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INFRARED SPECTROSCOPIC STUDIES ON EDIBLE AND MEDICINAL OILS

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ABSTRACT: The paper reports IR spectroscopic data of edible and medicinal oils of plant origin. For IR analysis, *Ten* edible oils and *Fifteen* medicinal oils were selected. FTIR spectra were recorded. The FT – IR spectra of edible and medicinal oils show a series of bands with different intensities and reveal the composition of fatty acids and degree of saturation of the selected oils.

The study suggests that IR spectroscopy can be considered as a vital technique for identification, analysis, determination of degree of saturation of fatty acids and detection of adulteration of oils of plant origin.

1. Introduction

As is known, infrared spectroscopy is a potential tool to provide valuable information in the study of biomaterials with respect to structure of macromolecular components and their conformations within the tissue. It can also supplement other physical and chemical methods of analysis for the qualitative and quantitative determinations of different components present in the biomaterials.

In specific cases infrared spectroscopy is helpful for the identification of inorganic and organic constituents of biomaterials. In general, spectral analysis of tissues of biological system depends upon the material present and the analyte being sought. The tissue itself is dominated by the spectrum of the macromolecular components, which are present in the large quantity. If living tissue is being examined, it is dominated by the water spectrum.

Safwan et. al. [1] used Fourier transform infrared (FT IR) spectroscopy to classify different edible oils including the virgin olive oil. They identified adulteration of virgin olive oil with different volume ratios (2 5, 50 and 75%) of both corn and sunflower oils quantitatively. The total spectral data in region of (4000 -400) cm^{-1} for all oil samples was recorded and then analyzed using different chemometric tools such as principal component analysis (PCA) clustering and partial least square discriminant analysis (P LSD A).

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Ersillia Alexa et al [2] did FTIR studies to identify the adulteration of some olive, peanut, corn germ and pumpkin oils with sunflower oil, the most common oil in Romania. Their results showed spectral differences in the spectrum of various types of vegetable oils, enabled to identify the addition of foreign oil in an oil sample using calibration curves established for certain characteristic frequencies in known mixed oils.

Marina Atena Poiana, et. al. [3] made an attempt to evaluate the use of FT-IR spectroscopy as an effective analytical tool in order to assess the olive oil (OO) adulteration with cheaper vegetable oil (sunflower oil). Their data demonstrated that FT-IR spectroscopy proved to be a valuable tool to identify the differences recorded in oil samples spectra and finally, to appreciate the degree of Olive Oil adulteration.

Valchos, et. al. [4] used FT-IR spectroscopy as an effective analytical tool in order: (a) to determine extra virgin olive oil adulteration with lower priced vegetable oils (sunflower oil, soyabean oil, sesame oil, corn oil) and (b) to monitor the oxidation process of corn oil samples undergone during heating or/and exposure to ultraviolet radiation.

A perusal of literature reveals that a systematic study has not been done on IR spectroscopy of various edible and medicinal oil of plant origin. Hence, different types of *Ten* edible and *Fifteen* medicinal oils were selected for systematic and comparative IR study.

2. Materials and Methods

The FT-IR spectra were recorded with Thermo Nicolet Nexus 670. The table top Thermo Nicolet Nexus 670 calibrated and checked with polystyrene film. The sample filled in the liquid cell of 1mm thickness with a micro syringe. The liquid cell was placed in the sample compartment. The resolution was kept at 4 cm^{-1} and scanning time was fixed at 38 Sec. A total number of 32 scans were carried out on each sample. The scanning range was fixed from 4000 – 400 cm^{-1} for each sample. And also the ranges 2000-1400cm^{-1}, 1400 – 600cm^{-1} and 1200 – 1000cm^{-1} were carried out.

3. Results and Discussion

Table 1 shows the FTIR data of 10 edible oils under investigation. The analysis is made based on percentage of transmittance and wave number. It is observed that for wave numbers 3005 to 3010 cm^{-1}, the % transmittance varies from 0.914 to 0.948. In this range, %transmittance is maximum for ground nut oil and is minimum for cottonseed oil i.e. 0.914. The percent transmittance at wave numbers 1741 to 1744cm $^{-1}$ is found to be maximum for Ground oil i.e. 0.671 and is minimum for Coconut oil and Cottonseed oil. For wave number

1652 cm^{-1}, percentage of transmittance is shown only by Ground nut oil and Safflower oil. The wave numbers from 1456 to 1459 cm^{-1} give the %transmittance from 0.824 to 0.873. In the above said range of wave numbers, Ground oil has maximum and Coconut has minimum value of %transmittance.

