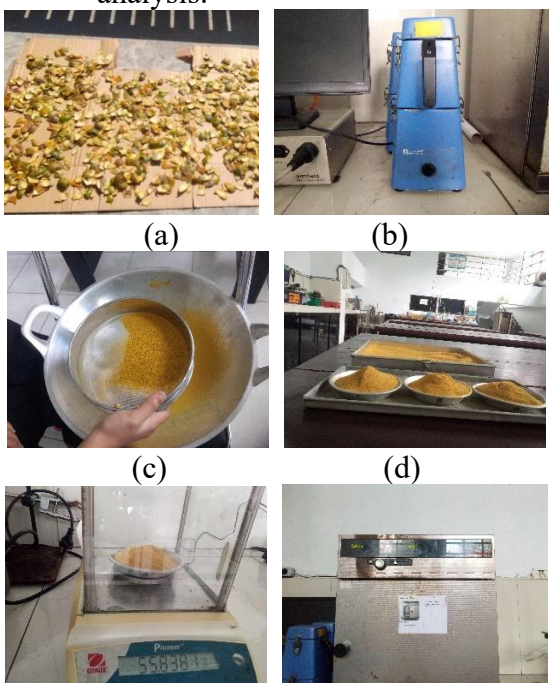
Table 1. Research design

Solvent	Material and solvent ratio		
	B1 (1:10)	B2(1:15)	B3(1:20)
n-hexane (A1)	A1B1	A1B2	A1B3
Water (A2)	A2B1	A2B2	A2B3

The stages of the research are described as follows:

1. **Making Semboro Orange Peel Powder**
   a. The waste of Semboro Siamese orange peel is obtained from the rest of the industrial products in Semboro Village, Semboro District, Jember Regency and is sorted between the flesh and the skin.
   b. Semboro orange peel is sorted.
   c. Then dried in the sun to dry, then weighed.
   d. The dry skin is then blended until smooth.

e. Dry in the oven at a temperature of 65°C for 5 hours.
f. The smooth skin was sieved using a 60 mesh sieve, then weighed again.
g. Determine the water content analysis.



(a) (b) (c) (d) (e) (f)

Figure 1. The process of preparing materials and testing the water content of materials

2. Maceration Extraction

a. The orange peel powder is put into a measuring flask as much as 30 grams, then the solvent is added according to the specified ratio then stirred using a magnetic stirrer for 15 minutes at a speed of 600 rpm.
b. Then the solution was allowed to stand for 15 minutes.
c. The solution is filtered through a sieve until all the liquid is used up and the dregs are discarded.
d. While the filtrate is distilled at 50°C for n hexane solvent and 80 °C for water solvent for 1 hour to separate the oil from the solvent. The distillate is the solvent, while the residue is the orange peel essential oil.

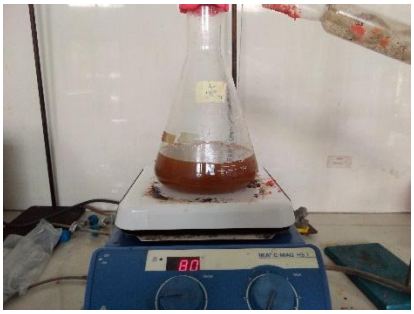


Figure 2. The extration process

RESULT

The results of testing the water content of the material obtained the following data. Mass of the cup: 9.8467 grams. The ingredients are put in a cup and in the oven for 5 hours at a temperature of 65°C. The following is a table of the results of testing the water content of the material.

Table 2. The results of testing the water content of the Semboro orange peel powder

Sample	Initial mass (m_1)	Mass after oven (m_2)	Water content
A	59.8467 gram	55.8381 gram	7.179%
B	59.8581 gram	55.7825 gram	7.306%
C	59.8577 gram	55.7639 gram	7.341 %
Average water content			7.275%

The ratio of Semboro orange peel powder and solvent is 1:10, the material is 30 grams and the solvent is 300 ml. The ratio of Semboro orange peel powder and solvent is 1:15, the material is 30 grams and the solvent is 450 ml. The ratio of Semboro orange peel powder and solvent is 1:20, the material is 30 grams and the solvent is 600 ml. The essential oil produced from the maceration extraction process can be seen in Table 3 and Figure 3.

Table 3. The essential oil produced from the maceration extraction process

Sample Variation	Essential Oil (ml)
A1B1	0
A1B2	0
A1B3	0
A2B1	0.5 ml
A2B2	0
A2B3	0

(a) (b)

Figure 3. (a) Yields (b) Essential Oil

The results of the GCMS test of essential oils produced from A2B1 are shown in Figure 4.

