



VOLATILE COMPOUNDS' CHARACTERIZATION OF N-HEXANE EXTRACTS OF TERASA'S FOREST HONEY

Aulia Winaldi*, Alfian Noor, and Firdaus

Department of Chemistry, Hasanuddin University, South Sulawesi, Indonesia

*Corresponding author: auliawinaldi@yahoo.co.id

ABSTRACT

This study aims to identify volatile compounds that are characteristic of the honey of Terasa village of Sinjai regency of South Sulawesi, volatile honey extracted with n-hexane solvent then identified by Gas Chromatography Mass Spectroscopy (GCMS) method. The results obtained 59 volatile compounds derived from honey are grouped into compounds namely hydrocarbons, aldehydes, ketones, alcohols, esters, bensenoid and carboxylic acids. Based on comparative results with other honey regions it is known that volatile compounds that become characteristic of honey Terasa village that is compound Tetrasilaoktana

Keywords: honey of Terasa, volatile compound, and GCMS.

Received : 28 November 2017, Accepted : 28 April 2018, Published online: 30 May 2018

1. INTRODUCTION

Forest as a source of life for some biological and animal species, has economic potential in the form of timber forest products (HHK) and non-timber forest products (HHBK). One of the HHBK is honey which is the priority of developing the Ministry of Forestry into superior products [5]. Honey is a special honey food made up of about 181 compounds classified as carbohydrates, enzymes, amino acids, minerals, vitamins, trace elements, volatiles, and polyphenols [2]. The composition of honey are influenced by two things, namely the basic composition of nectar produced and successfully collected by bees and external factors such as weather and climate. This component affects the nutrients of

honey so it is believed to be beneficial to health and helps cure diseases [3] and gives unique taste and aromatic nature. The smell of honey comes from the substances produced by the gland cells mixed in the nectar and the fermentation process of sugars, amino acids, and vitamins during the maturation of honey so it tends not to settle because this substance will evaporate with increasing time, especially if the honey is not stored properly. Some volatile compounds, all derived directly from the flowers that have been visited by bees, so the aroma of honey produced has a floral origin, color and flavor of honey depends on the plants around the honeycomb [8]. Therefore, one of the methods for the

falsification of honey products is the detection of aroma, taste and color.

Factor affecting the characteristics of honey aroma is the volatile elements contained in them, especially gluconic acid and proline [9] can be seen in monofloral honey with pine flower source there are 42 types of volatile compounds [11] and typical honey of the Chilean state, volatile compounds that contained in this type of honey amount to 45 types of compound [5], whereas in multifloral honey there are 68 types of volatile compound [13], so the forest honey is multifloral honey because the source of nectar varies.

Forest honey bee makes nests in altitudes and hilly areas so as to pick up the flowers as a common source of nectar that grows at altitude. One of the conditional areas for growing and developing honey bees is the Terasa village. It is considered a rainforest area with a topographic altitude, sloping and bumpy. Because the shape of its topography is thought to be volcanic rock and igneous rocks from the coast to an altitude, so it is estimated that part of the spermonde islands in South Sulawesi because it has the same geographical and correlation conditions. Based on the above description, it is necessary to do research the influence of the volatile compound from Terasa village forest honey as a giver characteristic of the honey.

2. MATERIAL AND METHODS

2.1 Tools and materials

The materials used are honey of Terasa Village, n-hexane, aquabides, Na₂SO₄ The tools used in this case include Agilent

5975C GCMS model, 100 ml glass, 10 ml volume pipette, 1 ml pipette, vortex, sonicator, analytical balance, mixer, centrifuges, 10 ml bottles, 30 ml chocolate bottles and commonly used laboratory equipment.

2.2 Time and Place of Study

This research was conducted in May 2015 until July 2015 at Chemical Laboratory of Chemistry Department Faculty of Mathematics and Natural Sciences Hasanuddin University, Test Laboratory of Indonesian Plantation Product Center Makassar and Makassar Forensic Laboratory

2.3 Research Procedure

Sample Preparation

Sampling was done in honey forest Village Terasa Kab. Sinjai South Sulawesi with three different points. The honey sample is stored in a cool, dry glass bottle. First the honey is weighed (1g) and diluted with 1 mL of aquabidest. The mixture of divortex for 2 -3 minutes to ensure mixing of honey and aquabidest before liquid-liquid extraction and followed by identification of compound using GC-MS.

Liquid Extraction

First, n-hexane (4 mL) is added to a closed vial containing diluted honey. Then, the mixture divortexes at 1500 rpm for 2 minutes before being disicited for 20 minutes to separate the organic layer from the aqueous layer. The top layer containing the organic solvent is transferred to the closed vial while the lower layer is added with the same solvent then the above method is repeated three times. The first, second and third extraction of organic

coatings was combined and centrifuged at 2500 rpm for 10 minutes and the top layer was piped. Then added Na₂SO₄ to remove the remaining water and ready to be analyzed using GC-MS.