Table 2 depicts the FTIR data of 15medicinal oils under study. It is observed that for Lemon Grass oil, Garlic oil and Cinnamon oil %transmittance is zero. But for rest of the medicinal oils the wave numbers from 3018 to 3006cm^{-1}, the % transmittance varies from 0.584 to 0.946. The highest value of transmittance is noted for Pistachio oil and lowest is for Clove oil and Black cumin seed oil. It is to be noted that for wave numbers 1737 to 1744cm^{-1} the transmittance values varies from 0.008 to 0.862. It is interesting to note that Cinnamon oil have zero %transmittance at 1737 to 1744 cm^{-1} wave numbers. The maximum value is observed for Eucalyptus oil and minimum for Clove oil. The wave numbers from 1601 to 1672cm^{-1} show variation of %transmittance from 0.20 to 0.981. The highest value is found for Guard oil and Poppy seed oil where as the lowest is for Cinnamon oil. It is to be noted that Garlic oil has zero transmittance for wave numbers 1601 to 1672cm^{-1}. For wave number 1440 to 1457cm^{-1} the medicinal oils show the % transmittance 0.450 to 0.887. The maximum value of %transmittance is found for Garlic oil and minimum is for Clove oil.

Table 1: FTIR Data of Edible oils

Oil	Wave Number in cm^{-1} (% Transmittance)													
GRN	3008 (0.948)	2923 (0.682)	2855 (0.792)	1744 (0.671)	1652 (0.958)	1459 (0.873)	1371 (0.929)	1235 (0.876)	1160 (0.744)	1098 (0.849)	-----	913 (0.974)	874 (0.982)	721 (0.861)
SEM	3008 (0.916)	2924 (0.665)	2855 (0.784)	1743 (0.519)	---	1457 (0.843)	1369 (0.785)	1222 (0.752)	1160 (0.749)	1098 (0.849)	972 (0.964)	909 (0.969)	-----	721 (0.863)
OLV	3005 (0.926)	2923 (0.650)	2854 (0.774)	1744 (0.492)	---	1458 (0.838)	1369 (0.776)	1222 (0.742)	1158 (0.737)	1097 (0.846)	-----	----	899 (0.965)	721 (0.867)
SNF	3008 (0.916)	2924 (0.684)	2855 (0.800)	1744 (0.509)	---	1457 (0.849)	1369 (0.797)	1222 0.761)	1160 (0.740)	1097 (0.841)	968 (0.957)	910 (0.962)	----	721 (0.847)
CCT	-----	2923 (0.664)	2855 (0.787)	1743 (0.462)	-----	1458 (0.824)	1369 (0.741)	1221 (0.694)	1155 (0.651)	1108 (0.763)	964 (0.948)	904 (0.961)	893 (0.956)	721 (0.862)
PAM	3006 (0.933)	2923 (0.639)	2854 (0,764)	1743 (0.494)	------	1458 (0.837)	1369 (0.774)	1222 (0.739)	1158 (0.730)	1108 (0.835)	------	901 (0.967)	-----	72 (0.882)
MST	3008 (0.916)	2924 (0.916)	2855 (0.795)	1744 (0.544)	----	1457 (0.853)	1369 (0.787)	1221 (0.768)	1161 (0.820)	1102 (0.895)	------	903 (0.971)	------	721 (0920)
CTS	3008 (0.914)	2924 (0.664)	2855 (0.787)	1743 (0.463)	-----	1457 (0.836)	1369 (0.749)	1222 (0.718)	1159 (0.734)	1100 (0.838)	970 (0.957)	908 (0.961)	-----	721 (0.862)
SYB	3008 (0.926)	2924 (0.670)	2855 (0.789)	1744 (0.523)	------	1458 (0.849)	1369 (0.810)	1222 (0.773)	1159 (0.734)	1100 (0.840)	968 (0.958)	910 (0.965)	-----	721 (0.857)
SAF	3009 (0.918)	2924 (0.695)	2855 (0.897)	1744 (0.544)	1655 (0.982)	1457 (0.857)	1369 (0.824)	1223 (0.786)	1160 (0.742)	1097 (0.841)	967 (0.957)	911 (0.960)	-------	721 (0.844)

