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Essential Oil Extraction of *Cananga odorata* Flowers using Hydrodistillation and Steam-Water Distillation Processes

Ika Oktavianawati

Dept. Chemistry, Fac. Math & Science, University of Jember

*Corresponding author: ika.fmipa@unej.ac.id

Abstract. This paper discussed about the effect of distillation processes in the extraction of cananga essential oil from *Cananga odorata*. In this research, cananga oils were extracted by steam-water distillation and hydrodistillation processes. The equipment used in this current research is a distillation home scale set-up plant which further will be applied on cananga local farmer in Sukorambi region, Jember district. Therefore, preliminary optimization process for this distillation equipment was required. The distillation batch (retort) contained 2 kg of cananga flowers and 20 L of water. Based on research result, the distillation equipment could run hydrodistillation process for 3 hours, while in steam-water distillation set-up could only run for 2 hours. The volume of water in distillation batch was a limiting factor in both processes. In general, the yield of essential oil was increase along the longer of distillation time that have been applied. However, the result showed that essential oil from hydrodistillation for 1 hour had the best quality compared to other distillation conditions, in terms of its volatile compound contents. Oxygenated hydrocarbons content in cananga oil from 1 hour hydrodistillation process (56.5%) was highest compared to 2 and 3 hour hydrodistillation (35.48% and 22.36%, respectively), and even to essential oil from steam-water distillation process (43.25%). Generally, extra grade cananga oil was markedly by the high content of esters, and linalool, but low in β -caryophyllene content. These characteristics could be found in cananga oil obtained from hydrodistillation process for 1 hour which contained major volatile compounds, i.e. geranyl acetate (16.58%), benzyl benzoate (13.38%), β -caryophyllene (16.35%), germacrene D (15.3%), and farnesol (9.54%). However, linalool was only found in cananga oil from steam-water distillation. Therefore the current distillation condition, hydrodistillation for 1 hour, would be applied in distillation of cananga oil at Jember local community.

1. Introduction

Cananga is a kind of tree native to parts of Asia, including Indonesia. This plant is known for its two famous varieties, Java cananga (*Cananga odoratum forma macrophylla*) and ylang-ylang (*Cananga odoratum forma genuina*), besides another variant dwarf ylang-ylang, locally known as kenanga perdu (*Cananga odoratum forma fruticosa*). This cananga tree can be found in relatively huge amount at Karangpring village, Jember district. Previously, Karangpring village is wellknown for producing natural resource, rose. In fact, cananga flower is also to be another feasible local product that can be explored from this village.

Cananga oil is derived from flower part of cananga tree. This oil was produced from cananga flowers in a yield of 1.5% - 2.5% [1,2]. Cananga oil has some benefits such as aromatherapy, soul relaxation, lowering blood pressure tension, pharmaceutical, perfumery, cosmetics, anti aging,



relieving acne, hair conditioner, flavoring agent, fixative and soap additive [2,3,4,5]. Some research have analyzed chemical compounds of cananga essential oil correlated to its bioactivities[6,7,8]. Oxygenated hydrocarbons are targetted to be extracted from plant tissues in order to provide higher bioactivities[9,10]. Extraction methods to obtain essential oil influence quantity and quality of the oil. Favorite conventional method to extract essential oil is distillation, which are in the form of hydrodistillation, steam-water distillation, and steam distillation. In addition, some research also have focused on essential oil fractionation to offer valuable and qualified essential oil in a series of fractionated groups toward time of distillation or purification by partition on non polar solvents [3,11-15].

Businesses of essential production in Indonesia are still require an encouragement due to some constrains on its marketing section, including low quality of the oil products, domestic marketing, and unstable prices [16]. In 2003, price of cananga oil was around Rp 500.000,-/kg [1], but in a range of 2005-2014, it varied as much as €60 to €120/kg on the average of all cananga oil grades in Comoros [17]. In fact, US import of ylang ylang and cananga oil from Indonesia was about 1.5 tonnes (in 2005), and increased up to 3.2 tonnes (in 2007), but decreased significantly in the following years into 1.0 tonnes (in 2012). ITC also mentioned that premium ylang ylang oil imported from Madagascar to US showed a stable high price ranged from US\$96 into US\$225/kg in 2006 – 2012, but was dropped in the following year into US\$149/kg [18]. Nowadays, cananga oil price is up to 1.2 million rupiahs per litre from local distillerin Indonesia [19] and can be marketed as much as US\$ 175.00/kg by Ultra International B.V [20] in Netherland. This price is quite satisfying and stable compared to prices of other essential oil products.

