

FTIR AND GC-MS ANALYSIS OF MADURAI MALLI (*JASMINUM SAMBAC* (L.)AIT.)-A GI TAGGED PLANT

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ABSTRACT

Geographical Indications have been given the status of intellectual property since the product gets more value commercially by its mere association with a particular place. GIs helps in the identification of a source of a good which in turn is related with the quality of good. Madurai Malli (*Jasminum sambac* (L.) Ait.) became eligible for registration as a GI as it fits the definition of a geographical indication under Section 2(e) of the GI Act. The colour and the fragrance of the flower stay intact for two days due to its thick petal. Generally "Madurai Malli" is mixed with jasmine from other places while being exported to countries and because of this adulteration of jasmine with poor quality, the demand from other countries is subsequently declined which result in heavy loss of revenue. Now the GI tag would protect the Madurai Malli which will eliminate adulteration when exported. The uniqueness of this Madurai Malli should be established and recorded in terms of other distinguishable characteristics apart from its morphological features. In this work, an FTIR and GC-MS analysis was carried out to get the spectral signature of this plant. This record would support the GI tag of this Madurai Malli to be unique and could be used to discriminate other jasmine plants in future.

Key words: Madurai Malli, IPR. GI tag, FTIR GC MS

INTRODUCTION

The IPRs are legal rights that protect creations and/or inventions resulting from intellectual activity in the industrial, scientific, literary or artistic fields (Savale and Savale, 2016). The most common IPRs include patents, copyrights, marks and trade secrets. Recently, geographical indication (GIs) has emerged as one of the most important instrument of protecting quality, reputation or other character of goods essentially attributable to their geographical origin. Geographical Indications have been given the status of intellectual property since the product gets more value commercially by its mere association with a particular place. GIs helps in the identification of a source of a good which in turn is related with the quality of good (Amikar Parwar, 2013)

Jasminum sambac(L.)Ait., belonging to the family Oleaceae, known as lily jasmine is a scandent or sub erect shrub with young pubescent branches. Madurai Malli, the jasmine flower known for its fragrance and its distinctive petal colour, which originates from the district of Madurai in Tamil Nadu, has been granted the GI (Geographical Indication) tag by the Registrar of Geographical Indications on January 11, 2013. Madurai Malli became eligible for registration as a GI as it fits the definition of a geographical indication under Section 2(e) of the GI Act (Barooah, 2013)

Madurai Malli is woven in six different forms by the local traders selling them. The colour and the fragrance of the flower stay intact for two days due to its thick petal. However, generally "Madurai Malli" is mixed with jasmine from other places while being exported to countries and because of this adulteration of jasmine with poor quality, the demand from other countries is subsequently declined which result in heavy loss of revenue. Now the GI tag would protect the Madurai Malli which will eliminate adulteration when exported (The Hindu, 2013). The uniqueness of this Madurai Malli should be established and recorded in terms of other distinguishable characteristics apart from its morphological features. While GC-MS and FTIR analyses have been made to identify the phytochemical compounds available in the plants (especially medicinal plants), they could also be used as the "finger prints" to identify the plants to certain extent. The valuable medicinal properties of different plants are due to presence of several constituents like saponins, tannins, alkaloids, alkyl phenol, glycol alkaloids, flavonoids, sesquiterpenes, lactones, terpenoids and phenol ethers (Cox, 1994). Within the last two decades, there have been a number of advanced analytical techniques, including FT-IR and GC-MS have been established. They have been proved as powerful tools for identification and determination of phytochemicals (Nithyadevi and Sivakumar, 2015). Hence this record would support the GI tag of this Madurai Malli to be unique and could be used to discriminate other jasmine plants in future.

MATERIALS AND METHODS

Collection and preparation of extract

Required quantity of the *Jasminum sambac* leaves were weighted and pulverized in a mechanical grinder, treated with the ethanol until the sample was fully immersed, incubated overnight and filtered. Phytochemical characterization has been done using FTIR and GC-MS analysis. Plants were collected from the cultivated farm near Madurai, Tamil Nadu.