Analysis with Gas Chromatography-Mass Spectrometry (GC-MS)

GC-MS analysis was performed on Agilent 5975c. The columns are HP-5MS fused silica columns of capillaries and helium running at a constant pressure used as carrier gas. The following conditions are used: initial temperature 40 oC, equilibrium time 5 minutes, road 25 oC / min, final temperature 310 oC. All peaks are identified based on mass spectral matching ($\geq 90\%$) of both NIST and Wiley libraries

3. RESULT AND DISCUSSION

Based on research that has been done, the amount of volatile compound in honey Terasa Village as much as 59 volatile compounds (table 1.) obtained from the extraction of honey using n-Hexan solvent. Previously volatile compounds were studied in honey in other areas, ie, in which many Palestinian honey were identified as 30 volatile compounds^[7], Malaysian honey has been identified as 34 volatile compounds^[12], as well as honey from the region Chile that produces 34 volatile compounds^[4]. As for the area located in one province and using n-Hexan solvent, trigone bone honey was successfully identified as many as 18 volatile compounds^[1], honey bone as much as 21 volatile compounds^[14] and Mallawa honey as much as 35 volatile compounds^[10].

Table 1. Results Analysis of Volatile Compounds from Honey Terasa Village

No	Compounds Name	M1	M2	M3
1	Sikloheksena	√	-	-
2	3,4,4 trimetil sikloheksana	-	-	√
3	Dodekana	√	-	-
4	Tetradekana	√	√	√
5	2 metil 3 desen 5 one	√	-	-
6	Fenol	√	-	-
7	Heptasiloksan	√	√	√
8	Heksasdekana	√	√	√
9	2 H Tetrasol 5 metil As. Oksalat	-	√	-
10	2 as. Tiopeniasetat	-	-	√
11	2H tetrasol Allyldimetil silen	√	-	-
12	2,6 Di isopropil naftalena	√	-	-
13	Trimetilsilil sulfanil asetat 2 Amino 2 okso as. Asetat 3,4 dimetil fenil	√	-	√
14	etil ester	-	√	√

15	Oktadekana	√	√	√
16	Dodekahidro indaseno oksiren	-	-	√
17	2,4,5,7 tetrasilaoktana	√	√	√
18	Sobutil ester sikloheksimetil as. Sulfat	√	-	√
19	Bromometil sikloheksana	-	√	-
20	Eikosana	√	√	√
21	Heptana	√	-	-
22	2 metil 3 desen 5 non	-	-	√
23	Oktametil Trisiloksana	-	-	√
24	9 As. Oktadekanoat	√	-	-
25	Heneikosana	-	√	-
26	Trikosana	-	√	-
27	As. Bensen asetat	√	-	-
28	Tetrakosana	√	√	√
29	Sikloheksana	√	-	-
30	Pentakosana	√	√	-
31	1,2 As. Bensendikarboksilat	√	-	-
32	Heksakosana	√	√	√
33	Siklotetrakosana	√	-	-
34	Tetrakosametil siklododekasiloksana	-	-	√
35	Heksasiloksan	√	-	-
36	Heptakosana	-	√	-
37	Nonadekana	-	-	√
38	Oktadekametil siklononasiloksana	-	-	√
39	Oktakosana	√	√	-
40	22 tetrakosahekesena	√	-	-
41	Dioksi Selidoniol	√	-	√
42	1,1,3,3,5,5,7,7,9,9,11,11,13,13,15,15 Heksadekametil oktasiloksana	-	√	√
43	5 triasin 2 etoksi 4,6 di (4 morpholinil)	√	-	-
44	3,5,7 trihidroksi4H-1-Benzopiran-4-non	-	√	-
45	1,2 dihidrometil 2okso 3 karbonitril piridin	-	√	-
46	11 metilnonakosana triakontana	-	-	√
47	Triankontana	√	-	-
48	(2 naftil)etil] sikloheksanon	√	-	-
49	13 tetradesenil asetat	-	√	-
50	Nonakosana	√	-	-
51	3 formil 10 metil fenotiasin	√	-	-
52	2 dipropenil tripensilena 1,4 dikarboksilat	√	-	-

53	6 H bensosiklohepta pirasol pirimidin	√	-	-
54	1,1,1,3,5,5,5 heptametiltrisiloksana	-	-	√
55	Dotriakontana	√	-	-
56	Compesterol	√	-	-
57	Stigmasterol 22,23 dihidro gamma sitosterol	√	-	-
58	1 bromo triacontana	-	√	-
59	dihidro 4 okso etil ester	√	-	-

In Terasa Village honey samples're taken from 3 different points (M1, M2 and M3) resulted from extraction using n-hexane solvent obtained 59 volatile compounds classified into hydrocarbon compounds, aldehydes, ketones, alcohols, esters, bensenoid and carboxylic acids. Of the three samples of honey were identified volatile compounds contained in the three types of samples are tetradecana, heptasiloksana, hexadecane, octadecane, tetrasilaoktana, eikosana, tetrakosana and heksakosana.