Distillers of cananga oil consider two parameter on deciding quality of cananga oil, ester number and saponifikasi value. Distillation process is normally carried out in fractions to differentiate its qualities and usages. First oil obtained from distillation is called as extra grade which is comprised of 40% of total oil produced and used as based oil in high class perfumes such as Aqua di Gio (Giorgio Armani), Poison (Christian Dior) and Champ Elysee (Guerlin)[2]. The following fractions, first grade, second grade, and third grade, are cananga oils with lower quality in proposed usage as cosmetic based, soap, and detergent. Some research apply steam-water distillation on cananga oil and fractionate them in some uptake based on certain time interval, i.e in the first two hour of distillation, four hours from the first oil uptake, six hours from the second oil uptake, and 8 hours from the third oil uptake[11,12]. It is correlated to previous report by Buccellato [3] which mentioned that ylang flowers resulted in ylang-ylang oil yields in a series of oil sampling (intake) time are 0.33% (1 hour distillation: extra grade), 0.33% (3 hours after first oil intake: first grade), 0.38% (5 hours after second oil intake: third grade); 1.41% (9 hours after third intake: fourth grade). This ylang-ylang extra grade is rich in linalool, which is responsible for citrus and floral aromas, methyl benzoate is responsible for the marker odor of ylang-ylang flower, benzyl acetate and geranyl acetate which contribute to fruity floral odor.

Extra grade cananga oil is majority consisted of esters and ethers also aromatics, while the lower grade contains mainly of sesquiterpenes. Ylang-ylang oil contains higher level of β -linalool, benzyl acetate and other esters [21], but lower level of β -caryophyllene compared to cananga oil. Third grade of ylang-ylang oil would be similar to the quality of cananga oil. In detail, Ekundayo [13] has compared the quality of ylang-ylang oil [22] and cananga oil [3]. Lawrence [22] identified linalool (1.0-19.03%), β -caryophyllene (6.8-10.6%), germacrene-D (10.23%), and esters of farnesyl acetate (1.88%), 3-methylbut-2-enyl acetate (0.13-3.2%), geranyl acetate (3.5-6.7%), linalyl acetate (~ 0.2%), cinnamyl acetate (1.01%), methyl benzoate (1.0-8.7%), methyl salicylate (0.1-1.34%), benzyl acetate (2.8-25.1%), benzyl benzoate (2.2-16.4%), benzyl salicylate (1.9-5.2%) to be present in ylang-ylang essential oil. In the other hand, Buccellato [3] emphasized that cananga oil has lower quality than ylang-ylang oil since it only contains linalool (1.7%), geranyl acetate (1.8%), benzyl benzoate (2.9%), benzyl salicylate (0.1%), and a bulky β -caryophyllene (37.0%). Rachmawati et al [23] also mentioned some major chemical compounds in Java cananga oil, including β -caryophyllene (19.39%), germacrene-D (13.36%), linalool (11.28%) and α -humulene (9.46%).

2. Methods

2.1. Materials

Cananga flowers were obtained from Karangpring village, Jember district and collected on October 2018. Cananga plant was determined in Balai Konservasi Tumbuhan Kebun Raya Purwodadi, Lembaga Ilmu Pengetahuan Indonesia (Indonesian Institute of Sciences) as *Cananga odorata* (Lmk) Hook.f. & Thoms. Samples for distillation were the whole parts of flowers and a mixture of young and mature cananga flowers.

2.2. Procedures

Distillation equipment was set up according to Figure 1. Main kettle provided dual function extraction methods only by modifying steam filter inside kettle. Kettle could be a chamber of hydrodistillation when steam filter was taken out from kettle, and be a chamber of steam-water distillation when steam filter was put in the kettle. Samples were directly contacted with water inside kettle during heating in hydrodistillation process, while in steam-water distillation, samples were steamed in the same kettle and contacted with hot water vapours only.



Figure 1. (a) Real picture of distillation reactor; (b) Schematic representation of distillation reactor for extraction of cananga oil

Each distillation process employed 2 kg of cananga flowers with the amount of water as much as 20 L. Steam-water distillation required maximum of 2 hour distillation to avoid an empty kettle since water was run out after 2 hours. Hydrodistillation method was run for longer time of distillation up to 3 hours since water still presented in kettle at the end of distillation.

Cananga essential oil was separated from aqueous phase in distillate using separatory funnel. Cananga oil was presented in upper layer (organic phase), while lower layer contained aqueous phase which commonly called as cananga water. Cananga oil was then added for a few of magnesium sulfate anhydrate to absorb water residues left in the oil. Further, this cananga oil was subjected on chemical analysis using GCMS (Gas Chromatography Mass Spectrometry).