FT-IR Spectrophotometer Analysis

Fourier Transform Infrared Spectrophotometer (FT-IR) is perhaps the most powerful tool for identifying the types of chemical bonds (functional groups) present in compounds. The wavelength of light absorbed is characteristic of the chemical bond and can be seen in the annotated spectrum. By interpreting the infrared absorption spectrum, the chemical bonds in a molecule can be determined. Dried powder of ethanol extract of powdered leaf was used for FT-IR analysis. 10 mg of the dried extract powder was encapsulated in 100 mg of KBr pellet, in order to prepare translucent sample discs. The sample was loaded in FT-IR spectroscope (SJASCO FT-IR 410 Spectrophotometer), with a scan range from 400 to 4000 cm with a resolution of 2 cm⁻¹.

Gas Chromatography-Mass Spectrometry Analysis

The GC-MS analyses were carried out in a Shimadzu GC-MS-QP 2010 gas chromatograph fitted with a DBI (Methyl phenyl siloxane, 30 m x0.25 mm id.d) capillary column. Helium gas was used as the carrier gas, with a flow rate of 0.7 ml/min. Column oven temperature was raised from 70⁰C to 180⁰C, then isothermal condition was maintained for 5 min in 180⁰; 180-260⁰C at 3⁰C/min, 5 min in 260⁰C; 260-280⁰C at 0.2⁰C/min and finally 5 min in 280⁰C. The injector temperature was 280⁰C and the detector temperature was 290⁰C; volume injected was 1 µL of TMS ether derivatives in *n*-hexane (2%) with a split ratio of 3:0. The MS operating parameters were as follows: ionization potential 70 eV; ion source temperature 200⁰C; quadrupole 100⁰C amu; eV voltage 3000 volts.

The concentrated extract was injected into the GC-MS instrument. The sample was volatilized at the injection port and eluted through a capillary column under increasing temperature. As the sample moved through the column, various components were separated due to their affinity for the stationary phase of the column and could be identified by retention time. The relative percentage amount of each component was calculated by comparing its average peak area to the total areas.

Compound Identification

Components of the ethanolic extracts were identified by comparison of their mass spectra and retention indices with those published in the literature and contained in the NIST '98 MS computer library (Wiley).

RESULTS AND DISCUSSION

FT-IR analysis

In the present study the exploration of phytochemical screening with ethanol extract of *Jasminum sambac* were revealed. FT-IR analysis peak values and the corresponding functional groups of are represented in Figure 1 & Table 1. The FT-IR spectrum confirmed the presence of functional groups such as Amines, Alcohol; Alkane,; Aldehyde, Alkane, Carboxylic Acids; Phosphine; Silane; Alkene, Amides; ; Sulfate; Sulfone; Phosphoramidate, Amine oxide (N-O); Thiocarbonyl, Silane, Anhydrides, Ether, Alcohol Ester, Amines, Sulfoxide, Esters; Esters, Alkane and Esters, Alkane; which are shown with the significant peaks at 3343.15, 2974.24, 2889.01, 2363.57, 2127.43, 1652.80, 1449.43, 1379.85, 1326.11, 1273.30, 1086.45, 1044.54, 879.31 and 802.54.

