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Isolation of Essential Oils from Qust Al Hindi (Saussurea Lappa) Plants via Hydro distillation and Characterization using GC-MS Analysis

Nila Tanyela Berghuis*, Indah Permata Sari , Brikita Eusebes Suka Tendel, Salsa Safitri Putri Ely, Ana Lailatul Farida

Departement of Chemistry, Faculty of Science and Computer, Universitas Pertamina, Jalan Teuku Nyak Arief Simprug, Kebayoran Lama, Jakarta 12220.

*corresponding author's: nila.tanyela@universitaspertamina.ac.id

ABSTRACT

This research is motivated by the increasing public need for essential oils. The increasing demand is due to the spread of public knowledge in traditional medicine. The essential oil obtained from the Qust al Hindi plant (Saussurea Lappa) has various secondary metabolites such as flavonoids, lignin, and sesquiterpenoids. Isolation of essential oils from Qust AL-Hindi (Saussurea Lappa) plants was carried out using the hydro-distillation method. The results obtained are a solution that has a distinctive aroma of 0.41%. Compound content analysis was carried out using the GC-MS (Gas Chromatography-Mass Spectroscopy) method and identified the dominant metabolite compounds namely 9,12,15-Octadecatrienoic acid, Caryophyllene oxide, beta-elemene, trans-Caryophyllene, Cyclohexane, and Bicyclo with variations different area sizes.

Keywords: Essential Oil Isolation, Hydro-distillation, *Saussurea Lappa*, Sesquiterpene.

INTRODUCTION

Essential oils better known as aromatherapy oils are oils extracted from plants. According to the Indonesian Essential Oil Council, there are 40 types of Indonesian essential oils traded on the world market. The demand for essential oils increases every year. This is due to the widespread information about the benefits of essential oils that the body gets when using these oils. Because essential oils are obtained by extracting plants, the Asian continent is the most potential producer of essential oils because it has a diverse climate. China and India are the main suppliers followed by Indonesia, Sri Lanka, and Vietnam [1]

Currently, essential oils are widely used by industry as raw materials for making perfume, cosmetics, fragrances, medicines and aromatherapy. Companies producing essential oils are still relatively few in Indonesia, so the prices are relatively expensive. This is inversely proportional to the raw materials for making essential oils which are very cheap. Meanwhile, Indonesia is very rich in plants that can be used as medicine, including producing essential oils [2]

Indonesia has various superior and potential plants that are capable of producing essential oils. However, essential oil production in Indonesia is still not optimal because the cultivation of essential oil-producing plants and their processing methods are not appropriate. So to improve the essential oil production process, optimization of the extraction process is needed. Essential oils are obtained through an extraction process from various plants including lemongrass, eucalyptus, cloves, basil, roses, pine, ylang-ylang, and patchouli. Patchouli plants are essential oil-producing plants that can contribute around 60% of foreign exchange [3]. According to sources from the Indonesian Essential Oil Association, essential oil production in

Indonesia has decreased by 200 tons. Meanwhile, the world's need for essential oils reaches 1,500 tons per year, and Indonesia only supplies 70% [4]. One of the natural ingredients, Qust al-Hindi.

Qust al-Hindi (Saussurea Lappa) known as Costus or kuth originates from South Asia [5]. As a medicine, this plant is used intensively in conventional medicine [6]. Such as tumors, seizures, duodenal ulcers, liver injury, microbial infections, and arthritis [7]. In Saudi Arabia this plant is also able to treat women's complaints, coughs, and is a carminative [8]. Saussurea Lappa contains costunolide and isodihydrocostunolides compounds. This compound is biologically active and holds great promise for developing new drugs [7]. Therefore, in this research, an analysis of the essential oil content of the Qust Al Hindi Plant was carried out.