Table 2: FTIR Data on Medicinal Oils

Oil Code	Wave Number in cm^{-1} (% Transmittance)										
ALM	3005 (0.919)	2922 (0.661)	2855 (0.782)	------	------	------	------	------	1744 (0.489)	------	------
CLV	3457 (0.823) 3018 (0.584)	2941 (0.634)	-----	2577 (0.968)	2434 (0.987)	2346 (0.984)	2131 (0.957)	2253 (0.983)	1737 0.015	1601 (0.904)	1511 (0.709)
ECT	3465 (0.992)	2924 (0.740)	2724 (0.983)	-----	-----	2355 (0.983)	-----	-----	1741 (0.862)	1640 (0.966)	----
NEM	3463 (0.968)	2924 (0.642)	2855 (0.781)	-----	-----	-------	-------	-------	141 (0.355)	----	----
PST	3006 (0.946)	2923 (0.683)	2856 (0.794)	-----	-----	-----	------	------	1742 (0.705)	-----	---
PPS	3008 (0.934)	2924 (0.696)	2856 (0.805)	----	-----	-----	-----	-----	1744 (0.681)	1657 (0.981)	---
GRD	3008 (0.938)	2923 (0.680)	2856 (0.794)	----	----	2350 (0.989)	----	----	1744 (0.653)	1655 (0.981)	---
NTG	3441 (0.890)	2964 (0.774	-----	----	-----	25 (0989)	2132 (0.992)	2254 (0.994)	1738 (0.464)	1628 (0.951)	507 (0.925)
WLN	3463 (0.971) 3010 (0.856)	292 (0.70)	2856 (0.801)	-----	------	------	-----	-----	1742 (0.352)	-----	------
CIN	3298 (0.983) 3057 (0.941)	----	2814 (0.913)	----	-----	2352 (0.986)	-----	-----	1896 (0.991)	1672 (0.20)	----
LMNG	3463 (0.950)	2968 (0.812)	2862 (0.876)	-----	------	------	2135 (0.988)	------	1734 (0.423)	1672 (0.585)	----
GRL	3334 (0.962)	2928 (0.787)	2865 (0.862)	-----	------	2351 (0.991)	-----	-----	1725 (0.584)	------	1589 (0.965)
CTR	3461 (0.939) 3011 (0.860)	2926 (0.660)	2857 (0.790)	-----	------	------	------	-----	1740 (0.302)	-----	------
BCS	3460 (0.895) 3014 (0.584)	2929 (0.516)	2857 (0.746)	2576 (0.969)	----	2349 (0.985)	2132 (0.956)	2252 (0.983)	1740 (0.008)	-----	------
LSD	3010 (0.928)	2924 (0.724)	2856 (0.826)	----	-----	-----	-----	----	1744 (0.677)	1654 (0.979)	----

Oil Code	Wave Number in cm^{-1} (% Transmittance)										
ALM	1456 (0.836)	1369 (0.771)	1221 (0.740)	----	1157 (0.742)	1096 (0.847)	----	904 (0.969)	-----	721 (0.865)	----
CLV	1441	1367	1270	1219	1120	1090	1022	908	805	738	695

