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EXTRACTION AND GC-MS ANALYSIS OF ORANGE (*CITRUS SINENSIS)* PEEL OIL

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ABSTRACT: Sweet orange (*Citrus sinensis* L.) is the most commonly grown tree fruit in Maharashtra. Orange peel is considered as a waste but can be used for the extraction of essential oil which has many applications ranging from food flavouring agent, insect repellent to cosmetics. In the present study an attempt was made to extract the oil from oranges, by steam distillation. In order to study the compositions of the oil extracted constituents were analyzed by GC-MS method. In all 15 compounds were detected of which D- limonene was found as a dominant contributor

KEYWORDS: D- limonene, GC-MS, Orange Peel, Steam Distillation

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1. INTRODUCTION

Sweet orange (Citrus sinensis) is the most commonly cultivated fruit tree in various districts Maharashtra. Orange trees are widely cultivated for its taste and natural value, which is peeled, cut, eaten whole or processed to extract orange juice and also for the fragrant peel. Orange essential oil is produced by cells within the rind of an orange fruit. The main components of this oil are Alpha Pinene, Citronellial, Geranial, Sabinene, Myrcene, Lirnonene, and Neral. Orange oil is a mixture of over a hundred compounds that can be approximated into three fractions: terpene hydrocarbons, oxygenated compounds nonvolatile compounds. The terpene fraction can constitute from 50 to more than 90% of the oil. Limonene gives citrus fruit their familiar aroma, is therefore used in

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perfume household cleaners for its fragrance. It an effective, environmentally friendly relatively safe solvent, which makes it an active ingredient of choice in many applications, such as adhesive stain removers, cleaners of various sorts strippers ((Akhilesh Khushwaha et. al. 2012, Ngele. et. al.2014). Limonene is also highly useful in agriculture. There are various methods of oil extraction. Steam distillation is a special type of distillation or a separation process for temperature sensitive materials like oils, resins, hydrocarbons, etc. which is insoluble in water may decompose at their boiling point. The fundamental nature of steam distillation is that it enables a compound or mixture of compounds to be distilled at a temperature substantially below that of the boiling point(s) of the individual constituent. Essential Oil contains components with boiling points up to 200°C or higher temperatures. In the presence of steam or boiling water, however, these substances are volatilized at a temperature close to 100°C, at atmospheric pressure (Mercy Nisha Pauline et. al.2015).The present work carried out on separation of essential oil from orange peel by steam distillation method. The compositions of the extracted constituents were analyzed using GC-MS.

2. MATERIAL AND METHODS

Experiment Setup: The present study was carried out in a distillation apparatus consist of a round bottom flask of 250ml which holds raw material. The top flask is connected with condenser through the connecter. The product is collected in conical flask. 1 kg of orange fruits was washed and dried by cloth. Rind was carefully removed using slicer. The samples were checked to ensure that none of the white flesh under the rind was included in the sample. In all 100 gm of the sample was obtained from the entire process which was used for the extraction of limonene. The rind samples were placed in distilled water in round bottom flask for steam distillation. The flask was heated at 500c in order to achieve steady distillation, approximately one drop per second of distillate. Limonene was collected along with water. As limonene is insoluble in water it was separated easily by using a pipette. The oil layer was carefully removed into a test tube (Fig. 5.)

GC-MS ANALYSIS:-The GC-MS analysis mainly for volatile and semi-volatile compounds was carried out at the Department of Chemistry, Savitribai Phule Pune University. The extract is prepared in Methanol. The experiment was performed by using a Perkin Elmer Clarus 500 gas chromatography equipped with an Elite-5ms capillary column (30nm X 0.25mm X 0.25µm) as well as mass detector turbo mass gold of the company which was operated in EI mode. The carrier's gas flow managed at a rate of 1ml/min. Simultaneously the injector was operated at 250ºC. The oven temperature was programmed as follows; 50ºC at 5ºC/min (5min) to 140ºC at 7ºC/min and to 275ºC (10min).

