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RESEARCH ARTICLE

Analysis of bioactive compounds for Jasmine flower via Gas chromatography-mass spectrometry (GC-MS)

Hesham Hussein Rassem*, Abdurahman Hamid Nour, Rosli Mohamed Yunus

Faculty of chemical and Natural Resources Engineering, Universiti Malaysia Pahang (UMP), Malaysia

* *Corresponding author: hesham_rassem@yahoo.com*

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Abstract

The conventional methods for bioactive compounds of jasmine flower is disadvantageous from both economic and environment perspectives. The current study used microwave-assisted hydro-distillation (MAHD) method to extract bioactive compounds of jasmine flower. Gas Chromatography-Mass Spectrometry (GC-MS) was used for chemical constituent's analysis. The GC-MS analysis revealed different peaks of ten compounds. Results have shown the main compounds were 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%). Functional group analysis was carried out on the volatile oil obtained through MAHD by using Fourier Transform Infrared Spectroscopy (FTIR), while SEM was used for surface morphologies of untreated dried powdered Jasmine flower (i.e. raw powdered jasmine flower without soaking into methanol solution prior to extraction) and pre-treated powdered Jasmine flower (soaked into methanol solution for 1 hr). Results have shown the capability of MAHD in extraction of bioactive compounds in a short period of time.

Keywords: Essential oil, jasmine flower, microwave-assisted hydro-distillation (MAHD), gas chromatography-mass spectroscopy (GC-MS)

INTRODUCTION

The aromatic blending of liquids, obtained via distillation of plant materials, is known as essential oil [1]. The essential oils are regarded in factory as volatile oils with aromatic components that are made up of chemical compounds such as esters, alcohols, aldehydes, ketones, hydrocarbons, and phenols. These components are the major components of essential oil [2]. Essential oils of different plants are available commercially in the market [3]. Numerous studies have demonstrated the effectiveness of essential oil in biological activity even at low dosage [4, 5].

Medicinal plants consisting of bio-compounds have been extensively used in the socio-cultural activities in Malaysia and other countries. Bio-compounds extracted from plants play a vital role in primary healthcare activities of many developing nations [6]. Various natural constituents can be extracted from various parts of a plant such as flower, bark, seed, root, leave, etc [7]. The discovery of active compounds from plants has led to the development of new medicinal drugs for treating diseases such as cancer [8] and Alzheimer [9].

Active compounds exhibit antibacterial, antioxidant and antifungal properties as well. The recent quantification and identification methods for active constituents in plants could be used for proper standardization of herbal and its formulations. GC/MS is the best method to detect the bioactive compounds of hydrocarbons (long chain), ethers, acids, esters, alcohols, etc. [10].

Jasmine tree was originated from tropical countries such as Africa, Australia and Southeast Asia (Fig. 1). 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 expectorant (for dry skin), antiseptic, antispasmodic and antidepressant. Also, these essential oils are used to cure depression, exhaustion, sensitive skin, headache and cough.

Fig. 1 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 CO_2 [11] and it is more efficient than the conventional steam distillation process [12, 13]. This technique has been used to extract many bioactive components from plants. In general, its efficiency is mainly dependent on the dielectric constants of solvent and sample [14]. 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 [15]. Few references have reported the extraction of essential oils from medicinal plants via microwave ovens [16].Thus, microwave-assisted hydro-distillation (MAHD) of significant plant materials is explored in this research.

EXPERIMENTAL

Materials

The fresh Jasmine flowers were purchased from Kuantan, 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 0C for 1h. The dried flowers were then grinded in order to increase the contact area between the solvent and the flower (powdered form). Methanol (Fisher Scientific, Hampton, United States) was used as solvent and dichloromethane (Fisher Scientific, Hampton, United States) was used to separate the essential oils from methanol.

Microwave-assisted Hydro-distillation (MADH)

The microwave oven (Milestone MWS Ethos E Solvent Extraction System: 2.5KW; 230 V-60Hz; 2450 MHz) (see Fig. 2) was modified for the current MAHD operation. Jasmine flowers of 35g 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 [17]. 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.

Fig. 2 Microwave-assisted hydro-distillation.

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

The Agilent 5975C Series GC/MS used in the current analysis employed a fused silica column packed with DB-WAX (30 m × 0.25 mm ID × 2.5μm) and 100% dimethyl poly siloxane. The operating oven temperature was set as 60 ^{0}C for 10 min and increased at a rate of 20^{0}C/min to attain 250^{0}C (held for 10 min). The helium gas of speed 30 cm/s was employed. The compounds were identified by comparison of mass spectra data obtained from the sample with that taken from pure commercially available standards injected under the same conditions. By using the database of National Institute Standard and Technology (NIST), the overall spectrum was interpreted and the spectrum components were analyzed through the NIST library.

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 wavenumbers ranging between 4000 cm^{-1} and 400 cm^{-1}.

Scanning electron microscopy (SEM) analysis

The morphological changes of Jasmine flowers (powder form) were investigated through SEM analysis. Images were acquired for untreated dried jasmine flowers powder sample and pre-soaked Jasmine flowers powder sample. The essential oils of Jasmine flower were extracted via MAHD after 120 min. These samples were observed by using Tabletop Microscope TM3030 plus. Sputter coating was done in order to eliminate the electrical discharge during SEM analysis. All test samples were examined in high vacuum condition. The voltage values ranging from 5 –15 KV (15 – 60000 magnification) and the analytical working distance of 11.3 mm were employed.

