

Chemical Composition of Essential Oil of Cendana Frangipani Flower (*Plumeria alba*) With Re-Extraction Using Ethanol Solvents

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Abstract

One potential source of essential oils in Bali which has not been developed is from cendana frangipani flower. Cendana frangipani flower has a very distinctive scent and very popular in Bali, it has an important function in the local culture, used as fragrances, incense (prayer facilities especially in Bali), and as an air freshener aromatherapy in the spa industry. The fragrance is typical due to their volatile oil which content in the cendana frangipani flower. The quality produced of essential oils are determined by their chemical composition of the extraction process. Previous research indicates that the good quality of frangipani immature essential oil extracted through the extraction process using hexane resulting in concrete form which still has the solvent aroma, semi-solid consistency and dark yellow. Therefore, it is necessary to modify the process by changing the concrete into the absolute product by re-extraction using ethanol. The purpose of this study was to determine the differences in chemical composition of essential oils of cendana frangipani flowers resulting from the re-extraction by using a particular comparison between ethanol and concrete. Fresh cendana frangipani flowers were sliced and then extracted with n-hexane in a Soxhlet extractor to produce concrete volatile oil. The concrete was further re-extracted using absolute ethanol to produce essential oil with a concrete and ethanol comparison treatment consisting of 1:4, 1:6, 1:8, and 1:10. Absolute essential oil were then analyzed by GC-MS. The analysis showed that the absolute cendana frangipani flower essential oil produced by different treatment has a different compounds composition and relative percentages varied. The chemical composition of cendana frangipani absolute essential oils was classified as alcohols, terpenes, ketones, esters, and acid with the concentration from 2.65 to 24.77%; 0.34 to 1.35%; 0 to 1.38 %; 13.38 to 24.23% and from 22.74 to 58.15% respectively.

Keywords : absolute, concrete, essential oil of cendana frangipani flower, *Plumeria alba*, re-extraction

1. INTRODUCTION

The success of making essential oils from raw materials and the quality of essential oil produced is determined by the extraction method and the conditions of extraction process (Wijaya, 1995; Yusufoglu *et al.*, 2004; Ozek *et al.*, 2006a; Boutekedjiret *et al.*, 2004; Ozek *et al.*, 2006b; Wartini *et al.*, 2008 and Wartini *et al.*, 2010).

The extraction method which performed to obtain the essential oil from raw material are steam distillation, extraction with volatile solvents and extraction with solid fats. Extract with volatile solvent resulting in a solid extract

that usually still mixed with other compounds which have the same or a similar polarity to the extracting solvent (Saputra, 2010 and Tenaya, 2011). Saputra (2010) showed that the solvent extract of fragrant pandan leaves composed of 3 methyl pentane (9.01%), 2,2 dimethyl pentane (8.95%), methylcyclopentane (21.10%), Cyclohexane (5, 06%), and 9,12,15 octadecatrienoic methyl ester (1.86%). Tenaya (2011) showed that extracts of sandalwood frangipani flowers which produced by solvent extraction using n-hexane contain 46 kinds of compounds consisting of a group of alkanes, alkenes, alcohols, esters, and terpenes. One

step extraction of frangipani flower was resulting in a substance which not meet the criteria for substance smelling aroma therapy. It's caused by the solvent which still mixed with other compounds such as waxes, pigments and semi-solid form, therefore the improvement of the process is still needed. To overcome the lack of this extraction process, a modification method was introduced by making the process of solvent extraction with n-hexane which followed by re-extraction using ethanol solvent.

The purpose of the study was to determine the chemical composition of cendana frangipani flower essential oil produced from the re-extraction treatment with ethanol.

2. MATERIAL AND METHODS

2.1. Material

Materials used in the study are cendana frangipani flowers fresh obtained in Mengwi, Badung regency, Bali. Chemicals used were n-hexane, 90% ethanol, anhydrous MgSO₄ and distilled water obtained from Bratachem store in Denpasar.

2.2. Equipment

Equipment used in the study (1) equipment for the extraction process is a set of tools Soxhlet extraction, stainless steel knife, thermometer, separating funnel, filter paper Whatman No. 1, rotary vacuum evaporator (Janke & Kunkel RV 06 - ML) and (2) equipment for gas-chromatographic analysis is mass spectrometry (GCMS-QP2010 Shimadzu) and the analytical balance (Ohaus P213).