EXPERIMENTAL SECTION

Material and Instrumental

The chemical tools used in this research were analytical balance (Ohaus Pioneer), 1 set of steam distillation tools, thermometer, stand, clamps, magnetic stirrer, 100-1000 mL beaker (Iwaki), 100 ml measuring cup (Pyrex), volume pipette (Iwaki), dropper pipette, Buchner funnel, filler, spatula, vial, rotary evaporator (Heidolph), filter paper, watch glass and hotplate (IKA C-MAG HS7). Meanwhile, the chemicals used in this research were Qust al Hindi powder, technical n-hexane, technical ethyl acetate, and anhydrous sodium sulfate p.a (Merck). Then the results of the essential oil extraction were characterized using Gas Chromatography – Mass Spectrometry (GC-MS) (Shimadzu QP 2010 SE).

Isolation of essential oils using the Hydro-distillation method

Isolation of essential oils is carried out through a steam distillation process. The first step taken when extracting essential oils is to put 300 grams of Qust al Hindi powder into flask 2 (figure 1) which measures 1000 mL, then add distilled water ¾ of the main flask (number 1). After that, the distillation tool is assembled with the help of stative and clamps. Turn on the hotplate with a temperature of 125°C. Distillation was carried out for 4 hours until the collected distillate was finished. The water temperature is maintained at around 100°C so that the compounds contained in Qust al Hindi powder are not damaged. The distillate obtained is then stored in a vial and then the mass is weighed.

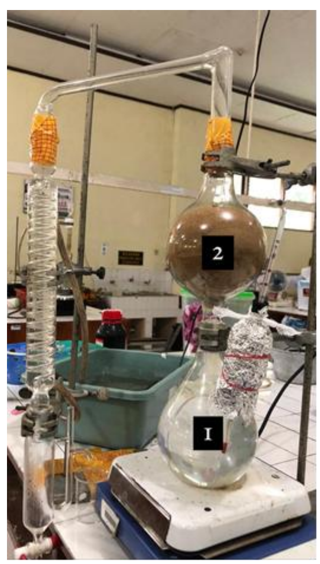


Figure 1. Steam distillation equipment circuit.

Characterization of Essential Oils

Qualitative and quantitative analysis of extracted essential oil of S. lappa was performed on Perkin-Elmer-Clarus-500 GC equipped with Clarus-500 MS and capillary column (60 m × 0.25 mm, film thickness 0.25 µm). Injector and detector temperatures were 210 and 280°C, respectively, while helium was used as carrier gas. Oven temperature was held for 5 min at 50°C with 5 min solvent delay, then programmed at

3°C/min up to 220°C/min, and then maintained isothermally at 220°C for 20 min. GC-MS was operated in electron ionization (EI)

RESULTS AND DISCUSSION

Isolation of essential oil from the Qust Al Hindi Plant through steam distillation for 4 hours produced a yellowish-white liquid of 1.2152 g (= 1.2152 g/300 g = 0.41%). The results of this research are better than research conducted by Negi, et.al 2013.

Figure 2. Products resulting from the isolation of essential oils from the Qust Al Hindi plant

Based on research conducted by Negi, et.al 2013, the individual constituents were separated by gas chromatography and identified by comparing their MS to those of the National Institute of Standards and Technology, U.S. Department of Commerce (NIST) and Wiley (John Wiley & Sons Ltd) mass spectral libraries. Upon GC-MS analysis, the hydrodistilled oil was found to contain 42 constituents eluted between 10 to 65 min. Among these constituents, 12 are found to be major constituents representing 58.18% of the oil, which are mainly comprised of sesquiterpenes such as β-costol (13.55%) and δ-elemene (12.69%) were α-selinene (5.02%), β-selinene (4.47%), α-costol (4.02%), 4-terpinol (3.38%), elemol (3.21%), α-ionone (3.13%), β-elemene (3.00%), (-)-γ- elemene (2.08%), p-cymene (1.96%) and 2-β-pinene (1.57%). Maurer and Grieder (1997) had also reported -)-α-selinene, (+)-selina-4, 11-diene, (-)-α-*trans*bergamotene, (-)-α-costol, (+)-γ-costol, (-)-elema-1,3,11 (13)-trien-12-ol, (-)-α-costal, (+)-γ-costal, (-)-elema- 1,3,11(13)-trien-12-al, (-)-(E)-*trans*-bergamota-2,12-dien- 14-al, (-)-*ar*-curcumene and (-)-caryophyllene oxide as major components of *S. lappa* essential oil [9]. In another study, Liu et al. (2012) identified 39 components from the essential oil of *S. lappa* roots. The principal compounds in *S. lappa* essential oil were dehydrocostus lactone (46.75%), costunolide (9.26%), 8- cedren-13-ol (5.06%) and α-curcumene (4.33%) [10].