3. RESULTS AND DISCUSSION

Citrus fruits have been of interest for extraction of essential oil by large number of researchers (Kumar et al., 2011; Kumar et al., 2010) but the peels have been less studied. So the current study targeted the extraction analysis of phytochemicals from the peels. Essential oil was extracted from peels by steam distillation process as suggested by Kumar et al., 2011. Water is used as solvent, Similar solvent have been used earlier by Kumar et al., 2011; Kumar et al., 2010). It was observed that the volume of essential oil extracted from the peels increases with time. This observation is identical with the observations made by V.I. Njoku et al., 2014. It was also noticed that 10 ml essential oil was extracted from 100g of the orange peels of citrus sinensis. This observation is in agreement with the findings of Kamal et al. (2011) while working with species of citrus and reported maximum oil yield from. C. sinensis. GC/MS analysis of Citrus aurantium L. carried out by Zahra Alhani et al., in 2004 and reported 56 compounds. In present work GC-MS chromatogram of the orange peel oil extract of Citrus sinensis displayed 15 peaks indicating the presence of fifteen compounds. The chemical compounds identified in the ethanolic peel oil extract of Citrus sinensis presented in Table 1. GC-MS analysis discovered that the presence of The 15 compounds mainly 5,5,10,10-Tetrachlorotricyclo7.1.0.0(4,6)] decane, Butane, 1-(2,2-dichloro-3,3-dimethylcyclopropyl)- Pentane, 3-Chloro-2-nitrobenzyl alcohol, 1-Oxaspiro[2.5]octan-4-one, D-Limonene, Cyclohexanol, beta.-Terpinyl acetate, gamma.-Terpinene, 4-Terpinenyl acetate. The GC-MS analyses discovered that the orange peel oil extract is mainly composed of terpene hydrocarbons, oxygenated compounds nonvolatile compounds (Chede P.S.et al, 2013). The terpene fraction can constitute from 50 to more than 95% of the oil. Citrus species peels normally contain more than 70% limonene (Dugo G.et al, 2002). In present study 65% of limonene detected. The major component of the oil is D-limonene and probably the antibacterial and antifungal property of the oils. (Wahab O. Okunowo et.al. 2013). It an effective, environmentally friendly, relatively safe solvent, which makes it an active ingredient of choice in many applications, such as adhesive stain removers, cleaners of various sorts strippers. Limonene is also highly useful in agriculture as it is insect repellent. (Akhilesh Khushwaha et. al. 2012, Ngele. et. al.2014)

4. CONCLUSION

Citrus fruits are the widely available and maximum consumed fruits. It has so many medicinal properties and very less toxic effect. In the present study essential oil was extracted by steam distillation method, it is the cheapest and easiest method. Many of the volatile chemicals are detected, which were naturally derived from fruit peel of Citrus sinensis. Were 15 compounds are identified by GCMS analysis of the oil of orange peel. The detected compounds may be helpful in

the study and association of various medicinal properties in the extract of fruit rind. In extracted oil 65% of limonene was detected. It is the major component of the orange peel oil and probably the antibacterial and antifungal property of the oils.

Table – 1: GC-MS analyzed chemical compounds in the Ethanolic extract of peel oil of *Citrus sinensis*