2.3. Methods

Cendana frangipani flower was extracted with n-hexane. Cendana frangipani flowers were sliced crosswise with size of ± 1 mm which aimed to expand upon the surface of the material extracted. Slices of frangipani flowers of approximately 50 g was extracted in a Soxhlet extraction flask using solvent of n-hexane. The extraction process was carried out for 4 hours to obtain a mixed solvent extracts. The mixed solvent extract was then added with anhydrous MgSO₄ (1% w/v) to absorb the remaining water in the extract. The extract was evaporated with a rotary vacuum evaporator to

obtain a semi-solid extract (concrete)

Re-extraction proses with ethanol

The concrete of cendana frangipani flower essential oil were put into the flask and it was added with ethanol according to the treatment (a comparison of concrete with ethanol as 1:4, 1:6, 1:8, and 1:10). It was allowed to stand in the cold temperature for 30 minutes until the wax precipitate was formed in the extract. The wax was then filtered with Whatman No. 1 filter paper to separate it from the extract. The solvent was then evaporated by a vacuum rotary evaporator at 40°C, 100 mbar to evaporate the ethanol and to obtain thick cendana frangipani flower extract (absolute essential oils). The absolute essential oils was put in the dark bottle and it was stored at cold temperatures (5-8°C in a refrigerator) prior to analysis.

Analysis by GC / MS

Identification of essential oil compounds was performed by GC-MS (Shimadzu GCMS-QP2010), using 30 m columns (Rastek stabilwakR-DA), and 0.25 mm of inner diameter. The operating conditions was as follows: the initial column temperature was 60°C, the final temperature of 215 °C with the increase 4°C / min, injector temperature 250°C, helium carrier gas, ionizing type EI (Electron Impack), injected sample volume of 0.1 mL. Identification of compounds was carried out by comparing the spectra of compounds in the essential oil of cendana frangipani flower with spectra in the NIST and Wiley libraries.

3. RESULTS AND DISCUSSION

Chromatogram of cendana frangipani absolute essential oils which analyzed with GC-MS is presented in Figure 1, 2, 3, and 4. Compounds which suspected as the constituent of cendana frangipani absolute essential oils obtained from re-extraction and its classification of such constituent compounds are presented in Tables 1 and 2. Tables 1 and 2 showed that the cendana frangipani flower absolute essential oil compounds are composed of alcohol group (2.65 to 24.77%), terpenes (0.34 to 1.35%), ketones (0 to 1.38%), ester (13.38 to 24.23%) and acid (22.74 to 58.15%).

The re-extraction with ethanol resulting in

differences in composition of the constituent for all treatment (a comparison of concrete and ethanol as 1: 4, 1:6, 1:8, 1:10).

The relative concentration of the class of compound for alcohols and acids increase correspond to the increasing number of ethanol used in the re-extraction. This relate to the solubility of a compound which determined by the polarity of the solven tresemblance with dissolved compounds. Similarly, the relative concentration of terpenes and esters increase correspond to the increasing number of ethanol used in the re-extraction despite the result of treatment 1:10 (comparison of concrete and ethanol) which showed that both compounds are not found. This is likely due to changes in the two compounds since both are unstable.

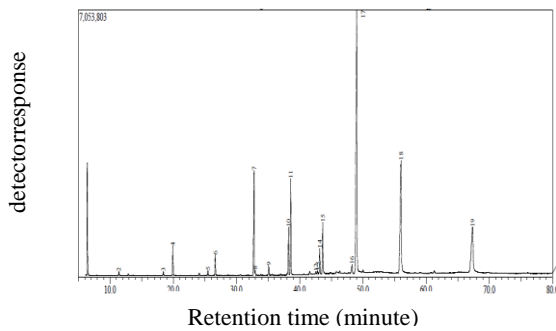


Figure 1. Chromatograms of compounds in the cendana frangipani flower absolute essential oil comparison of the results of re-extraction with ethanol concrete 1: 4

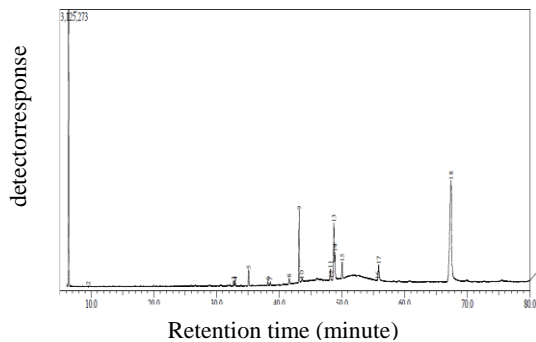


Figure 2. Chromatograms of compounds in the cendana frangipani flower absolute essential oil comparison of the results of re-extraction with ethanol concrete 1: 6

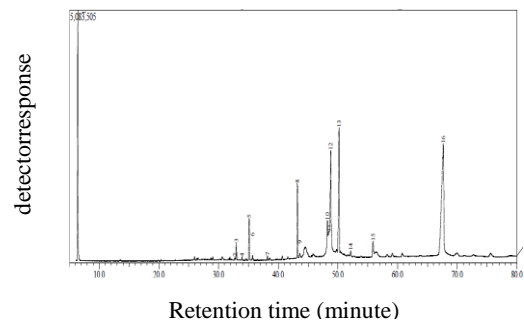


Figure 3. Chromatograms of compounds in the cendana of frangipani flower absolute essential oil comparison the results of re-extraction with ethanol concrete 1: 8

