

GC-MS ANALYSIS OF BIOACTIVE CONSTITUENTS OF JASMINE FLOWER

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ABSTRACT

Gas Chromatography-Mass Spectrometry (GC-MS) the best technique to identified the compounds of essential oils by comparison of mass spectra data obtained from the sample with that taken from pure commercially available standards injected under the same conditions. To characterize the chemical constituents of Hibiscus Flower using GC-MS, the shade dried flower powder was extracted with methanol by using Microwave-assisted Hydrodistillation (MAHD). The GC-MS analysis provided different peaks determining the presence of ten compounds. These compounds have biological activity namely 2-Phenylthiolane (57.31%), Cyclohexene, 3-ethenyl- (25.91%), Acetaldehyde (12.70%), N-Methylallylamine (9.99%), Propanamide (6.79%) and Phthalic acid, bis (7-methyloctyl) ester (5.21%). From the results, it can be concluded that Jasmine flower extract shows the presence of 10 phytochemicals. The presence of various bioactive compounds justifies the use of the jasmine flower for various ailments by traditional practitioners.

Keywords: Microwave-Assisted Hydrodistillation (MAHD); Gas Chromatography-Mass Spectrometry (GC-MS); Phytochemical constituents; Methanol extract

1.0 INTRODUCTION

Jasmine tree was originated from tropical countries such as Africa, Australia, and Southeast Asia. Currently, Jasmine trees are grown all over the world. In the present study, essential oils extracted from local Jasmine flowers in Malaysia such as Melati and Melur were investigated. The essential oil of Jasmine exhibits the essence of flavor and aroma of Jasmine flower. Its properties are dependent on the types of glycerides and hydrocarbon. Essential oils of Jasmine flower have been extensively used as an expectorant (for dry skin), antiseptic, antispasmodic and antidepressant (Mojay, 1999). Also, these essential oils are used to cure depression, exhaustion, sensitive skin, headache, and cough (Lawless, 2013).

These are several species of Jasmine, out of several varieties, Jasmine sambac, Jasmine auriculatum, and Jasmine grandiflorum are used industrially for essential oil production (Green & Miller, 2009). In this study, Jasmine grown in Malaysia was explored.

In the extraction of Jasmine oil, different extraction methods had been employed which include hydrodistillation, solvent extraction, and maceration. Nevertheless, the methods had been reported to suffer some drawbacks which include lower yield of oil (Reverchon, 1997; Zizovic, Stamenić, Orlović, & Skala, 2007). However, to an outcome this drawback, Microwave-Assisted Hydrodistillation (MAHD) technique was employed for recovering oil from Jasmine flower.

Microwave-Assisted Hydrodistillation (MAHD) is one of the popular techniques used in extracting biological compounds because it offers the solvent selectivity. Also, it is able to shorten the extraction time and control the heating process. MAHD is environmentally friendly as it emits less CO₂ (Lucchesi, Chemat, & Smadja, 2004) and it is more efficient than the conventional steam distillation process (Farhat, Ginies, Romdhane, & Chemat, 2009). This technique has been used to extract many bioactive components from plants (Golmakani & Rezaei, 2008). In general, its efficiency is mainly dependent on the dielectric constants of solvent and sample (Brachet, Christen, & Veuthey, 2002). Conventional techniques used to extract bioactive compounds are time-consuming and they are unable to control the heating process. Also, the analysis of constituents in plant material is restricted by extraction step (Mandal, Mohan, & Hemalatha, 2007).

Thus, this study investigated the extraction of oil from Jasmine flower using Microwave-Assisted Hydrodistillation (MAHD) technique and the chemical compositions of the extracted oil at the optimal conditions were examined using gas chromatography-mass spectrometry (GC-MS).

2.0 MATERIALS AND METHODS

2.1 Collection and preparation sample

The fresh Jasmine flowers were purchased from (Nature Flower Enterprise), Pahang, Malaysia in April 2016. The flowers were washed with water in order to remove the impurities. The flowers were dried in an oven operating at 90 °C for 1h to reduce the moisture content to 10.0–12.5%. The dried flowers were then grinded by using (Grinder Ultra Centrifugal Mill ZM 200, Retsch, Germany) in order to increase the area of contact between the solvent and the flower (powdered form). After grinding, the samples were sieved using a mechanical sieve shaker to obtain 80 µm size of jasmine flowers powder. Methanol (Fisher Scientific) was used as solvent and dichloromethane (Fisher Scientific) was used to separate the essential oils from methanol.