Sr. no.	Compounds present	Molecular Weight	Molecular Formula	CAS Value	Retention time
1.	5,5,10,10-Tetrachlorotricyclo7.1.0.0(4,6)]decane	272	C10H12Cl4	17725-81-4	1504
2.	Butane, 1-(2,2-dichloro-3,3-dimethylcyclopropyl)- $$ 3-Butyl-1,1-dichloro-2,2-dimethylcyclopropane # $$	194	C9H16Cl2	24551-91-5	1104
3.	Pentane, 1,1,1,5-tetrachloro- $$ 1,1,1,5-Tetrachloropentane $$	208	C5H8Cl4	2467-10-9	1131
4.	3-Chloro-2-nitrobenzyl alcohol	187	C7H6ClNO3	77158-86-2	1612
5.	1-Oxaspiro[2.5]octan-4-one, 2-(4-chlorophenyl)-5,5-dimethyl- $$ 2-(4-Chlorophenyl)-5,5-dimethyl-1-oxaspiro[2.5]octan-4-one # $$	264	C15H17ClO2	0-00-0	1983
6.	D-Limonene $$ Cyclohexene, 1-methyl-4-(1-methylethenyl)-, (R)- $$ p-Mentha-1,8-diene, (R)-(+)- $$ (+)-(R)-Limonene $$ (+)-(4R)-Limonene	136	C10H16	5989-27-5	1018
7.	Cyclohexanol, 1-methyl-4-(1-methylethenyl)-, acetate $$ p-Menth-8-en-1-ol, acetate $$ .beta.-Terpinyl acetate $$ 4-Isopropenyl-1-methylcyclohexyl	196	C12H20O2	10198-23-9	1348
8.	gamma.-Terpinene $$ 1,4-Cyclohexadiene,	136	C10H16	99-85-4	998
9.	4-Terpinenyl acetate $$ 3-Cyclohexen-1-ol, 4-methyl-1-(1-methylethyl)-, acetate $$ p-Menth-1-en-4-ol, acetate $$ Terpinene 4-acetate $$ 1-Terpinen-4-ol acetate $$ 4-Terpineol acetate $$	196	C12H20O2	4821-04-9	1327

Fig.1 - Sample preparation

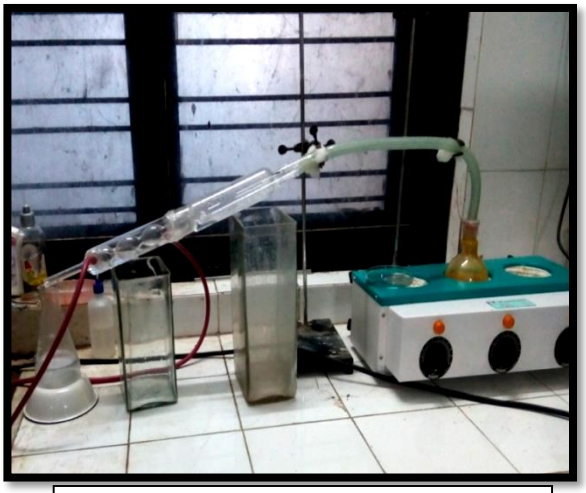

Fig.2 – Distillation unit

Fig.3 – Loaded sample

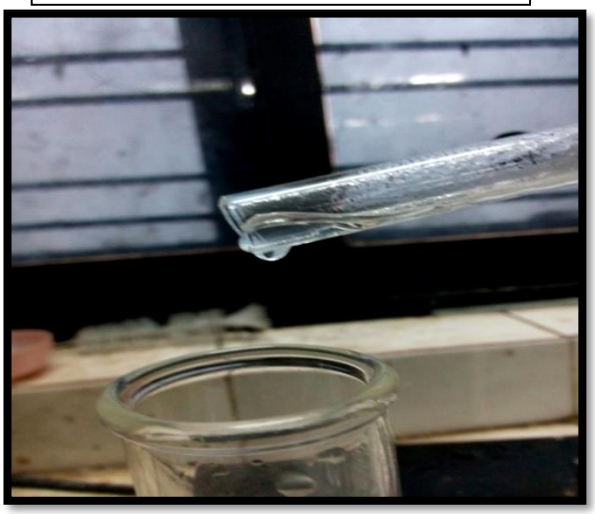

Fig.4 – Extraction Process

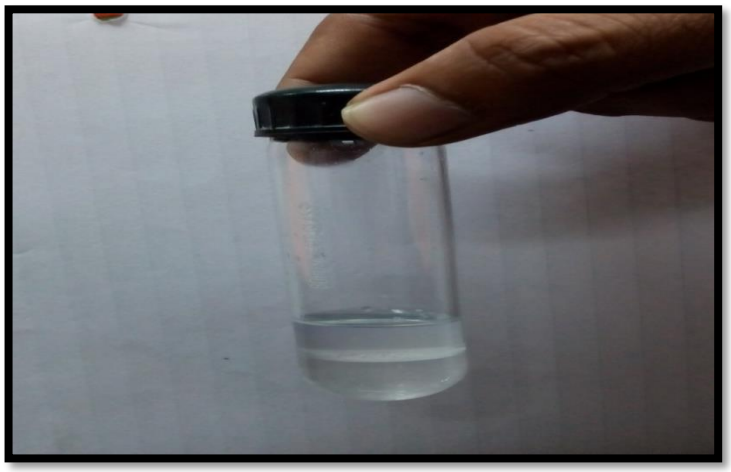

Fig.5 – Extracted oil

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2017 Jan- Feb RJLBPCS 2(5) Page No.45