3. Results and Discussion

Hydrodistillation of cananga flowers resulted in cananga essential oils with percent yield of 0.33% (1 hour), 0.46% (2 hours), and 0.67% (3 hours), while steam-water distillation for 2 hour process has produced 0.60% of cananga oil. Previous research by Ferdiansyah et al [24] showed that eight hour steam distillation of cananga flower results in 1.95% of cananga oil, while Rachmawati [23] also obtain 1.066% of cananga oil from similar process to Ferdiansyah et al [24].

Table 1. Compositions of cananga oils in various distillation methods.

No	Compound Names	Type	Composition (%)			
			SWD 2 h	HD 1 h	HD 2 h	HD 3 h
1	p-methylanisole	LOC	0.62	5.07	0.17	0.21
2	methyl benzoate	LOC	-	0.33	-	-
3	β -linalool	OM/LOC	8.89	0.17	-	-
4	nonanal	LOC	-	0.11	-	-
5	benzyl acetate	LOC	0.1	0.11	-	-
6	geraniol	OM/LOC	2.03	3.65	0.13	0.11
7	Z-citral	OM/LOC	0.1	0.53	-	-
8	bicyclogermacrene	S	0.21	0.14	0.22	0.25
9	δ -elemene	S	0.1	-	0.14	0.16
10	eugenol	OM/LOC	1.77	0.56	0.11	-
11	geranyl acetate	OM/LOC	7.79	16.58	7.1	4.26
12	β -elemene	S	0.59	0.44	0.66	0.71
13	β -caryophyllene	S	20.31	16.15	25.1	28.46
14	β -gurjunene	S	0.5	0.36	0.5	0.54
15	alloaromadendrene	S	0.31	-	-	-
16	8-methylene-dispiro[2.1.2.4]undecane	HC	-	0.28	-	-
17	1-(2-methylene-3-butenyl)-1-(1-methylenepropyl) cyclopropane	HC	-	-	0.22	0.24
18	α -copaene	S	-	-	0.43	0.43
19	α -amorphene	S	0.44	0.32	-	-
20	α -humulene	S	6.5	5.42	8.13	8.96
21	δ -cadinene	S	0.14	0.14	0.2	0.15
22	germacrene D	S	22.57	15.3	22.79	25.76
23	farnesene	S	3.78	3.16	4.59	5.36
24	acetyl eugenol	OM/HOC	0.19	-	-	-
25	γ -cadinene	S	-	-	0.19	0.2
26	aromadendrene VI	S	1.46	1.78	1.43	1.4
27	1S-cis-calamenene	S	0.2	-	-	-
28	(-)-isolekene	S	-	0.21	0.21	0.19
29	α -cubebene	S	-	-	0.09	0.09
30	elemol	OS/HOC	0.31	0.34	0.27	0.18
31	cis-3-hexenylbenzoate	HOC	0.23	0.24	0.16	0.11
32	germacrene D-4-ol	OS/HOC	0.69	0.75	0.24	0.18
33	patchulane	S	0.15	0.15	0.14	0.15
34	spathulenol	OS/HOC	0.33	0.49	0.32	0.28
35	α -cedrol	OS/HOC	0.18	0.19	0.34	0.36
36	δ -cadinol	OS/HOC	0.63	0.71	0.91	0.9
37	α -cadinol	OS/HOC	0.67	0.79	1.08	1.06
38	α -copaene-11-ol	OS/HOC	-	-	0.11	-
39	farnesol	OS/HOC	6.93	9.54	8.47	6.42
40	farnesal	OS/HOC	-	0.1	0.11	0.11
41	benzyl benzoate	HOC	9.2	13.38	12.47	9.93
42	10-(acetylmethyl)-(+)-3-carene	OM/HOC	-	0.11	0.16	0.15
43	Farnesyl acetate	OS/HOC	0.51	0.38	0.53	0.59
44	2-(acetylmethyl)-(+)-3-carene	OM/HOC	-	0.15	0.2	0.18
45	benzyl salicylate	HOC	1.32	1.57	1.68	1.47
46	geranyl isobutyrate	OM/HOC	0.26	0.29	0.42	0.46

SWD 2 h: steam-water distillation for 2 hours; HD 1 h: hydrodistillation for 1 hour; HD 2 h: hydrodistillation for 2 hours; HD 3 h: hydrodistillation for 3 hours.

Quality of those cananga oils were analyzed based on their chemical compound compositions using GCMS [14,15,21]. Based on previous research by Buccellato [3] and Ekundayo [13], it was mentioned that high quality of cananga oil relies on the content of esters and its marker compounds, such as methyl benzoate and benzyl acetate, two dominant esters in cananga oil, and also β -linalool. Table 1 listed the number of chemical compounds together with their percentages presented in cananga oils. Some esters presented in these cananga oils were methyl benzoate, benzyl acetate, benzyl salicylate, geranyl format, benzyl benzoate, and geranyl acetate (as major esters).