Extracted volatile compounds of honey using n-hexane solvent obtained from three different points are shown in the following chromatogram fraction:

The characteristics of honey Terasa village compared to honey from other regions namely compounds Tetrasilaoktana (Table 2) which is only found in honey Terasa Village and is not found in other types of honey.

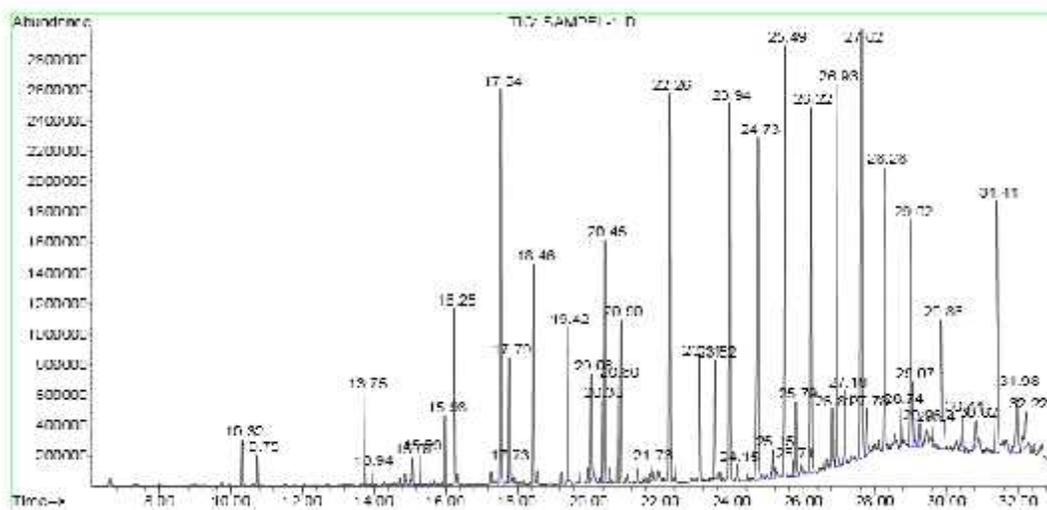


Figure 1. Chromatogram Sample M1

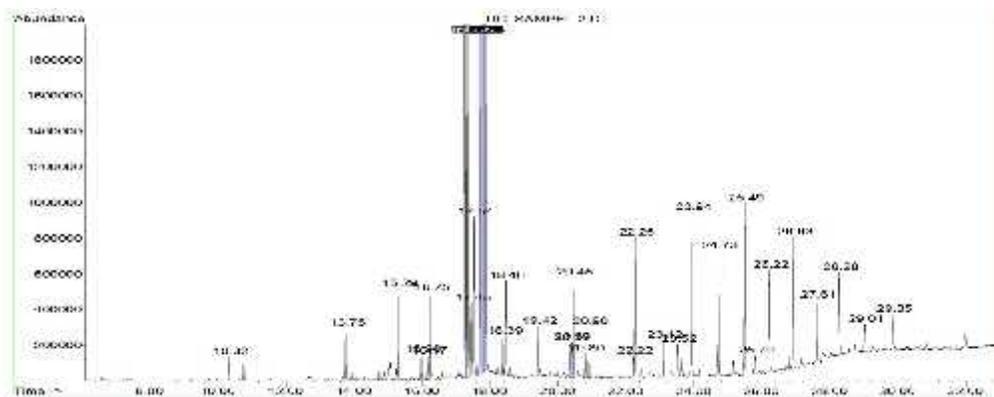


Figure 2. Chromatogram Sample M2

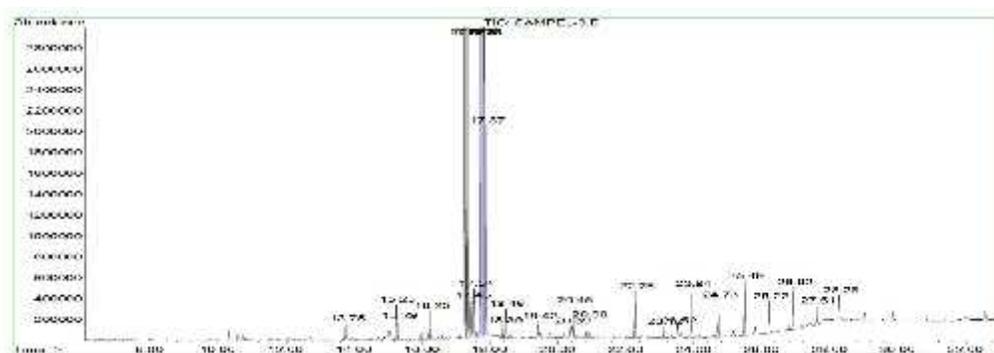


Figure 3. Chromatogram Sample M3

Table 2. Characteristics of Tasted Village Honey

Terasa Village Honey	Tualang Malaysia Honey	Asphodel Kroasia Honey	Afrika Honey	Trigona Sul sel Honey	Bone Sul Sel Honey	Palestina Honey	Mallawa Honey
Tetradekana	-	√	√	√	-	-	√
Heksadekana	-	-	-	-	-	-	√
Heptasiloksan	-	-	-	-	-	-	√
Oktadekena	-	-	-	-	√	-	√
Tetrasilaoktana	-	-	-	-	-	-	-
Eikosana	√	√	-	√	√	√	-
Tetrakosana	-	-	-	-	-	-	√
Heksakosana	-	-	-	-	-	-	√

4. CONCLUSIONS

From the research of honey Terasa village, can be concluded that there are 59

types of volatile compounds that can be classified into hydrocarbons, aldehydes, ketones, alcohols, esters, bensenoid and

carboxylic acids. Of 59 types of volatile compounds there are 8 compounds that are in the three types of samples of tetradecana, heptasiloksana, hexadecane, octadekana, tetrasilaoktana, eikosana, tetrakosana and heksakosana. Tetrasilaoktana is a typical compound of honey Terasa village because the compound is not found in honey in other areas.

REFERENCES

- [1] Astuti, 2014. *Kajian Senyawa Volatil Madu Trigona Sulawesi selatan sebagai Antimikroba*. Tesis tidak diterbitkan. Makassar. Program Pascasarjana Fakultas Kesehatan Masyarakat Universitas Hasanuddin.
- [2] Alvarez-Saures *et al.*, 2009. Contribution of Honey in Nutrition and Human Health. *Mediterr J Nutr Metab* (2010) 3:15–23