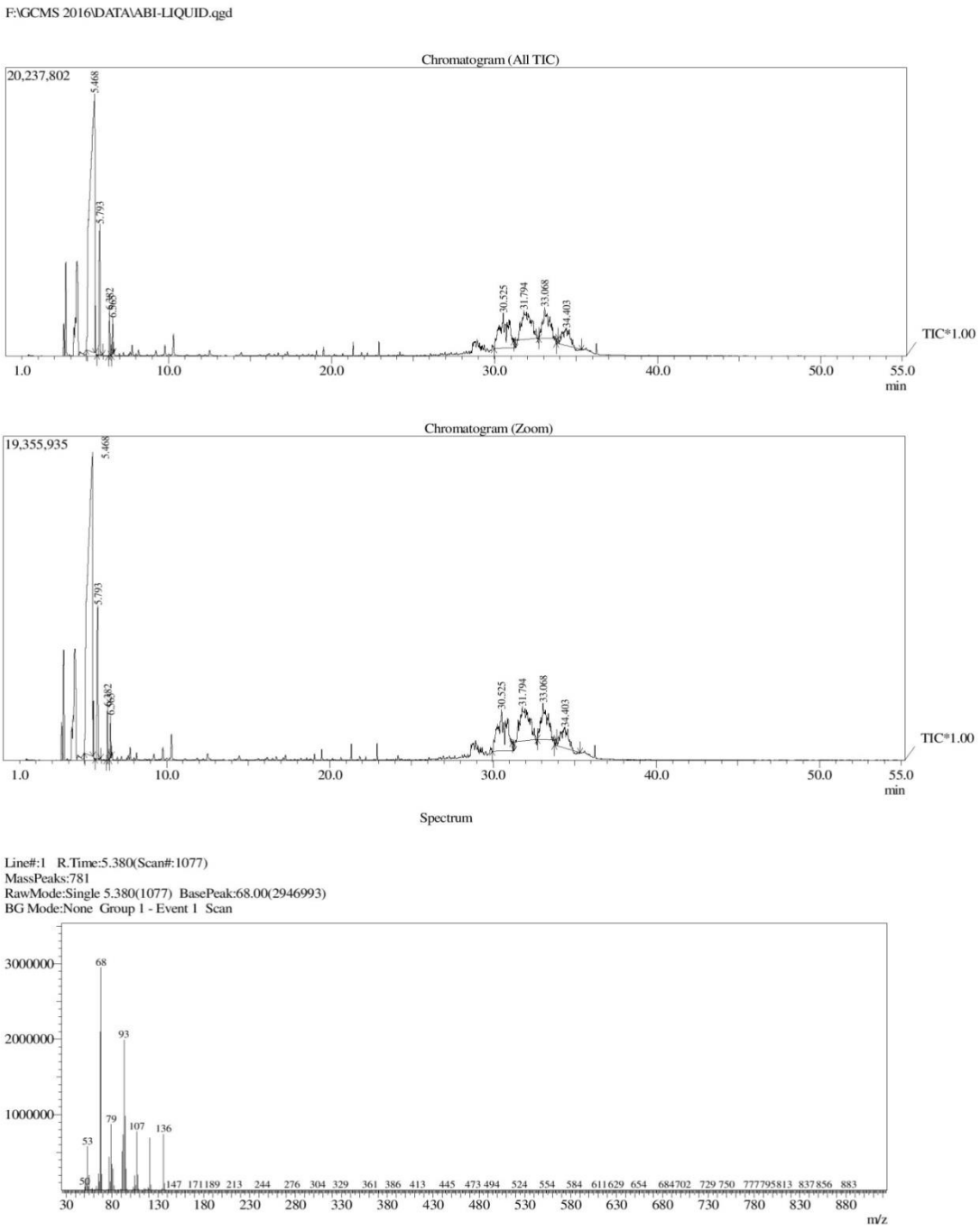

Fig.6- GC-MS Chromatogram of ethanolic extract of orange peel oil.