2.2 Extraction Method

The microwave oven (Milestone MWS Ethos E Solvent Extraction System: 2.5KW; 230 V-60Hz; 2450 MHz) was modified for the current MAHD operation. Jasmine flowers of 35 g were put in a 1L flask with 280 ml methanol. The flask was heated in the microwave oven. The microwave oven was operated by power 400W for 120 minutes (Rassem, Nour, & Yunus, 2017). The essential oil in the flask evaporated and the vapors were passed to the condenser (Clevenger device). The extract, i.e. mixture of methanol and essential oil was collected and put into the separating funnel with dichloromethane for separation purpose. The yield of the essential oil, i.e. 0.89 % (v/w)

was calculated on a dry weight basis. The yield of oil that obtained was calculated by using Equation (1):

$$\text{Yield of essential oil} = \frac{\text{Amount of essential oil (g)}}{\text{Amount of raw materials (g)}} \quad (1)$$

2.3 GC-MS analysis

The Agilent 5975C Series GC/MSD used in the current analysis employed a fused silica column packed with DB-WAX (30 mm × 0.25 mm ID × 2.5 μm) and 100 % dimethyl poly siloxane. The operating oven temperature was set as 60 °C for 10 min and increased at a rate of 20 °C/min to attain 250 °C (held for 10 min). The helium gas speed 30 cm/s was employed.

2.4 Identification of components

Identification was based on the molecular structure, molecular mass and calculated fragments. Interpretation on mass spectrum GC-MS was conducted using the database of National Institute Standard and Technology having more than 62,000 patterns. The name, molecular weight and structure of the components of the test materials were ascertained. The relative percentage amount of each component was calculated by comparing its average peak area to the total areas. The spectrum of the unknown component was compared with the spectrum of the component stored in the NIST library version (2005).

2.5 Analysis with Fourier transform infrared spectroscopy (FTIR)

Analyses were performed by using Thermo Scientific Nicolet iS5 FT-IR Spectrometer. The spectrum has been recorded for wave numbers ranging between 4000 cm⁻¹ and 400 cm⁻¹ and the functional groups in the essential oil for jasmine flower were identified.

3.0 RESULTS AND DISCUSSION

GC-MS is one of the best techniques to identify the constituents of volatile matter, long chain, branched chain hydrocarbons, alcohols acids, esters etc. The GC-MS analysis of jasmine flower revealed the presence of ten compounds (phytochemical constituents) that could contribute the medicinal quality of the plant. The identification of the phytochemical compounds was confirmed based on the peak area, retention time and molecular formula. Figure 1 shows the results of the present study. The compound prediction was performed based on NIST Database. The identified compounds were 2-Phenylthiolane (57.31%), Cyclohexene, 3-ethenyl- (25.91%), Acetaldehyde (12.70%), N-Methylallylamine (9.99%), Propanamide (6.79%), Phthalic acid, bis(7-methyloctyl) ester (5.21%), 10-Methylnonadecane (2.25%), (Aminomethyl) cyclopropane (0.79%), 1H-Tetrazol-5-amine (0.49%) and 1,2-Benzenedicarboxylic acid diisooctyl ester (0.37%). As shown in Table 1, the Retention Times (RTs) of ten major components are 6.253, 33.029, 40.656, 45.090, 45.587, 50.118, 50.850, 56.001, 58.499 and 60.371, respectively. The phytochemicals identified through GC-MS analysis showed many biological activities relevant to this study are listed in Table 2. The biological activities

listed are based on Dr. Duke's Phytochemical and Ethnobotanical Databases created by Dr. Jim Duke of the Agricultural Research Service/USDA. The functional groups in the essential oil were identified via FT-IR. As can be seen from these spectra, there is an overlap of the absorption spectrum of each of the different components of the oil. This is due to the mixture complexity of volatile oils. The region from 4000 to 400 cm^{-1} is highly informative as the peaks for the characteristic fingerprint of jasmine flowers oil lie within this range. The functional groups present in the essential oil were determined by comparing the vibration frequencies in wave numbers of the sample spectrograph obtained from an FT-IR spectrophotometer with those of an IR correlation chart. As observed from the FT-IR spectrum, the absorption bands or frequencies of 3286.39 cm^{-1} and 2920.65 cm^{-1} show the presence of O-H stretch for phenol and C-C stretch, respectively. Meanwhile, the absorbance band of 1627.08 cm^{-1} reveals the presence of C=O bond for aldehyde. The formation of the peak at 1304.70 cm^{-1} is due to ring stretching. The peak at 1151.75 cm^{-1} corresponds to C-O stretching vibration. The peak at 767.11 cm^{-1} is an attribute of a vibrational characteristic of benzene ring absorption of C=H group. Some important peaks and their representations are reported in Table 3 and Figure 2.