RESULTS AND DISCUSSION

Compositional analysis of chemical compounds present in Jasmines flower oil obtained through MAHD

From the GC-MS analysis, several compounds of the essential oil of Jasmine flowers were detected by mass spectrometry in GC. In Table 2 the comparison of chemical compounds in crude extract (%) of jasmine flower oil obtained through MAHD. In jasmine flowers crude extract, some chemical classes were presented namely: oxygenated monoterpene, sequiterpene hydrocarbon, oxygenated and other oxygenated compounds. 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 Fig. 3, 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. Thus the compounds obtained MAHD are of immense benefit.

Structural analysis of essential oil

The functional groups in the essential oil were identified via FT-IR. 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 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 vibrational characteristic of benzene ring absorption of C=H group. Some important peaks and their representations are reported in Table 2 and Fig. 4.

Morphological changes to jasmine flowers after extraction with MAHD

Due to soaking, the oil glands in the powder may become puffy and widened as shown in Fig. 5a. This could develop smooth and prompt release of oil from the material during extraction. Without soaking, the oil glands would undergo contraction and a higher stress is needed to release its oil. Fig. 5b shows the SEM image of the oil gland after MAHD extraction. There is disruption of the oil gland which can be associated with the nature of heat distribution during MAHD extraction. In MAHD, the irradiated microwave energy, being highly absorbed by methanol (high dielectric properties) is immediately converted into heat energy which is transferred directly to the plant material. This heat energy can, therefore, be localized at the particular part of the plant containing the oil glands for effective and convenient oil release without necessarily causing a damaging rupture to the glands.

Table 1 Chemical compositions for essential oil of jasmine flowers obtained by MADH.

No	Compounds	Molecular Formula	MW	RT	Area%
1	Acetaldehyde	CH_3CHO	44.05	6.253	12.70
2	2-Phenylthiolane	$C_{10}H_{12}S$	164.26	33.029	57.31
3	Propanamide	C_3H_7NO	73.09	40.656	6.79
4	(Aminomethyl)cyclopropane	C_4H_9N	71.12	45.090	0.74
5	Cyclohexene, 3-ethenyl	C_8H_{12}	108.18	45.587	25.91
6	N-Methylallylamine	C_4H_9N	71.12	50.118	9.99
7	1H-Tetrazol-5-amine	$C_8H_{12}O$	390.55	50.850	0.49
8	1,2-Benzenedicarboxylic acid, diisooctyl ester	$C_{24}H_{38}O_4$	124.18	55.99	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

Table 2 FT-IR spectral analysis functional theory calculations studies.

NO.	Vibration assignment (v) (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

Fig. 3 GC/MS chromatogram of the volatile constituents of Jasmin flowers.

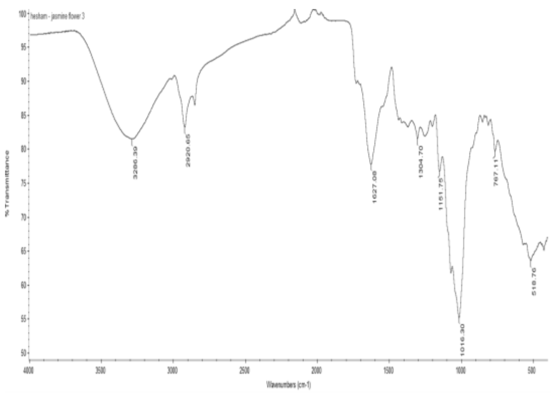

Fig. 4 FT-IR spectrum of essential oil for jasmine flower.

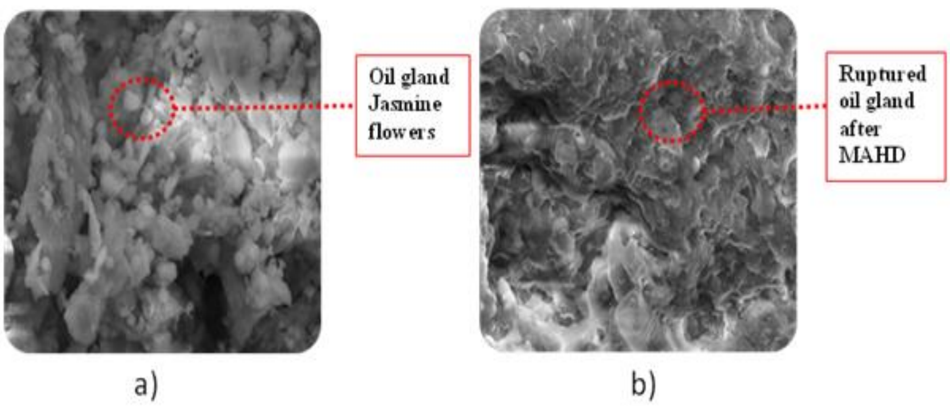

Fig. 5 SEM images of oil cell glands of jasmine flower (a) after pre-treatment (soaking for 60 min), (b) after MAHD extraction (120 min).

CONCLUSIONS

MAHD was used for bioactive compounds extracted from jasmine flower. 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%). These compounds exhibit antioxidant, cancer preventive, pesticide, Hypocholesterolemic, Dermatitigenic and Anemiagenic properties. Antioxidant and antimicrobial properties are exhibited by compounds such as N-Methylallylamine, Propanamide and 1H-Tetrazol-5-amine. The current GC-MS analysis is useful in extracting active principles of medicinal plant such as Jasmine. This technique could be applied for other medicinal plants as well.

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