In this study, Characterization of essential oil content was carried out using the GC-MS analysis method. The results were obtained in the form of a chromatogram with various peaks in the GC spectrum as in the image above.

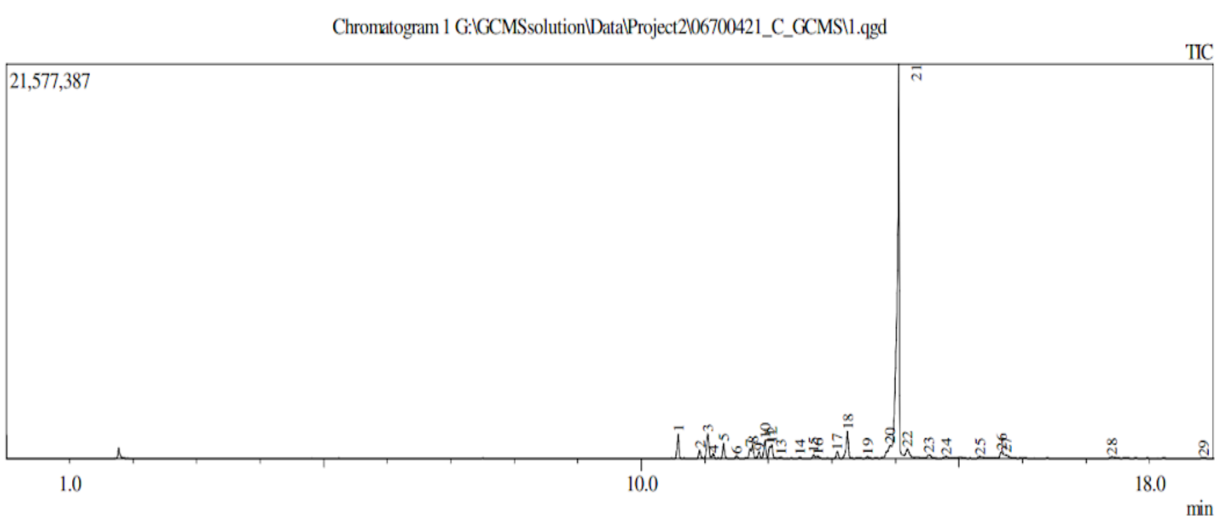
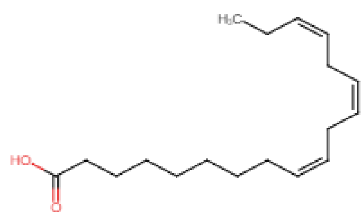
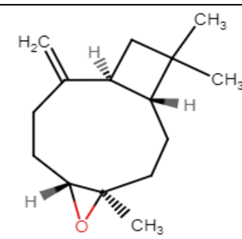
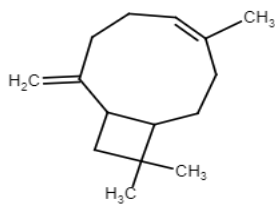
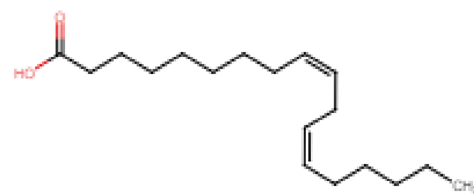
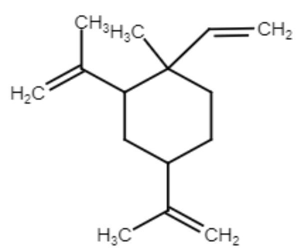
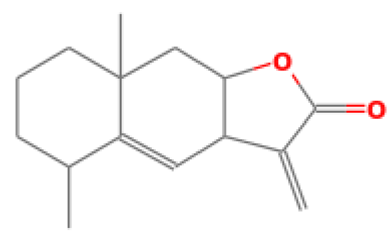