	(0.450	(0.075)	(0.768)	(0.065)	(0.869)	(0.896)	0,680)	(0.750)	(0.894)	(0.616)	(0672)
ECT	1456 (0.847)	1368 (0.791) 1306 (0.937)	1265 (0.927)	1219 (0.796)	1166 (0.867) 1116 (0.947)	1081 (0.847)	1018 (0.922)	984 (0.716) 922 (0.934)	885 (0.927) 844 0.899	787 (0.935)	649 (0.981)
NEM	1453 (0.790)	1367 (0.594)	------	1219 (0.565)	1160 (0.755)	1106 (0.844)	1035 (0.914)	903 (0.946)	----	722 (0.881)	-----
PST	1457 (0.872)	1370 (0.919)	1236 (0.867)	1161 (0.754)	1104 (0.854)	-----	980 (0.940)	----	----	721 (0.881)	------
PPS	1457 (0.874)	1369 (0.923)	1233 (0.871)	----	1159 (0.743)	1100 (0.849)	-----	913 (0.969)	-----	720 (0.851)	-----
GRD	1457 (0.867)	1368 0.900)	1228 (0.854)	----	1158 (0.733)	1104 (0.842)	----	973 (0.952) 911 (0.971)	----	720 (0.865)	----
NTG	1445 (0.769)	1368 (0.554)	-----	1218 (0.567)	1125 (0.814)	-----	1030 (0.839)	914 (0.844)	831 (0.969)	740 (0.814)	695 (0.849)
WLN	1453 (0.805)	1367 (0.610)	----	1218 (0.581)	1161 (0.759)	1100 (0.849)	----	907 (0.941)	------	719 (0.849)	------
CIN	1492 (0.929)	1394 (0.957)	-----	1297 (0.888) 1247 (0.913)	1120 (0.346)	-----	-----	972 (0.566)	843 (0.940)	746 (0.446)	686 (0.546)
LMNG	1444 (0.755)	1370 (0.585)	1281 (0.726)	1219 (0.635)	1122 (0.754)	1076 (0.848)	1032 (0.872)	-----	841 (0.885)	746 (0.865) 703 (0.952)	---
GRL	1457 (0887)	1383 (0.850)	1272 (0516)	1204 (0808)	1126 (0.634)	-----	1072 (0.724)	989 (0.887) 950 (0.90)	------	778 (0.908) 741 (0.780) 701 (0.872)	657 (0.954)
CTR	1452 (0.785)	1367 (0.546)	-----	1218 (0.581)	1164 (0758)	1093 (0.824)	----	901 (0.941)	----	722 (0884)	---
BCS	1440 (0.490)	1367 (0.075)	1219 ()	----	1101 (0.798)	----	----	903 (0.817)	----	720 (0.860)	----
LSD	1456 (0.881)	1368 (0.928)	1234 (0.872)	----	1160 (0.740)	1100 (0.848)	-----	915 (0.964)	----	716 (0.832)	----

For the spectroscopic analysis, the biological material may be classified into *Four* types:

1. Organic tissues like muscle (skeletal, cardiac and visceral), brain liver, kidney, spleen, etc.
2. Mineralised or calcified tissues such as teeth, bone, integuments, bone, calculi, gallstones, etc.
3. Body fluids, such as cerebral fluid, spinal fluid, pleural fluid, saliva, blood, urine, etc.

4. Biological macro molecules, such as amino acids, proteins, lipids, fatty acids, etc.

In the present investigation, IR analysis on edible and medicinal oils is aimed to characterized, to evaluate adulterants, to examine heating effects of oils. Further, it is an attempt to determine what variations occurred in the spectrum of normal oils. What changes might occur in normal composition of oils with storage period and heat treatment (ie frying) ?

It is a well known fact that the visible region of electromagnetic radiation extends from 0.38 to 0.78 µm. The IR region extends from the end of the visible region at 0.78 µm to the microwave region with wave length of ~1mm. In general IR spectrum is divided in to *Three* regions.

The region between visible and mid infrared is called *Near Infra red.* This region of IR has been used for many applications, especially quantitative analysis.

The region used by the material scientists is the *mid infra red* region extending from 4000 cm^{-1} to 200 cm^{-1}.

The region beyond 200 cm^{-1} is called the *Far Infra red* region. This region is concerned with low frequency vibrations and some molecular rotations.

In infra red spectrum, some spurious bands may be found due to various factors. A list of such bands is presented in Table 3, with a view to take care while recording and analyzing the spectrum.

The FT – IR spectra of edible and medicinal oils reveal a series of bands with different intensities (Table 1 & 2.). For the convenience of analysis the IR spectra from 4000 cm^{-1} to 400 cm^{-1} are divided into the following regions (Table 4).