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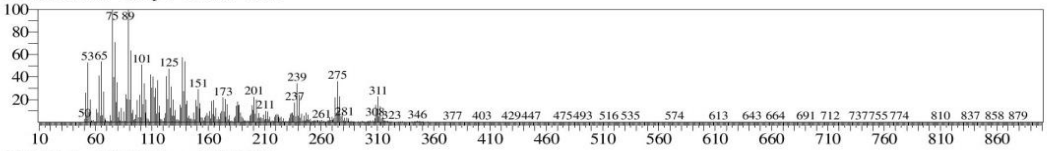

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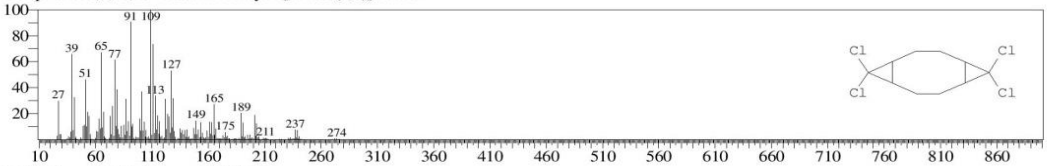

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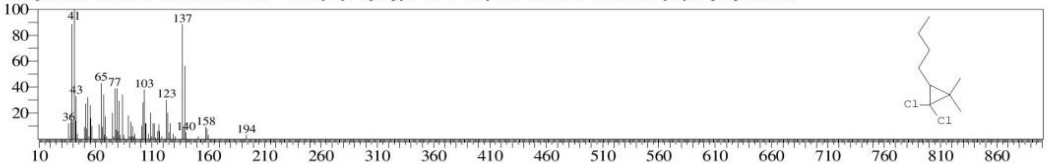

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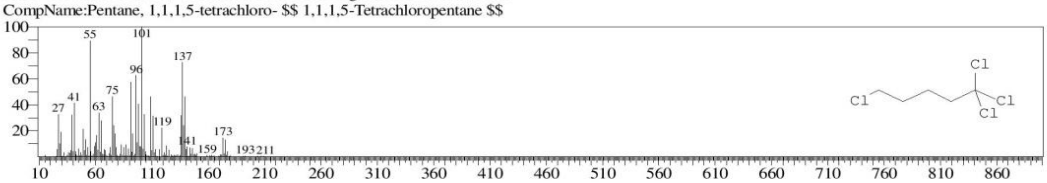

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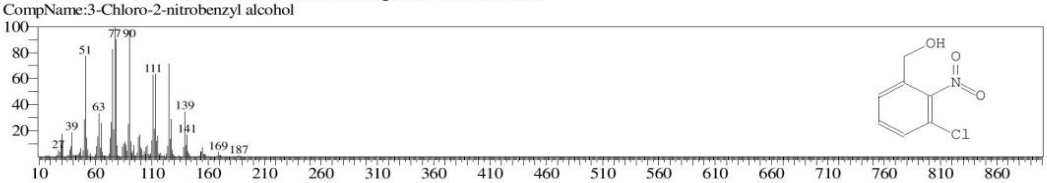

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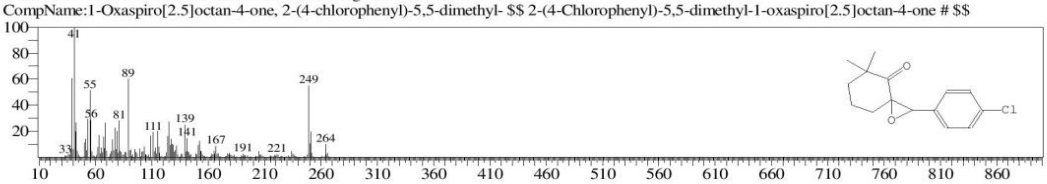

