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Full Length Research Paper

Gas chromatography mass spectrometry (GC-MS) analysis of the hexane extract of the *Syzygium cumini* bark

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The oily fraction of the bark of *Syzygium cumini* of Indian origin, led to the identification of number of ester and hydrocarbon compounds. The hexane eluate of n-hexane extract of *S. cumini* yielded a waxy fraction which was rechromatographed on silica gel column. Its hexane eluate yielded waxy liquids, which were small in amount and was not separated by column chromatography. Hence, they were separated by gas chromatography mass spectrometry (GC-MS) analysis which revealed the presence of 39 compounds. The compounds were identified by comparing their retention time and covate indexes with that of literature and by interpretation of mass spectra. Many of them are used in industry for various applications like perfumes, flavors, deodorants, antiseptic, and pharmaceuticals. The aim of the present study was to analyze the hexane extract of the *S. cumini* bark which was useful for identification of number of hydrocarbons and esters.

Key words: *Syzygium cumini* (bark), esters, hydrocarbons, gas chromatography mass spectrometry.

INTRODUCTION

Syzygium cumini (Hindi- Jamun, Family- Myrtaceae) is an indispensable and beautiful tree. It is reported to be a medicinally important plant. Almost all the parts of the plant, namely, root, leaves, fruits, stem bark, flowers, gum, and young branches are used as medicine, food, fiber and for other miscellaneous purposes, such as fish poison, dye, fodder, utensils, etc (Villaseñor and Lamadrid, 2006). *S. cumini* bark is sweet, sour, acrid, refrigerant, carminative, anthelmintic, febrifuge, constipating, stomachic, and antibacterial. It is useful in, leucorrhoea, stomachalgia, fever, gastropathy, strangury, and dermatopathy (Muruganandan et al., 2002; Khelwal et al., 1994). The bark of *S. cumini* shows the following activities, antidiabetic, diuretic, digestive, astringent, anti-hyperglycemic, antifungal, anti-inflammatory, gingivitis, and controlling blood pressure (Kirtikar and Basu, 1975; Veigas et al., 2007). *S. cumini* is also used in sore throat, bronchitis, asthma, thirst, biliousness, dysentery, blood purifier, ulcers, and diabetes. Recently, it is used in DNA damage in the male albino rates. It is useful in, leucorrhoea, stomachalgia, fever, gastropathy, strangury, and dermatopathy. Bark paste was applied to chest, chronic wounds, uterine disease, douche, piles, tumor, and wounds (Sari et al., 2012). A decoction of the bark is used in the cases of asthma, bronchitis, diarrhea, dysentery, and dyspepsia, and are gargled or used as mouthwash for the astringent effect on mouth ulcerations, spongy gums, and stomatitis (Banerjee et al., 2005). The antibiotic activity of black berry extract has been widely studied and was found useful against a number of microbial agents (Krinshna and Veni, 2012; Sari et al., 2009; Faria et al., 2011). In the present paper, we report the compounds isolated from hexane fraction by gas chromatography mass spectrometry (GC-MS) and their applications.

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Table 1. Esters and hydrocarbons present in hexane extract of *S. cumini* bark.