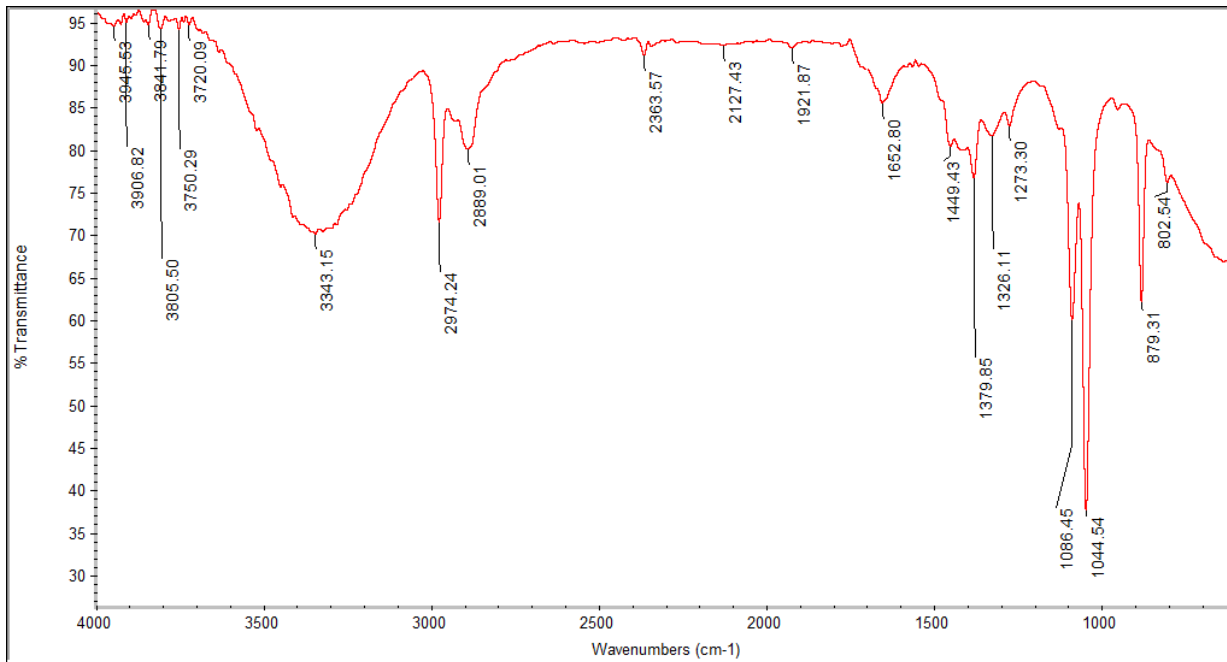


FIGURE 1: FTIR Spectral peak values and functional groups obtained for the ethanolic extracts of *Jasminum sambac*

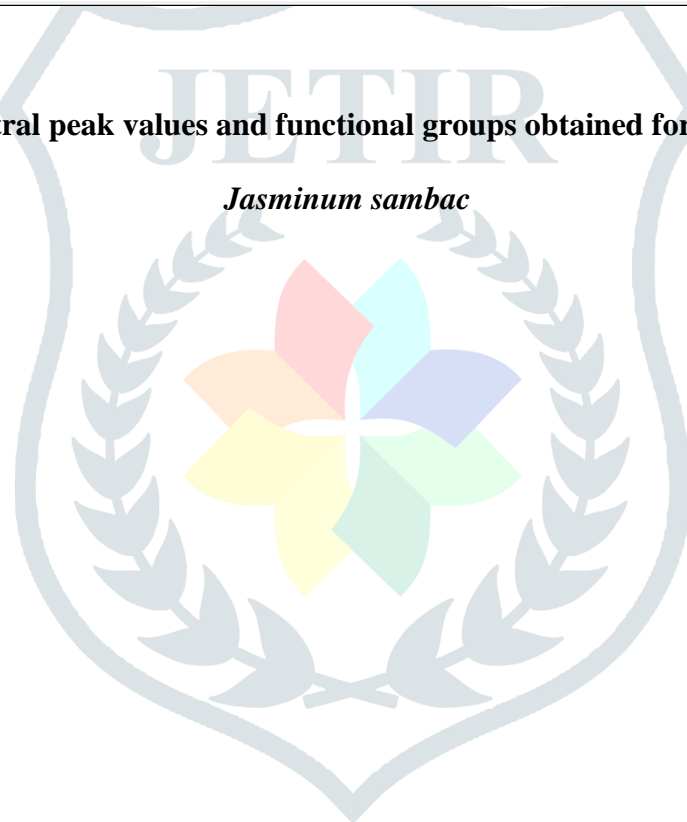


TABLE 1: FTIR Spectral peak values and functional groups obtained for the ethanolic extracts of *Jasmonium sambac*.

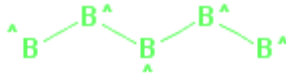
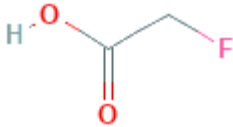
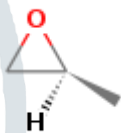