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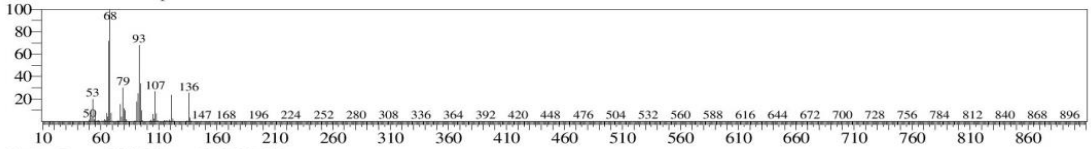

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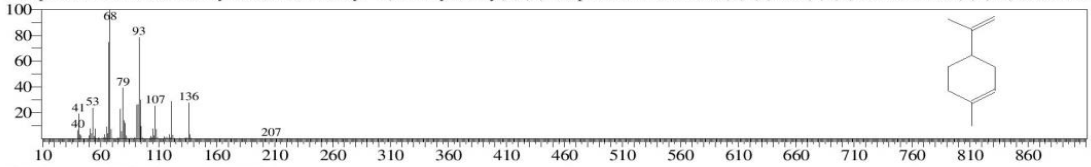

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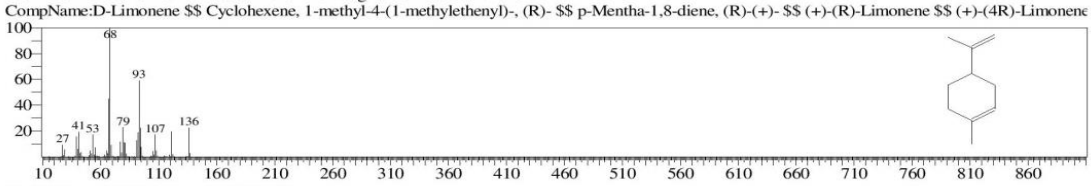

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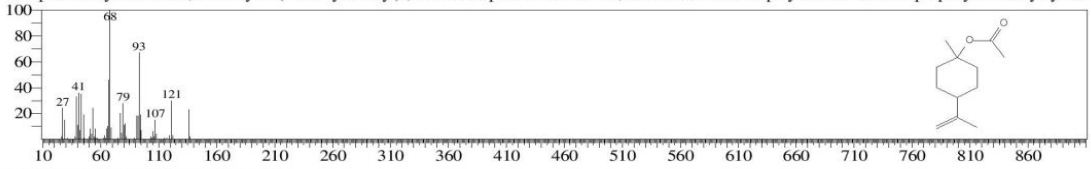

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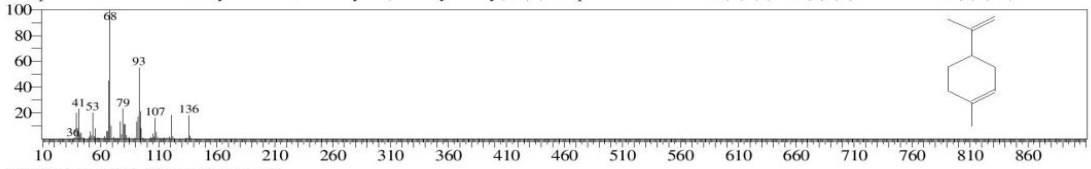

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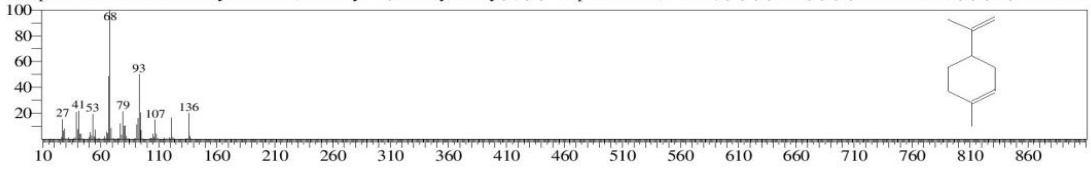