Table 2. List of the compound types which determine the quality of in cananga oils.

Components	Composition (%)			
	SWD 2 h	HD 1 h	HD 2 h	HD 3 h
Non-oxygenated hydrocarbons	57.26	43.85	65.04	73.05
Oxygenated hydrocarbons	42.75	56.14	34.98	26.96
Esters	19.60	32.88	22.36	16.82
β -linalool	8.89	0.17	-	-

SWD 2 h: steam-water distillation for 2 hours; HD 1 h: hydrodistillation for 1 hour;
HD 2 h: hydrodistillation for 2 hours; HD 3 h: hydrodistillation for 3 hours.

Therefore, it can be summarized in Table 2 that one hour and two hour hydrodistillation contained 32.88% and 22.36% of esters, respectively, while in two hour steam-water distillation contained only 19.60% of esters. Interestingly, β -linalool could be obtained in significant amount only from steam-water distillation method as much as 8.89%, while from one hour hydrodistillation obtained only for 0.17%. No β -linalool can be found in cananga oil resulted from longer time of hydrodistillation processes.

It also can be seen from Table 2 that oxygenated hydrocarbons were much higher in hydrodistillation for an hour process (56.14%), and were reduced significantly in a longer time of distillation processes (two hours and three hours). Steam-water distillation for 2 hours brought about 42.75% of oxygenated hydrocarbons which much higher than hydrodistillation product in similar time of distillation process. Oxygenated hydrocarbons content was important to know since it was responsible for bioactivity of cananga oil. On the other hand, non-oxygenated hydrocarbon contents of cananga oils increased during the longer time of hydrodistillation method. β -caryophyllene, germacrene-D, α -humulene and farnesene were four non-oxygenated hydrocarbons that increased in time to time during hydrodistillation process.

Table 3. Classes of essential oil compounds in cananga oils.

Components	Composition (%)			
	SWD 2 h	HD 1 h	HD 2 h	HD 3 h
Non-oxygenated monoterpenes (M)	-	-	-	-
Oxygenated monoterpenes (OM)	21.03	22.04	8.12	5.16
Non-oxygenated sesquiterpenes (S)	57.26	43.57	64.82	72.81
Oxygenated sesquiterpenes (OS)	10.25	13.29	12.38	10.08
Light oxygenated compounds (LOC)	21.30	27.11	7.51	4.58
Heavy oxygenated compounds (HOC)	21.45	29.03	27.47	22.38
Hydrocarbons non-terpenoid and aromatic (HC)	-	0.28	0.22	0.24

SWD 2 h: steam-water distillation for 2 hours; HD 1 h: hydrodistillation for 1 hour; HD 2 h: hydrodistillation for 2 hours; HD 3 h: hydrodistillation for 3 hours.

Table 3 shows the detail of groups on essential oil compounds based on data on Table 1. Oxygenated monoterpenes (OM) is defined as monoterpenes containing oxygen atom in their structures which then could be in a form of alcohol or ester monoterpenes. This term also applied for oxygenated sesquiterpenes (OS) group. Light oxygenated compounds (LOC) is defined as oxygenated

compounds with low molecular weight and boiling point properties, including some oxygenated monoterpenes and oxygenated aromatics. Heavy oxygenated compounds (HOC) consisted of oxygenated sesquiterpenes, other oxygenated aromatics and monoterpenes with higher molecular weight and boiling points. Interestingly, no monoterpenes could be found in all cananga oils. Data on Table 3 also supported information on the previous paragraphs whether the highest quality of cananga oil was obtained from hydrodistillation process for an hour. The presence of 22.04% of oxygenated monoterpenes, 13.29% of oxygenated sesquiterpenes, and 27.11% of light oxygenated compounds were leading to a judgement for quality characteristic of cananga oil.

According to those information, it can be suggested that one hour hydrodistillation of cananga flowers could gain first grade quality of cananga oil, compared to the following hours of hydrodistillation oil products, in term of the highest content of oxygenated hydrocarbons and esters. However, steam-water distillation method was quite satisfying on producing cananga oil with a huge amount of β -linalool, and higher amount of oxygenated compounds and esters compared to hydrodistillation method in the same time of distillation, 2 hours running.

4. Conclusion

Yield of cananga oils increased with the longer time of hydrodistillation processes was took place. However, it had found that the best quality of cananga oil was obtained in one hour hydrodistillation process since it was counted for the highest content of esters, such as geranyl acetate and benzyl benzoate. The non-oxygenated hydrocarbon contents in cananga oil increased considerably during the longer time of hydrodistillation, which majority were consisted of β -caryophyllene, gemacrene-D, α -humulene dan farnesene. Nevertheless, β -linalool was available in significant amount up to 8.89% only from steam-water distillation processes.

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