Table 1: Components detected in the plant of methanol extract of Jasmine flowers

No	Compounds	Molecular Formula	MW	RT	Area%
1	Acetaldehyde	C ₂ H ₄ O	44.05	6.253	12.70
2	2-Phenylthiolane	C ₁₀ H ₁₂ S	164.26	33.029	57.31
3	Propanamide	C ₃ H ₇ NO	73.09	40.656	6.79
4	(Aminomethyl)cyclopropane	C ₄ H ₉ N	71.12	45.090	0.74
5	Cyclohexene, 3-ethenyl	C ₈ H ₁₂	108.18	45.587	25.91
6	N-Methylallylamine	C ₄ H ₉ N	71.12	50.118	9.99
7	1H-Tetrazol-5-amine	C ₈ H ₁₂ O	390.55	50.850	0.49
8	1,2-Benzenedicarboxylic acid, diisooctyl ester	C ₂₄ H ₃₈ O ₄	124.18	55.999	0.37
9	10-Methylnonadecane	C ₂₀ H ₄₂	282.54	58.499	2.25
10	Phthalic acid, bis(7-methyloctyl) ester	C ₂₆ H ₄₂ O ₄	418.61	60.371	5.21

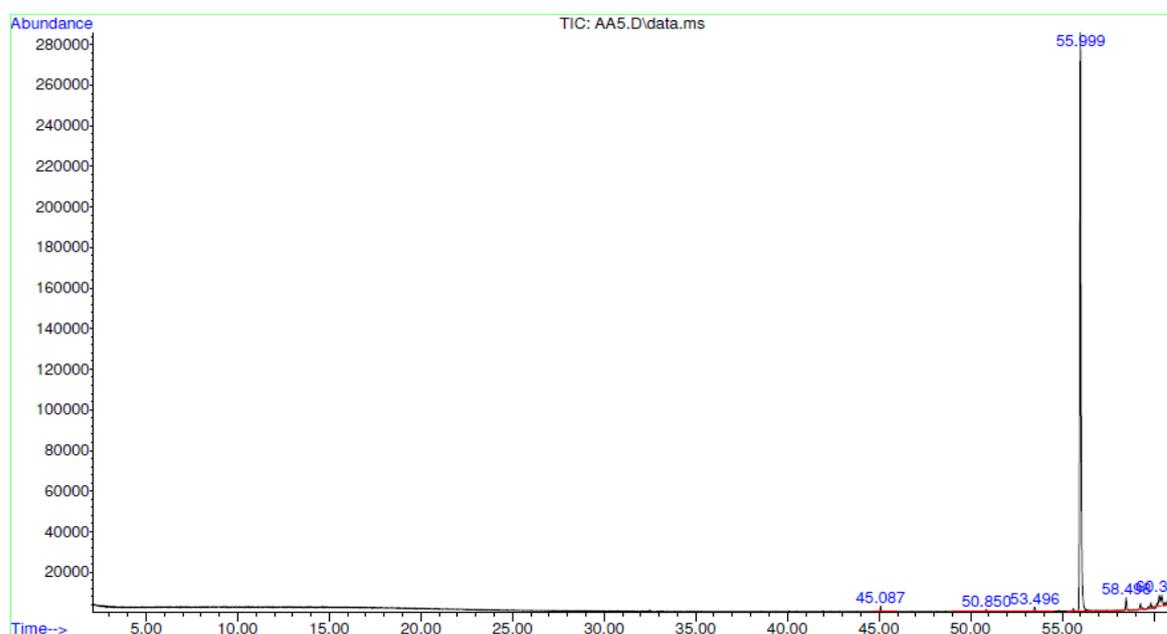


Figure 1: GC-MS Chromatogram of Methanolic extract of *Jasmine flower*

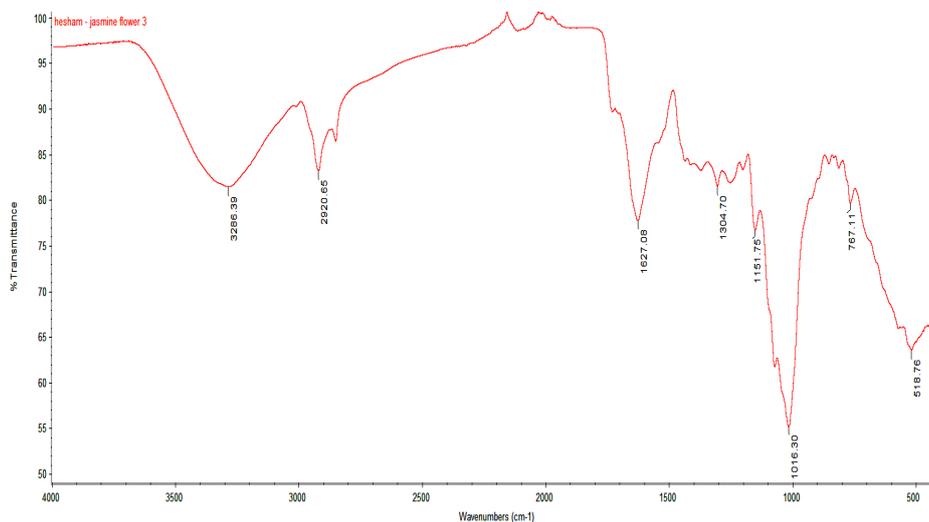
Table 2: Activity of Phyto-Components identified in the methanol extracts of Jasmine flowers.

No	Compounds	Molecular Formula	**Activity
1	Acetaldehyde	CH ₃ CHO	Antioxidant, Cancer preventive.
2	2-Phenylthiolane	C ₁₀ H ₁₂ S	Antioxidant, Hypocholesterolemic, Antimicrobial.
3	Propanamide	C ₃ H ₇ NO	Antioxidant, Pesticide, Cancer preventive.
4	(Aminomethyl)cyclopropane	C ₄ H ₉ N	Antioxidant, Antimicrobial.
5	Cyclohexene, 3-ethenyl	C ₈ H ₁₂	Antioxidant, Antimicrobial.
6	N-Methylallylamine	C ₄ H ₉ N	Antioxidant, Cancer preventive.
7	1H-Tetrazol-5-amine	C ₈ H ₁₂ O	Antioxidant.
8	1,2-Benzenedicarboxylic acid, diisooctyl ester	C ₂₄ H ₃₈ O ₄	Antioxidant, Anemiagenic,
9	10-Methylnonadecane	C ₂₀ H ₄₂	Antioxidant, Cancer preventive Antimicrobial.
10	Phthalic acid, bis(7-methyloctyl) ester	C ₃ H ₆ N ₆	Antioxidant, Cancer preventive.

**Activity source: Dr. Duke's Phytochemical and Ethnobotanical Database

Table 3: FT-IR spectral analysis functional theory calculations studies

NO.	Vibration assignment (ν) (cm^{-1})	Absorption band
1	3286.39	O-H
2	2920.65	C-C stretching
3	1627.08	C=O stretching
4	1304.70	Ring stretching
5	1151.75	C-O stretching
6	1016.30	C-OH deformation vibration
7	767.11	C-H vibration of benzene ring
8	518.76	C=C vibration of benzene ring