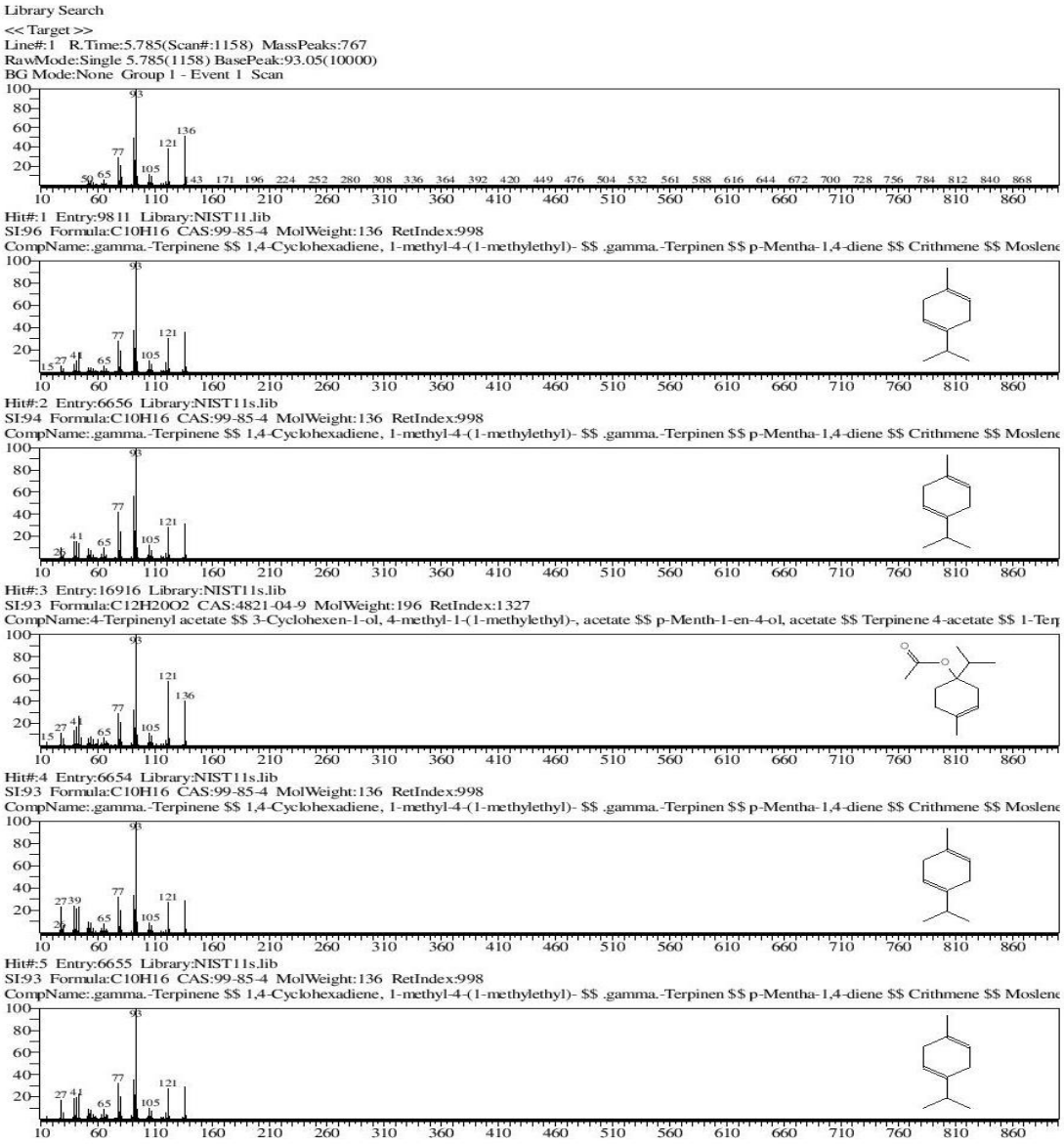

Fig.7- GC-MS plot for orange peel essential oil.

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CONFLICT OF INTEREST

There are no conflicts of interest to disclose.

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