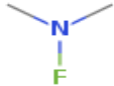

| S. No | Peak value | Functional groups | Class |
|-------|------------|-------------------------------------|--|
| 1. | 3945.53 | Unknown | Unknown |
| 2. | 3906.82 | Unknown | Unknown |
| 3. | 3841.79 | Unknown | Unknown |
| 4. | 3805.50 | Unknown | Unknown |
| 5. | 3750.29 | Unknown | Unknown |
| 6. | 3720.09 | Unknown | Unknown |
| 7. | 3343.15 | N-H, O-H | Amines, Alcohol |
| 8. | 2974.24 | C-H, O-H | Alkane, Carboxylic Acids |
| 9. | 2889.01 | C-H, O-H, C-H | Aldehyde, Alkane, Carboxylic Acids |
| 10. | 2363.57 | P-H | Phosphine |
| 11. | 2127.43 | Si-H | Silane |
| 12. | 1921.87 | Unknown | Unknown |
| 13. | 1652.80 | C=O, C=C | Alkene, Amides |
| 14. | 1449.43 | S=O | Sulfate |
| 15. | 1379.85 | S=O | Sulfate |
| 16. | 1326.11 | S=O | Sulfone |
| 17. | 1273.30 | P=O, aromatic, C-O | Phosphoramidate, Amine oxide (N-O), Carboxylic Acids |
| 18. | 1086.45 | C=S, Si-OR, P-H, C-N, O-C, C-O, C-O | Thiocarbonyl, Silane, Phosphine, Amines, Anhydrides, Ether, Alcohol |
| 19. | 1044.54 | C-O, C-N, S=O, P-OR | Ester, Amines, Sulfoxide, Esters |
| 20. | 879.31 | S-OR, C-C | Esters, Alkane |
| 21. | 802.54 | S-OR, C-C | Esters, Alkane |

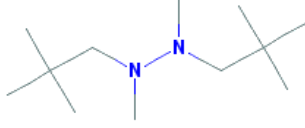

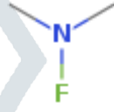
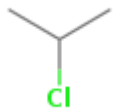
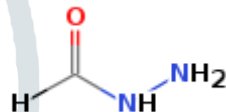

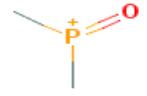

GC-MS analysis


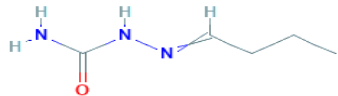

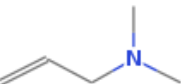
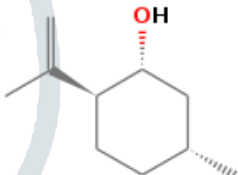
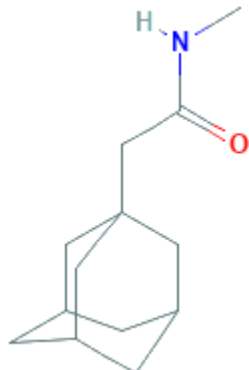
GC-MS spectrum of *Jasminum sambac* showed twenty two different major peaks which indicated the presence of twenty two compounds. The compounds Pentaborane(3.90)Fluoroacetic acid (2.09) ;Oxirane, methyl-, (S)- (1.95);1,4-Pentadiene(8.03); Dimethylfluoroamine (2.41); Ethene, chloro (5.71) ; Hydrazine, 1,2-dimethyl (1.47); 2-Propenenitrile (2.85); Dimethylfluoroamine (1.27) ; Propane, 2-chloro (1.51) ; Formic acid hydrazide (2.23); Phosphine, ethyl (2.42); Dimethylphosphine (3.59); Propanal (1.49) ; Ethanamine, N-ethyl (4.46); Butyraldehyde, semicarbazone (5.50); 1,5-Hexadiyne (4.34); N-Allyl-N,N-dimethylamine (3.61); Isopulegol (14.91) ; N-Methyl-1-adamantaneacetamide (6.46); 1-(3,4-Methylenedioxyphenyl)-2-propanone oxime, methyl ether (3.75); Silicic acid, diethyl bis(trimethylsilyl) ester (16.03). The spectrum profile of GC-MS confirmed the presence of two major components mentioned above with the retention time of 2.702 min, 3.250 min, 3.402 min, 4.271 min, 5.330min,5.983 min, 7.099 min, 7.505 min, 7.798 min, 8.224 min, 8.734 min, 9.311 min, 9.443 min, 10.257 min, 10.692 min, 11.694 min, 12.318 min, 12.564 min, 13.377 min, 14.058, 14.46 4min and 15.088 min respectively (Table 2 & Figure 2).The mass spectrum range of the components was compared with the compounds record in the NIST library and PubChem were characterized and recognized. The names of the identified compounds, molecular weight (MW), along with their retention time (RT) based on capillary column, molecular formula and their concentration (Peak %) are tabulated.