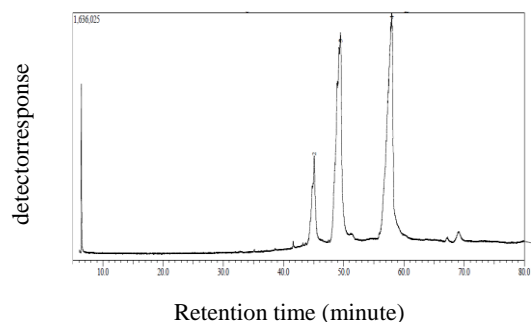


Figure 4. Chromatograms of compounds in the cendana frangipani flower absolute essential oil comparison of the results of re-extraction with ethanol concrete 1: 10

The relative concentration of the class of compound for alcohols and acids increase correspond to the increasing number of ethanol used in the re-extraction. This relate to the solubility of a compound which determined by the polarity of the solven tresemblance with dissolved compounds. Similarly, the relative concentration of terpenes and esters increase correspond to the increasing number of ethanol used in the re-extraction despite the result of treatment 1:10 (comparison of concrete and ethanol) which showed that both compounds are not found. This is likely due to changes in the two compounds since both are unstable.

Table 1. Compounds suspected as a constituent of cendana frangipani flower absolute essential oils

No.	Retenti- on time (minute)	Compounds	Relative concentrations (%)			
			Comparison of concrete and ethanol			
			1:4	1:6	1:8	1:10
1.	6.395	Etanol	6.15	24.77	16.97	2.65
2.	11.390	2-Heptanone	0.20	-	-	-
3.	18.43	2-nonanone	0.18	-	-	-
4.	19.934	Ethyl octanoate	1.84	-	-	-
5.	25.489	2-Undecanone	0.28	-	-	-
6.	26.658	Decanoic acid, ethyl ester	1.18	-	-	-
7.	32.791	Dodecanoic acid, ethyl ester	7.56	0.60	0.12	-
8.	32.957	Geraniol	0.34	0.69	1.35	-
9.	35.096	Phenylethyl Alcohol	0.64	2.30	1.77	-
10	38.243	Tetradecanoic acid, ethyl ester	3.01	0.45	0.22	-
11	38.593	Octanoic acid	7.28	0.28	-	-
12	41.589	Eugenol	-	0.63	-	-
13	42.582	Delta decalactone	0.10	-	-	-
14	42.848	-	0.20	-	-	-
15	43.178	Hexadecanoic acid, ethyl ester	1.83	8.47	5.60	-
16	43.655	Decanoic acid	3.77	-	-	-
17	45.102	Octadecanoic acid	-	-	-	10.38
18	48.287	Octadecanoic acid, ethyl ester	0.76	1.54	3.73	-
19	48.705	Ethyl Oleate	-	8.93	13.98	-
20	49.032	Dodecanoic acid	32.51	3.98	-	-
21	49.467	Hexadecenoic acid	-	-	-	40.38
22	50.033	Llinoleic acid, ethyl ester	-	2.32	14.86	-
23	56.050	Tetradecanoic acid	16.96	3.65	1.82	-
24	57.591	9,12-Octadecadien-1-ol	-	-	-	46.59
25	67.341	Hexadecanoic acid	9.11	41.31	33.56	-
26	90.625	-	2.88	-	-	-
27	90.717	-	0.62	-	-	-
28	90.801	Heptadecene-(8)-carbonic acid	2.58	-	-	-

Table 2. Classification of compounds suspected as a constituent cendana frangipani flower absolute essential oil

Class of compound	Relative concentrations (%)			
	Comparison of concrete and ethanol			
	1:4	1:6	1:8	1:10
Alcohol	6.79	27.76	18.74	49.24
Terpene	0.34	0.63	1.35	-
Keton	1.38	-	-	-
Ester	14.34	13.38	24.23	-
Acid	22.74	58.15	49.36	50.76

4. CONCLUSIONS

Cendana frangipani flower absolute essential oil which obtained from re-extraction with ethanol in difference comparison of concrete and ethanol has different compounds composition and relative percentages varied.

The constituent of cendana frangipani flower absolute essential oils compounds belonging to the class of alcohols, terpenes, ketones, esters, and acids with the relative concentrations of 2.65 to 24.77%; 0.34 to 1.35%; 0 to 1.38%; 13.38 to 24.23%; and from 22.74 to 58.15% respectively.

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