S/N	Compound	Retention time (min)	Area (%)	Molecular mass	Molecular formula	Application
1	4-propyl-1-benzene propyl ester	13.7	3.13	206	$C_{12}H_{18}O_2$	Pharmaceuticals
2	1, 4-benzene dimethyl ester	14.1	6.50	194	$C_{10}H_{10}O_4$	Pharmaceuticals
3	1, 3-benzene dimethyl ester	14.6	7.77	194	$C_{10}H_{10}O_4$	Pharmaceuticals
4	2-(1-oxoethyl)-1-benzene methyl ester	15.0	3.50	178	$C_{10}H_{10}O_3$	Unknown
5	1, 2-benzene dimethyl ester	15.3	1.87	194	$C_{10}H_{10}O_4$	Pharmaceuticals
6	5-Tridecanene	15.5	0.46	182	$C_{13}H_{26}$	Piping material
7	2-(1-oxopropyl)-1-benzoic acid	16.6	1.04	178	$C_{10}H_{10}O_3$	Unknown
8	2-(1-oxoethyl)-1-benzoic acid	16.7	2.88	164	$C_9H_8O_3$	Unknown
9	1, 2-benzene diethyl ester	16.8	3.05	222	$C_{12}H_{14}O_4$	Such as high tensile strength, impact resistance, resistance to heat and to UV irradiation, as well as electrical properties.
10	2-ethyl-1-benzene ethyl ester	16.9	0.58	178	$C_{11}H_{14}O_2$	Laboratory uses
11	4-methyl-1-benzene ethyl ester	17.0	1.39	164	$C_{10}H_{12}O_2$	Laboratory uses
12	Decane	17.9	0.73	142	$C_{10}H_{22}$	Petroleum jelly, petrolatum, white petrolatum or soft paraffin
13	5-Nonanone	19.1	0.86	142	$C_9H_{18}O$	Medical, domestic, cosmetic uses, and laboratory uses
14	Tetradecane	25.9	0.84	198	$C_{14}H_{30}$	Petroleum jelly, anti-bacterial effect
15	Pentadecane	26.7	1.83	212	$C_{15}H_{32}$	Petroleum jelly, cosmetic skin care
16	Heptadecane	27.5	4.01	240	$C_{17}H_{36}$	Petroleum jelly, ointment
17	Pentacosane	28.3	5.57	352	$C_{25}H_{52}$	Petroleum jelly, cosmetic skin care
18	5-teradecanone	29.3	9.76	212	$C_{14}H_{28}O$	Cosmetic uses and laboratory uses
19	Hexadecane	31.4	8.38	226	$C_{16}H_{34}$	Petroleum jelly, cosmetic skin care, personal lubricant
20	5-Nonadecanene	33.7	20.41	266	$C_{19}H_{38}$	Cosmetics, pharmaceuticals
21	Eicosane	36.2	7.93	282	$C_{20}H_{42}$	Petroleum jelly, ointment, cosmetic skin care
22	Heneicosane	39.1	7.51	296	$C_{21}H_{44}$	Petroleum jelly, cosmetic skin care, ointment
23	(6-(1-methyl)-propyl)-undecan-2, 9-di ene	6.837	2.35	208	$C_{15}H_{28}$	Pharmaceuticals,
24	Tetracosane	7.833	4.77	310	$C_{24}H_{50}$	Petroleum jelly, ointment
25	Phenyl hexanyl ketone	7.984	1.23	190	$C_{13}H_{18}O$	Pharmaceuticals
26	6-methyl -1-decanol	8.380	1.57	172	$C_{11}H_{24}O$	Bactericidal effect

Table 1. Contd.

27	Bicyclo [7.2.0] undec-2, 4, 6-triene-2, 4, 11, 11-tetramethyl-8-methylene	8.515	4.62	214	$C_{16}H_{22}$	Unknown
28	Hexanyl pentanoate	8.787	4.09	186	$C_{11}H_{22}O_2$	Perfume, soaps, cosmetics and food packaging
29	5-tetradecanene	9.699	5.06	196	$C_{14}H_{28}$	Flavoring agent, pharmaceuticals
30	1-tridecanol	10.568	6.82	200	$C_{13}H_{28}O$	Bactericidal effect
31	Octadecane	11.409	5.92	254	$C_{18}H_{38}$	Vaseline, skin lotions and cosmetics
32	Hexadecan-5-ol	12.203	3.14	242	$C_{16}H_{34}O$	Bactericidal effect
33	Tetradecan- 5, 8-di-ol	12.968	2.13	230	$C_{14}H_{30}O_2$	Bactericidal effect
34	6-tridecanene pentanoate	17.283	3.87	282	$C_{18}H_{34}O_2$	Soaps, cosmetics, food packaging
35	Butan tridecanoate	17.934	8.32	270	$C_{17}H_{34}O_2$	Soaps, cosmetics
36	5-nonadecanone	18.571	8.92	282	$C_{19}H_{38}O$	Medical, cosmetic uses , food packaging
37	Heneicosan-13-ene- 2-one	19.228	12.76	308	$C_{21}H_{40}O$	Cosmetic uses
38	5-eicosanene	19.979	9.31	280	$C_{20}H_{40}$	Formation of PVP/ eicosene copolymer
39	Tricosane	20.842	15.11	324	$C_{23}H_{48}$	Additive effect, repelled egg-laying by mosquitoes at the concentrations of those compounds found in nature
36	5-nonadecanone	18.571	8.92	282	$C_{19}H_{38}O$	Medical, cosmetic uses , food packaging

MATERIALS AND METHODS

Plant

S. cumini plant material was collected from the nearest area of Ujjain (Madhya Pradesh). A voucher specimen was then deposited at the Herbarium of Vikram University Ujjain. The essential oil was obtained from the fresh bark by steam distillation for 4 h. The species produced yellowish oil with a pleasant odor in 0.30% yield (based on fresh weight).