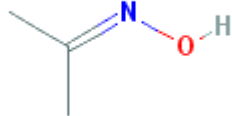
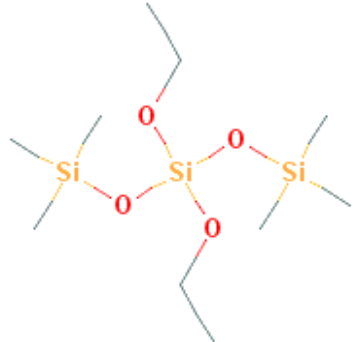
The flowers, leaves and roots of *Jasminum sambac* have been subjected to GC MS analysis by earlier workers also (Elumalai *et al.*, 2010; Krishnaveni and Thaakur, 2012; Zeng *et al.*, 2012). Most of the findings agree with our results as many constituents are commonly available. However it is evident that the spectrum of constituents is not always uniform. Rao and Rout (2003) have analyzed the essential oil of the flowers of *Jasminum sambac* collected from three different locations in India which showed a considerable variation in the composition of phytoconstituents. This current record of FTIR and GC MS will support in the identification of the Madurai Malli and will serve as one of the criteria to eliminate other adulterants.

TABLE:2 GC-MS analysis of ethanol extract of stem of *Jasminumsambac*

| S. No | Retention time | Name of the compound | Molecular formula | Molecular weight | Structure |
|-------|----------------|------------------------|--|------------------|---|
| 1. | 2.702 min | Pentaborane | B ₅ H ₉ or B ₅ | 54.05 g/mol |  |
| 2. | 3.250 min | Fluoroacetic acid | C ₂ H ₃ FO ₂ or CH ₂ FCOOH | 78.042 g/mol |  |
| 3. | 3.402 min | Oxirane, methyl-, (S)- | C ₃ H ₆ O | 58.0791 g/mol |  |
| 4. | 4.271 min | 1,4-Pentadiene | C ₅ H ₈ | 68.119 g/mol |  |
| 5. | 5.330 min | Dimethylfluoroamine | C ₂ H ₆ FN | 63.0741 g/mol |  |
| 6. | 5.983 min | Ethene, chloro | C ₂ H ₃ Cl | 62.498 g/mol |  |

| | | | | | |
|----|------------|-------------------------|-------------------|---------------|---|
| 7. | 7.099 min | Hydrazine, 1,2-dimethyl | $C_{12}H_{28}N_2$ | 200.37 g/mol |  |
| 8. | 7.505 min | 2-Propenenitrile | C_3H_3N | 53.0626 g/mol |  |
| 9. | 7.798 min | Dimethylfluoroamine | C_2H_6FN | 63.0741 g/mol |  |
| 10 | 8.224 min | Propane, 2-chloro | C_3H_7Cl | 78.541 g/mol |  |
| 11 | 8.734 min | Formic acid hydrazide | CH_4N_2O | 60.0553 g/mol |  |
| 12 | 9.311 min | Phosphine, ethyl | C_2H_7P | 62.0507 g/mol |  |
| 13 | 9.443 min | Dimethylphosphine | $C_2H_6OP^+$ | 77.043 g/mol |  |
| 14 | 10.257 min | Propanal | C_3H_6O | 58.0791 g/mol |  |

| | | | | | |
|----|------------|--------------------------------|------------------|----------------|---|
| 15 | 10.692 min | Ethanamine, N-ethyl | $C_4H_{11}N$ | 73.1368 g/mol |  |
| 16 | 11.694 min | Butyraldehyde, semicarbazone | $C_5H_{11}N_3O$ | 129.163 g/mol |  |
| 17 | 12.318 min | 1,5-Hexadiyne | C_6H_6 | 78.1118 g/mol |  |
| 18 | 12.564 min | N-Allyl-N,N-dimethylamine | $C_5H_{11}N$ | 85.1475 g/mol |  |
| 19 | 13.377 min | Isopulegol | $C_{10}H_{18}O$ | 154.2493 g/mol |  |
| 20 | 14.058 min | N-Methyl-1-adamantaneacetamide | $C_{13}H_{21}NO$ | 207.317 g/mol |  |

| | | | | | |
|----|------------|--|-----------------------|---------------|---|
| 21 | 14.464 min | 1-(3,4-Methylenedioxyphenyl)-2-propanone oxime, methyl ether | C_3H_7NO | 73.095 g/mol |  |
| 22 | 15.088 min | Silicic acid, diethyl bis (trimethylsilyl) ester | $C_{10}H_{28}O_4Si_3$ | 296.585 g/mol |  |