**Figure 2:** FT-IR spectrum of essential oil for jasmine flower

4.0 CONCLUSION

The source of many plants (herbs and spices) can often be identified from the peak pattern of the chromatograms obtained directly from headspace analysis. Similarly, unique qualitative and quantitative patterns from a GC analysis will often help identify the source of many alcoholic beverages. The technique of fingerprint could really identify the false herbal products. The construction of chromatographic fingerprints aims at evaluating the quality of Herbal Medicines (Liang, Xie, & Chan, 2004). A total of ten compounds have been identified from the current GC-MS analysis performed on the methanolic extract of Jasmine flowers. The functional groups present in the essential oil were determined by comparing the vibration frequencies in wave numbers of the sample spectrograph obtained from an FT-IR spectrophotometer with those of an IR correlation chart. It was through these active groups that they verified the authenticity of

the vehicles detected through GS-MS. Those components are 2-Phenylthiolane (57.31%), Cyclohexene, 3-ethenyl- (25.91%), Acetaldehyde (12.70%), N-Methylallylamine (9.99%), Propanamide (6.79%), Phthalic acid, bis(7-methyloctyl) ester (5.21%), 10-Methylnonadecane (2.25%), (Aminomethyl) cyclopropane (0.79%), 1H-Tetrazol-5-amine (0.49%) and 1,2-Benzenedicarboxylic acid, diisooctyl ester (0.37%). Therefore, GC-MS method is a direct and fast analytical approach for identification of terpenoids and steroids and only few grams of plant material is required. The importance of the study is due to the biological activity of some of these compounds. The present study, which reveals the presence of components in jasmine flower suggest that the contribution of these compounds on the pharmacological activity should be evaluated. The current GC-MS analysis is useful in extracting active principles of the medicinal plant such as Jasmine. This technique could be applied to other medicinal plants as well.

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