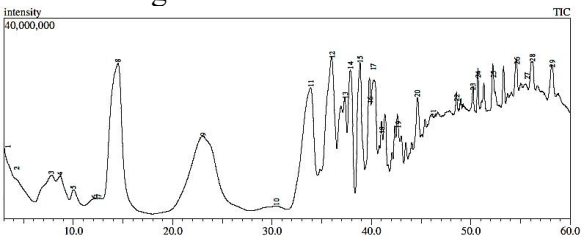


Figure 4. The results of the GCMS test of essential oils produced from A2B1

Table 4. Alleged Essential Compounds in the Chromatogram of Essential Oils of Semboro Orange Peel Powder Based on Database

Peak	R.time	Area	Area %	Name
1	3,042	400300096	1,79	Linalool
2	4,300	189509576	0,85	Limonene oxide
3	7,791	317472955	1,42	Trans-Para-2,8-Menthadien-1-Ol
4	8,686	294849977	1,32	Citronella
5	10,024	121145235	0,54	beta.-Myrcene
6	12,117	65870480	0,29	Trans-Para-2,8-Menthadien
7	12,564	74893839	0,33	-1-Ol Alpha.-D2-3-Phenylpropylnitrite
8	14,529	2475355384	11,05	dl-Limonene
9	23,090	2904651398	12.96	3-Cyclohexen-1-ol, 4-methyl-1-(1-methylethyl)- (CAS) 4-Terpineol
10	30,440	88601021	0,40	Bicyclo[3.3.0]Oct-2-En-7-One, 6-Methyl-
11	33,900	1938019997	8,65	3-Cyclohexene-1-methanol, alpha, alpha.,4-trimethyl-, (S)- (CAS) p-Menth-1-en-8-ol
12	36,044	1754313871	7,83	2,6-Octadien-1-Ol, 3,7-Dimethyl- (CAS) 3,7-Dimethyl 2,6-Octadiene-1-Ol
13	37,367	1662763295	7,42	2-Cyclohexen-1-one, 2-methyl-5-(1-methylethenyl)- (CAS) 2-Methyl-5-isopropenyl-2-cyclohexenone
14	37,889	446770569	1,99	Z-Citral
15	38,887	761544337	3,40	Methyl benzoate
16	39,930	1130932369	5,05	dl-Limonene

Peak	R.time	Area	Area %	Name
17	40,224	225456821	1,01	Beta Elemene
18	41,103	479964480	2,14	Bicyclo[4.1.0]heptane, 7-butyl- (CAS) Norcarane, 7-butyl
19	42,714	521959280 2	2,33	Z)6,(Z)9-Pentadecadien-1-ol
20	44,638	127262460	0,57	1,5,9-Cyclotetradecatriene, 1,5,9-trimethyl-12-(1-methylethenyl
21	46,233	1160311332	5,18	Epizonaren
22	48,643	1646535683	7,35	Hexadecanoic acid, methyl ester (CAS) Methyl palmitate
23	50,274	297441388	1,33	Dodecanamide, N,N-bis(2-hydroxyethyl)
24	50,770	540115582	2,41	13,16-Octadecadienoic acid, methyl ester (CAS)
25	52,306	550984291	2,46	Methyl Oxirane, tetradecyl
26	54,650	1050730581	4,69	Heptadecane (CAS) n-Heptadecane
27	55,733	198525347	0,89	1b,4a-Epoxy-2H-cyclopenta[3,4]cyclopropa [8,9]cycloundec [1,2-b]oxiren-5(6H)-one, 7-(acetyloxy
28	56,236	643637870	2,87	Dodecane, 2,7,10-trimethyl- (CAS)
29	58,239	337061324	1.50	Heptadecane (CAS) n-Heptadecane

DISCUSSION

Essential oils are only produced from the A2B1 variation, which has a ratio of Semboro orange peel powder material to 1:10 solvent with the solvent used is water and extraction temperature 80 °C. The yield of essential oils is more optimal using water as a solvent compared to n-hexane (10). The average water content of the Semboro orange peel powder is 7.275%.

Identification of the results of essential oils using the maceration method is carried out as an effort to ensure that the oil produced is essential oil. The identification results showed that there were no transparent stains on the filter paper, this proved that the oil obtained was essential oil. Further analysis was carried out 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 identification results of Semboro orange peel powder essential oil 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 three compounds with the highest % area are 3-Cyclohexen-1-ol, 4-methyl-1-(1-methylethyl)- (CAS) 4-Terpineol (12.96%), dl-Limonene (11.05%), 3-Cyclohexene-1-methanol, alpha, alpha.,4-trimethyl-, (S)- (CAS) p-Menth-1-en-8-ol (8.65%) belonging to the sesquiterpene group (10).

CONCLUSION

Essential oils are only produced from the A2B1 variation, which has a ratio of Semboro orange peel powder material to 1:10 water solvent. The yield of essential oils is more optimal using water as a solvent compared to n-hexane. The three compounds with the highest % area are 3-Cyclohexen-1-ol, 4-methyl-1-(1-methylethyl)- (CAS) 4-Terpineol (12.96%), dl-Limonene (11.05%), 3-Cyclohexene-1-methanol, alpha, alpha.,4-trimethyl-, (S)- (CAS) p-Menth-1-en-8-ol (8.65%) belonging to the sesquiterpene group.

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