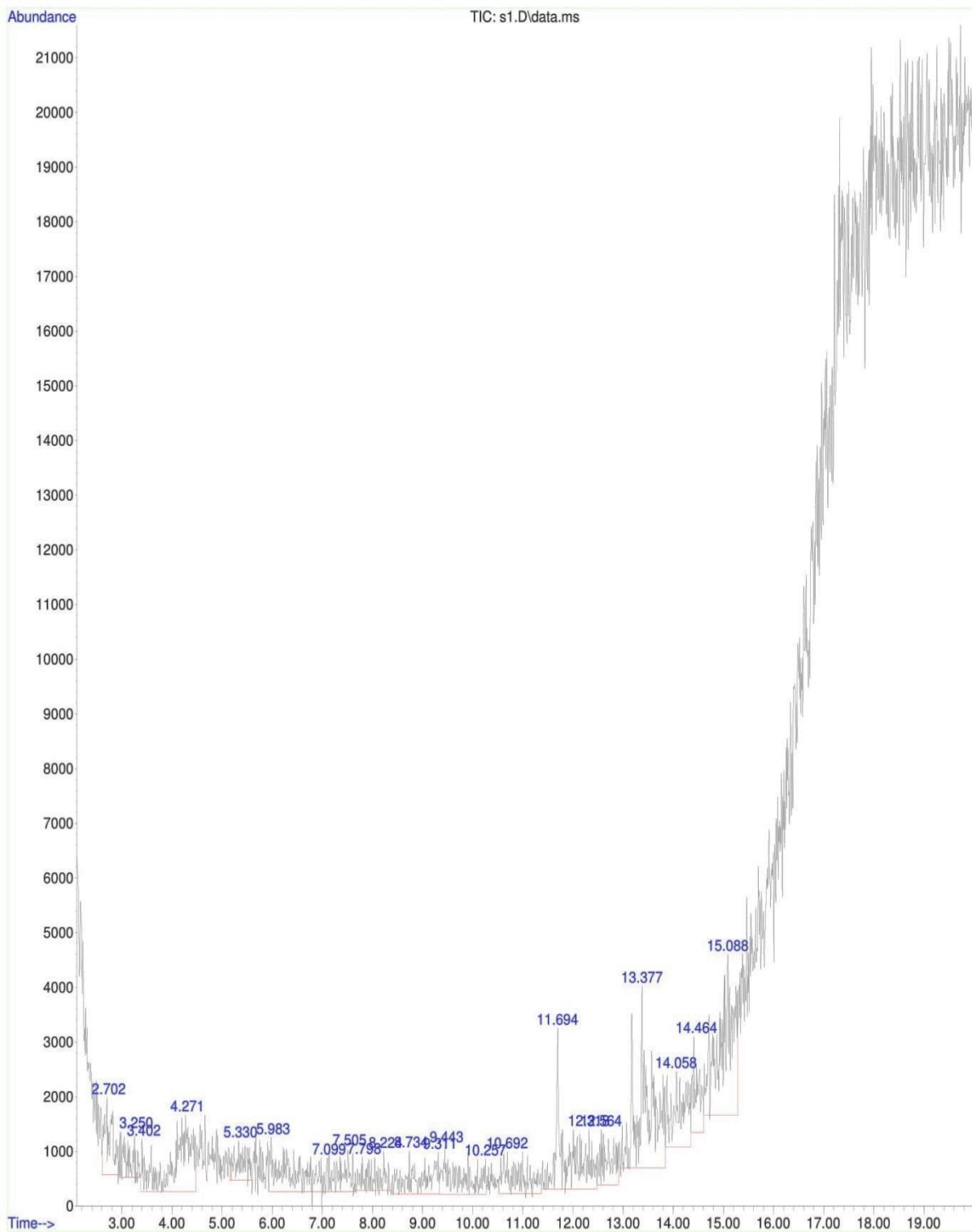


FIGURE: 2 GC-MS analysis of ethanol extract of *Jasminum sambac*

CONCLUSION

A geographical indication is a geographical name signifying that a product originates in a country or a specific locality. Madurai Malli became eligible for registration as a GI as it fits the definition of a geographical indication under Section 2(e) of the GI Act. This protects the Madurai Malli from adulteries and the cultivators and traders are given legal protection under the GI Act against other jasmine flowers are sold as Madurai Malli.

In this work, plants were collected from the cultivated farm near Madurai, Tamil Nadu. The phytochemical characterization has been done using FTIR and GC MS analysis. FTIR showed 21 peak values with 15 functional groups and GC-MS analysis showed twenty two different major peaks which indicated the presence of twenty two phytoconstituents with the ethanolic extract. This will help to protect Madurai Malli to be identified with its GI tag.

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