Figure 3. GC-MS results of Qust al-Hindi essential oil sample

Sample has 29 peaks which can be seen in table 1. In sample there are 6 dominant compounds that appear. there are 6 dominant compounds. 9,12,15-Octadecatrienoic acid (60,81%); *Caryophyllene oxide (4,32%); trans-Caryophyllene (4,16 %); 9,12-Octadecadienoic acid (3,77%);* Beta elemene (2,52 %). However, the percentages of all these compounds greatly varied in the other reported studies. Existing variations in essential oil composition of *S. lappa* may be attributed to factors related to ecotype, chemo-type, phenophases and the variations in environment conditions such as temperature, relative humidity, irradiance and photoperiod. Moreover, the genetic background may also affect the chemistry of secondary metabolites of plants [11]. The following is a summary of the structures of the dominant compounds in essential oils in Table 1.

Table 1. Structure of the dominant compounds of Qust al-Hindi essential oil sample

 *9,12,15-Octadecatrienoic acid (Compound A)*	 *Caryophyllene oxide (Compound B)*
 *trans-Caryophyllene (Compound C)*	 *9,12-Octadecadienoic acid (Compound D)*
 Beta elemene (Compound F)	 Eudesma-5,11 (13)-dien-8,12-olide (Compound G)

From the GC-MS results on the IK.1 sample, it can be concluded that the predictions of the compounds that appear are following previous research [12]. It's just that the difference between the two lies in the most dominant compound. In sample IK.1 the most dominant compound was 9,12,15-Octadecatrienoic acid at a retention time of 14.052 min with an area % of 60.81. Meanwhile, in previous research [12] the most dominant compound was Eudesma-5,11 (13)-dien-8,12-olide at a retention time of 45.30 min with a % area of 52.01 (compound G). This is because, during extraction, the solvent used is different. In previous research, dichloromethane solvent was used, while in this research water was used.

Compound	(%) Area	Retention Time (mnt)
Eudesma-5,11 (13) -dien-8,12-olida	52.01	45.30
Elemene	7.18	48.21
Fenantrenon	2.97	46.11
Caryophyllene oxide	2.39	42.27
9,12-Octadecadienoic acid (Z, Z) -	2.13	52.20
Sikloheksana	2.12	23.76
Germacra-1 (10), 4,11 (13) -trien-12-oic acid, 6à-hydroxy-, ç-lactone, (E, E) -	1.96	43.84
Androstan-17-one, 3-ethyl-3-hydroxy- , (5à) -	0.82	41.68
Bicyclo [10.1.0] tridec-1-ene	0.81	34.81
Naftalena	0.74	27.42
Cedren-13-ol, 8-	0.62	35.00
4a, 8-Dimetil-2- (prop-1-en-2-yl) -1,2,3,4,4a, 5,6,7-oktahidronaftalena	0.47	26.71

Figure 4. Data from GC-MS analysis [12].

CONCLUSION

Based on the results of the final project research regarding the isolation of essential oil extraction, the percent yield (%) was 0.41% with the dominant compound being 9,12,15-Octadecatrienoic acid (60.81%); Caryophyllene oxide (4.32%); trans-Caryophyllene (4.16%); 9,12-Octadecadienoic acid (3.77%); Beta element (2.52 %).

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