Fatty Acid Characterization from Flowers of Tulipa Systola Stapf. Using Gas Chromatography-Mass Spectrometry (GC-MS)

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Abstract:

The structural analysis of fatty acid mixtures by gas chromatography-mass spectrometry (GC-MS) has been characterized from Tulipa systola Stapf. Flowers, wild plant growing in Kurdistan region-Iraq. The fatty acids of the lipid fraction were mainly saturated fatty acids, the highest value recorded for isopropyl palmitic acid methyl ester (47.02%), glycerol α-palmitic acid methyl ester (19.66%) and stearic acid methyl ester (8.64%). In addition, two unsaturated fatty acids that characterized were oleic acid methyl ester (6.95%) and undecylenic acid methyl ester (2.22%).

Keywords*: Tulipa systola, Fatty acid methyl ester, Gas chromatography-mass spectrometry.*

Introduction:

The structural analysis of fatty acid mixtures by gas chromatography-mass spectrometry (GC-MS) has always been a challenge since its introduction in lipid analysis in 1960 as a result of the technical progress in high resolution capillary GC, GC-MS equipment and derivatization methods, a great number of new applications for biochemistry, food chemistry, phytochemistry, microbiology etc. have been developed in the last decade, and GC-MS is today without doubt one of the most powerful techniques for fatty acid analysis (Christie 1989).

For structural characterization of fatty acids, information about the chain length, the chain type, the double bond equivalents, the types (double or triple bond) of unsaturation, the positions of the unsaturated bonds in the hydrocarbon chain, the geometric configurations of double bonds, and the types of other functional groups, e.g. hydroxy or epoxy groups, and their positions in the hydrocarbon chain and stereochemistry, are needed. The informations were provided by a GC-MS analysis of the routinely made fatty acid methyl esters (FAMEs) consists of the GC retention times that can also be compared with distinct structural properties (Christie 1983).

The first of many important advances in the early development of GLC for analytical purposes was the separation and determination of FA reported by (James and Martin 1952). Soon after, the analytical separation of FAME by vapor-phase chromatography was described by (Cropper and Heywood 1953). Since then, the characterization of FA composition by esterification to FAME and subsequent determination by GC has become one of the most widely performed analyses in lipid research laboratories and has found broad application to biochemical, biomedical, microbiological, agricultural, and ecological research (Eric 2005).

Plant material

Tulipa systola Stapf. was collected on April 2014 from Korek Mountains in Rewanduz - Erbil / Kurdistan region. The materials were identified and classified by Dr. Abdullah Shukur from Education Salahaddin University Herbarium (ESUH) at the University of Salahaddin, Erbil-Iraq. A voucher specimen was deposited with the accession number (7201). Flowers were cleaned and air-dried in the shade at room temperature (20-25^{o}C). After drying, grounded using a laboratory grinding mill, to provide a homogeneous fine powder for the analysis. Powdered materials were stored in dark bottles and maintained at room temperature until required.

Experimental

Dried and powdered flowers of *T. systola* (70 g) were macerated with Methanol (Sigma Aldrich) (500 mL) at room temperature with continuous shaking for (3hrs). The solvent was evaporated to dryness under vacuum at ~35°C and then (1g) of the extract fractionated by MPLC (Isolera) R-C_{18}, (MeOH/H_2O; 20:80 – 100%MeOH gradient). Twelve fractions were obtained, fraction ten (12.4mg) a mixture of fatty acids obtained which then kept at -20°C before using for GC-MS analysis. In order to analyze the oily mixtures of *T. systola* firstly, the fatty acid mixture was dried with anhydrous sodium sulphate attentively. After dissolving in

HPLC grade n-hexane, it was treated with 2 M Methanolic KOH at room temperature for 30 s. The upper phases of the reaction mixtures (1μL) were analyzed by GC-MS system.