Table 3: Some common spurious absorption bands in infrared spectrum

WaveNumber (cm^{-1})	*Wavelength (cm)*	*Compound Or Group*	*Source*
3700	2.70	H_2O	Any Source
3650	2.74	H_2	Any Source
3450	2.90	H_2O	Hydrogen bonding in water, usually in KBr Disc
2350	4.26	CO_2	Atmospheric absorption
2000-1430	5.0 – 7.0	H_2O	Atmosphere
1640	6.10	H_2	Water of crystallization
1430	7.00	CO_3	Contaminant in halide widow
1360	7.38	NO_3	Contaminant in halide widow
1270	7.90	$SiCH_3$	Silicon oil or grease
1110-1000	9.0 -10.0	SiO	Glass
667	14.98	CO_2	Atmosphere

Table 4: IR spectral data presented in selected regions for the analysis

Region	*Band*	*Characteristic Wave number*	*Remark*
I	4000-3100		Edible oils mostly do not have IR absorption. Hence it is not useful.
		3400	Carbonyl C=O stretching bond
II	3100-2800	3010 2965,2935, 2895 & 2855	• CH stretching vibration of the *cis* double bond (=CH) within unsaturated fatty acyl ester. • Index of degree of unsaturated oil. • Detection of adulteration of oil. • Classification of vegetable oils. • Characteristic to the symmetric & asymmetric vibrations.
III	1800-1600	1750, 1660	• High content in saturated fatty acids • Short Hydro carbon chain.
		1665	• Corresponds to C=C • Correlated to the content of poly unsaturated fatty acids.
		1654	• *Cis* isomers
		1743	• Characteristic to saturated fatty acid. • Lipid absorption arising from the C=O group of cholesterol ester
IV	1650-1390	1440-1460 1460 1650 1370	• Used to determine total unsaturation. • CH_2 bend • C=C • CH_3 symmetric bending
V	1390-1230	1303, 1270 1375	• Double links *Cis* unconjugated. • CH_3 Symmetric deformation
	1230-700	1160,1230, 1110 720-725	• Carbohydrate radical from triglyceride structure of oil. • Triglycerides • CH_2 Rocking

Region 1 (4000 – 3100 cm^{-1}) does not show any band for edible oils, but reveals bands around 3400 cm^{-1} for some of the medicinal oils namely CLV, ECT, NEM, NTG,WLN,CIN,LMNG,GRL,CTR and BCS oils, which can be attributed to Carbonyl C=O stretching bond.

Region 2 (3100-2008 cm^{-1}) is the region of functional groups such as (1) Hydrogen's stretching, (2) double bond's stretching and (3) deformation and bending of other bonds.

The IR spectra of edible (except CCT) and medicinal (except CLV, ECT, NEM, NTG, WLN, CIN, LMNG, GRL, CTR and BCS) oils show notable differences in the band near 3008 cm^{-1} assigned to the C-H stretching vibration of the *cis* – double bond (=CH). Interestingly, the band near 3008 cm^{-1} is missing in coconut (CCT) oil. This band at 3008 cm^{-1} can be the index of degree of unsaturation of edible and medicinal oils and also be used for their characterization. Also, the IR spectrum of oils under study presents bands near 2923 cm^{-1} & 2855 cm^{-1}, which are characteristics to symmetrical and asymmetrical stretching vibration of aliphatic CH_2 group of triglycerides. These bands are more significant in vegetable oils.

Region 3 (1800-1600 cm^{-1}) The bands at 1746 cm^{-1} and 1654 cm^{-1} present in spectra are concerned with double bond stretching. The band at 1746 cm^{-1} is concerned to oils with high content in saturated fatty acids and short carbohydrate chain. But the medicinal oils namely CLV (1737 cm^{-1}), CIN (1896 cm^{-1}) and LMNG (1734 cm^{-1}) do not show this band. The spectral band at 1654 cm^{-1} corresponds to the double C=C link and may be related to the polyunsaturated fatty acids. It is found in spectra of edible (GRN and SAF) and medicinal (PPS, GRD and LIN) oils.

Region 4 (1600-1370) corresponds to deformation and bending vibrations. The spectra of oils of the present investigation reveal bands near 1460 cm^{-1} related to bending vibration of CH_2 and $CH_{3;}$ and at 1370 cm^{-1} concerned with the bending vibrations CH_2 groups. These bands can be used to determine total unsaturation.

Region 5 (1230 – 700 cm^{-1}) corresponds to carbohydrate radical from the triglyceride structure of oils. From the spectra of selected oils, the bands at 1220 cm^{-1} and 1160 are evident, which are correlated to stretching vibration of C-O ester group. The band near 721 cm^{-1} is concerned with CH_2 rocking.

The study suggests that IR spectroscopy can be considered as a vital technique for identification, analysis, determination of degree of saturation of fatty acids and detection of adulteration of oils of plant origin.

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