Extraction

The air dried *S. cumini* (bark) were Soxhlet extracted, successively with n-hexane and was fractionated on silica gel column. The column was eluted with different solvents in their increasing order of polarity, (Table 1).

Vacuum liquid chromatography

A portion (100 g) of hexane extract was subjected to vacuum liquid chromatography (VLC) on silica gel 60 H using gradient of hexane: benzene: acetic acid (6:4:0.1, v/v) as solvent system to obtain two VLC fractions using a rotator evaporator at a maximum temperature of 40℃.

Preparation of trimethylsilyl (TMS) ether derivatives

The aliphatic compounds present in the VLC fractions of the hexane and benzene extract were converted to their trimethylsilyl derivatives. Each fraction was mixed with Tri-Sil reagent (0.1 ml) in glass sealed tubes using an ultrasonic bath for 2 min and then vortexing briefly. The tubes were then incubated at 60℃ for 45 min. Thereafter, the solvent was evaporated under a stream of nitrogen and the TMS ether derivatives were dissolved in 0.2 ml of n-hexane

and benzene respectively, the tubes were sonicated in an ultrasonic bath for 2 min, and was vortexed and centrifuged for 3 min. The n-hexane and benzene layers were transferred to other tubes, avoiding any solid particles, and were analyzed by the GC-MS. After derivatization, the tubes were stored at -20℃ for subsequent analyses within 3 days.

GC-MS

A Hewlett-Packard 5890 Series II Chromatograph equipped with a flame ionization detector (FID) detector and HP-2 fused silica columns (25 m × 0.32 mm, 0.25 µm film thicknesses) was used. The samples, dissolved in hexane, were injected in the split less mode into helium carrier gas. Injector and detector temperatures were maintained at 250℃. The column temperature was programmed from 60 (after 2 min) to 220℃ at 4℃ min $^{-1}$, and the final temperature was held for 20 min. Peak areas and retention times were measured by electronic integration of computer. The relative amounts of individual components are based on the peak areas obtained, without FID response factor correction.

GC-MS analyses were carried out on a Hewlett-Packard 5970A mass selective detector (MSD), directly coupled to HP 5790A gas chromatograph. A 26 m × 0.22 mm column, coated with 0.13 µm of CP-Sil 5CB was employed, using helium carrier gas. The oven temperature program was 60℃ (3 min), then 5℃ min $^{-1}$ to 250℃ (30 min). Other conditions were the same as described under GC. Electron ionization (EI) mass spectra were acquired over a mass range of 10 to 400 Da at a rate of 2 s^{-1}.

Identification of the compounds

The identification of the compounds present in the VLC fractions of the hexane extract was based on direct comparison of the retention times and mass spectral data with those for standard compounds, and by computer matching with the Wiley 229, Nist 107, 21 Library, as well as by comparison of the fragmentation patterns of the mass spectra with those reported in the literature (Revenkar and Sen, 1978; Santhakumari et al., 2003; Sharma et al., 1989).

RESULTS AND DISCUSSION

The oily fraction of *S. cumini* (bark) was isolated from the separation of hexane extract. The n-hexane extract of *S. cumini* yielded a waxy fraction which was rechromatographed on silica gel column. Hexane eluents yielded waxy liquids in very small amount and was not separated by column chromatography. Hence, they were separated by GC-MS which revealed the presence of thirty nine compounds, respectively. The compounds were identified by comparing their retention time and covate indexes with that of literature and by interpretation of mass spectra. The quantitative estimation of each peak was made by estimating area of the peak by computer, attached by GC-MS instrument. The results of GC-MS analysis are reported in Table 1.

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