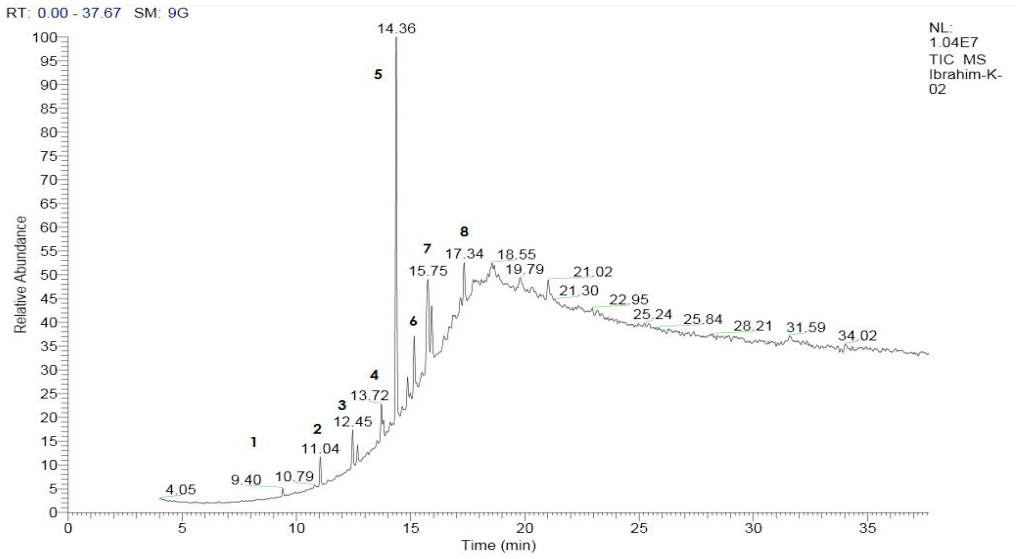
The methylated ester fatty acids were injected in to GC-MS (Thermo DSQ) with an (HP5 30m x 0.25mm) column, FOCUS GC, Inlet Temperature (250°C), carrier gas (Helium 1 ml/min in constant flow), ramp: (60°C x 1 min, 10°C/min to 260°C, 260°C x 15min). DSQ MS, ion source (250°C), positive ions, mass range (45-600 full scan).

Results and discussion

One of the most significant advances that have been made understands the importance of dietary fatty acids for human health. In biomedical research, GC data on human tissue fatty acids had already been published (Horning). The basic features of polyunsaturated, especially of essential fatty acid, metabolism, studied earlier in animal tissues (Mead) and (Holman) could be verified in more detail by GC analysis (Mohrhauer and Holman 1963).

It is known that the prevention of many diseases is possible to have healthy and balance diet, especially the balance of EFAs (Suheyla Kirmizigul 2012), Growth temperature, had a consistent effect on the quantitative composition with greater amounts of more saturated lipids occurring at the higher growth temperatures, however the variations in fatty acid contents are attributable both to environmental and genetic differences (Sanchez-Machado and Robert 2004).

In this study the composition of a mixture of fatty acid, methyl esters from the flowers of *Tulipa Systola* were analyzed using GC-MS (Table 1) for the first time. The fatty acids of this lipid fraction, in general, mainly saturated, the highest value being Isopropyl Palmitic acid, methyl ester (47.02%), Glycerol α-palmitic acid, methyl ester (19.66%) and Stearic acid methyl ester (8.64%) while the two unsaturated fatty acids that characterized were oleic acid methyl ester (6.95%) and Undecylenic acid, methyl ester (2.22%).



Figure(1): GC-MS chromatogram of the fatty acid methyl esters from *Tulipa systola*.

Table (1): Fatty acid methyl esters percentage from *Tulipa systole* Stapf.

Peak no.	m/z	Fatty acid methyl ester	t_R (min)	Percentage (%)
1	198	Undecylenic acid, methyl ester	9.4	2.22
2	258	Dodecanedioic acid, dimethyl ester	11.05	4.06
3	272	Brassylic acid, methyl ester	12.45	5.18
4	296	Oleic acid, methyl ester	13.72	6.95
5	298	Isopropyl Palmitic acid, methyl ester	14.36	47.02
6	298	Stearic acid, methyl ester	15.15	8.64
7	330	Glycerol α-palmitic acid, methyl ester	15.75	19.66
8	370	Adipic acid dioctyl ester	17.34	6.26