- [3] Badge *et al.*, 2013. Therapeutic and Nutritional Values of Honey (Madhu). *International Research Journal of Pharmacy*, 4 (3) : 19-22
- [4] Barra, M. P. G., Ponce-Diaz., Venegas-Gallegos, C, 2010. Volatile Compounds In Honey Produced In The Central Valley Of Nuble Province, Chile. *Chilean J. Agric Res* 70(1): 75-84
- [5] Montenegro *et al.*, 2009. Analysis of Volatile Compounds in Three Unifloral Native Chilean Honeys. *Oyton* (78) : 61-65.
- [6] Novandra, A., 2013, *Peluang Pasar Produk Perlebahan Indonesia*, Balai penelitian teknologi hasil hutan bukan kayu, Jakarta.
- [7] Odeh *et al.*, 2007. A Variety Of Volatile Compounds As Markers In Palestinian Honey from *Thymus Capitatus*, *Thymelaeae Hirsuta*, And *Tolpis Virgata*. *Chemistry* 101 (2007) 1393-1397
- [8] Ratnayani *et al.*, 2008. Penentua Kadar Glukosa dan Fruktosa pada Madu Randu dan Madu Klengkeng dengan Metode Kromatografi Cair Kinerja Tinggi. *Jurnal Kimia* 2 (2) : 77-86
- [9] Suarez, 2010. Contribution Of Honey In Nutrition And Human Health: A Review. *Mediterr J. Nutr. Metab* (2010) 3:15-23.
- [10] Sukmawati, Alfian Noor, Firdaus 2016. Analysis of Volatile Organic Compound of Mallow Honey. *Marine Chimica Acta, Vol 12 (2):52-58*
- [11] Sibel Silici, 2011. Determination of Volatile Compounds of Pine Honeys. *Tubitak Turk J Biol* 35: 641-645
- [12] Syazana *et al.*, 2012. Analysis of Volatile Compounds of Malaysian Tualang (*Koompassia Excelsa*) Honey Using Gas Chromatography Mass Spectrometry. *Afr J Tradit Complement Altern Med* Vol 10 (2)
- [13] Tadeuz *et al.*, 2006. Identification of Honey Volatile Components by Solid Phase Microextraction (SPME) and Gas Chromatography / Mass Spectrometry (GC/MS). *Journal of Apicultural Science* Vol. 50 (2) : 115-125
- [14] Zakaria, 2013. *Analisis Senyawa Organik Volatil Pada Madu Bone*. Laporan Akhir Penelitian. STAIN Watampone. Bone