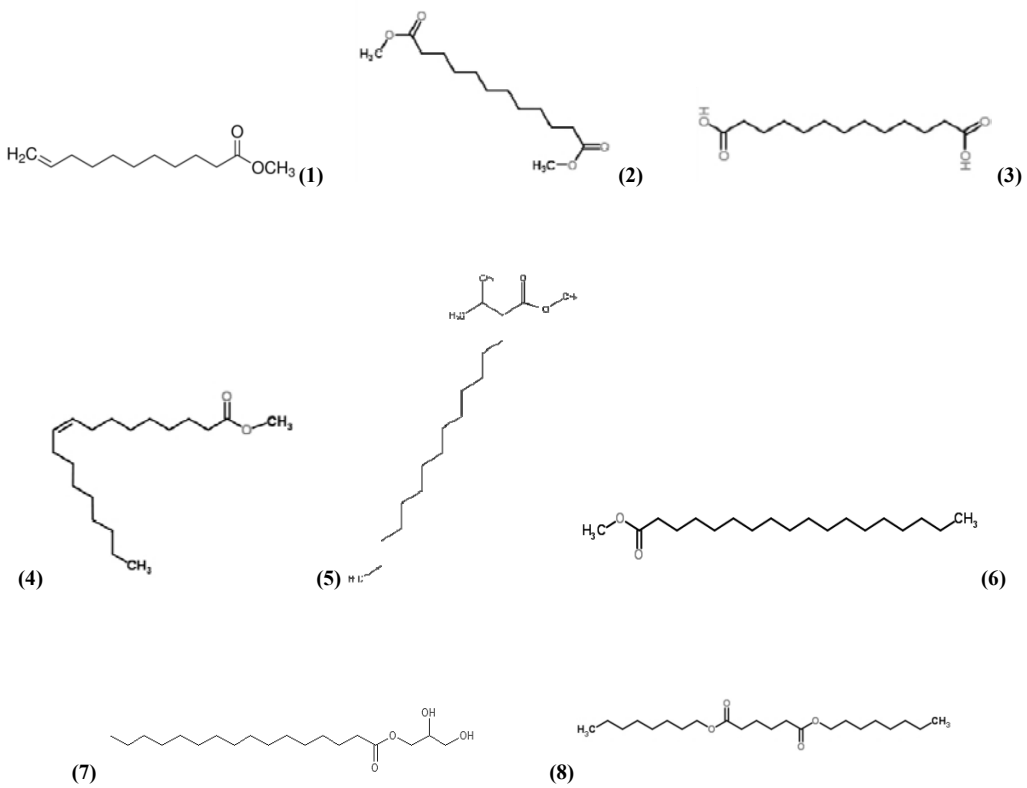


Figure (2): chemical structure of the characterized fatty acid methyl esters from *Tulipa systola*.

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ناسینەوەی تێکەڵەی مەسیلی ئیستەری ترشە چەوریەکان لە گوڵی تولیپ بە بەکارهێنانی ئامێری (GC-MS)

کورتیا لێکولینی:

لەم توێژینەوەیە بۆ یەکەمین جار بڕ و پێکهاتەی تێکەڵەی مەسیلی ئیستەری ترشە چەوریەکان لە گوڵی تولیپ دیاری کرا بە بەکارهێنانی ئامێری (GC-MS). پێکهاتەی سەرەکی ئەم دەرهێنراوە چەوریە بریتی بوو لە ترشە چەوریە تێرەکان کە بەرزترین ڕێژە بۆ مەسیل ئیستەری ئایزۆپرۆپیل پالمیتەیت بە ڕێژەی (47.02%)، مەسیل ئیستەری ئایزۆپرۆپیل گلیسرۆل بە ڕێژەی (19.66%) و مەسیل ئیستەری ترشی ستیریك بە ڕێژەی (8.64%) تۆمارکران. لەو کاتەی کە پێکهاتەی ترشە چەوریە ناتێرەکان بریتی بوون لە مەسیل ئیستەری ترشی ئۆلیك بە ڕێژەی (6.95%) و مەسیل ئیستەری ترشی ئوندیسایلینیك بە ڕێژەی (2.22%).

تشخيص مزيج من الأسترات المثيل للأحماض الدهنية من أزهار نبتة التوليب الأحمر بتقنية (GC-MS)

الخلاصة:

تم تحليل مزيج من الأسترات المثيل للأحماض الدهنية من الأزهار نبتة التوليب الأحمر وتم تشخيصها بتقنية (GC-MS). وقد تبين أن الأحماض الدهنية في المستخلص الدهني هي دهون مشبعة بشكل رئيسي، الحامض الدهني الأيسوبروبيل استر البالمتيك (47.02٪)، الحامض الدهني المثيلي الجلسرين البالمتيك استر (19.66٪) والحامض الدهني المثيلي ستيريك استر (8.64٪) بينما كان نسبة كل من الحامض الدهني المثيلي أوليك استر (6.95٪) والحامض الدهني المثيلي أوندیسایلینیك استر (2.22٪).