



A Research Article On Unsaturation Of Oils By Using Ftir Spectroscopy And Heavy Metal Analysis By Aas

Sai Deekshitha G, Sridevi P,

Department of Pharmaceutical Analysis, Sri Venkateshwara college of Pharmacy, Madhapur, Hyderabad, Telangana.

ABSTRACT:

FTIR is the technique used to obtain spectrum of absorption emission. The IR is used as the measurement of degree of unsaturation of fats and oils. AAS is a technique used to determine trace elements in the edible oils. This technique is used to detect low concentration of elements in oil samples. Analysis of fats and oils in determination of isolated trans isomers, official method by AOCS (American oil Chemists society), Association of Official Analytical Chemist AOAC). The determination is based up on peak height of absorption band of isolated trans bonds range 966 cm^{-1} which is due to C=C- H bending vibration. Edible oils like Sesame oil, Sunflower oil, Rice bran oil, Groundnut oil. The unsaturation of oils are done to know the Unsaturation of oils. Different types of edible oils are Olive oil, Safflower oil, Sunflower oil, Rice bran oil, Groundnut oil, Coconut oil, Sesame oil. These oils consist of fatty acids like saturated fats, monosaturated fats, polyunsaturated fats, triglycerides, Cholesterol. Dietary fats are closely related to Coronary Artery Diseases (CAD), cholesterol, saturated fats and trans fats are harmful whereas MUFA, PUFA (Omega-3 PUFA) are beneficial to heart, inflammation, etc. This article reviews the benefits/harms of common edible oils on different disease. The variety of olefinic bonds in alkyl chains, including their position and shape, causes the fatty acids to differ from one another.

KEYWORDS: IR Spectroscopy, AAS, Oils.

INTRODUCTION:

Edible oils are essential for nourishment because they come from plants. A high-quality edible oil contains a variety of physicochemical characteristics and a distinct flavour. When assessing the quality of edible vegetable oils, a number of quality parameters, such as the saponification value, acid value, peroxidase value, and iodine value, will be useful. The goal of the current study was to compare the quality of cooked and uncooked oils.

Along with proteins and carbs, edible fats and oils are essential parts of the human diet. While fish oils like sardine and tuna oils are frequently added to processed meals, edible oils are primarily vegetable oils like soybean, canola, palm, and corn oils, among others. High energy and important fatty acids, including linoleic, linolenic are found in fats and oils. Pan- and deep-frying, salad dressing, mayonnaise, and processed foods like chocolate, cream, and baked goods all include edible fats and oils.

Additionally, during storage, fats and oils in oil meals are vulnerable to auto- and photo-oxidation processes, which could result in unfavourable flavour. During storage, fats and oils in oil meals are also vulnerable to auto- and photo-oxidation reactions, which can result in unfavourable flavour and taste. To guarantee and preserve the quality of the food items, it is crucial to assess the extent of fat and oil degradation during heat cooking and storage.

DIFFERENT TYPES OF OILS:

- Sunflower oil
- Sesame oil
- Groundnut oil
- Rice bran oil

1 SUNFLOWER OILS: -

Sunflower oil is a vegetable oil that comes from the seeds of the sunflower plant, *Helianthus annuus*. It's a popular cooking oil with many uses, including frying, baking, and salad dressings.

There are 3 types of sunflower oils

High oleic

Linoleic

NuSun

Oil crops stand out for providing energy and protein to the human diet. With a 50% oil content and 15–21% protein, sunflower is an important crop. Because of its nutritional value, it is ranked among the best plant oils for human diets and ranks second in the world in the production of edible oils, after soy oil. Magnesium, iron, copper, calcium, zinc, salt, potassium, phosphorus, selenium, and manganese are among the minerals that are abundant in its seeds, which also include a significant number of vitamins, minerals, and tocopherols. A variety of techniques have been used to produce an advanced spectrum of sunflower oils with higher oleic acid, stearic acid, linoleic acid, palmitic acid, and low saturated fatty acids.

2 SESAME OIL: -

Sesame oil is an edible vegetable oil derived from sesame seeds. The oil is one of the earliest-known crop-based oils. It is used in cooking, flavouring.

Sesame oil is used in cooking, especially for stir-frying and salad dressings, and as a flavouring agent in East Asian dishes. It is also used in soaps, paints, cosmetics, perfumes, and insecticides

Sesame oil can be used as a vehicle for intramuscular oily injections. After burning, it can produce high quality black ink.

sesame oil comes in two types. One type of sesame oil is a pale-yellow liquid and has a pleasant grain-like Odor and somewhat nutty taste.

3 GROUNDNUT OIL: -

Groundnut oil, also known as peanut oil or arachis oil, is a vegetable oil that's used for cooking, making confectioneries, and as a shortening. Groundnut oil is used in American, Chinese, Indian, African, and Southeast Asian cuisine. It's commonly used for frying because of its high smoke point. It's also used in salad dressings and pourable dressings because it keeps solids in suspension for a long time.

Groundnut oil contains more than 80% unsaturated fatty acids, including 41.2% oleic acid and 37.6% linoleic acid.

4 RICE BRAN OIL: -

Rice bran oil (RBO) is a cooking oil extracted from the outer layer of rice grains, called the bran RBO is a by-product of rice milling. The bran is the hard, brown outer layer of rice RBO is a popular cooking oil in many Asian countries, including India, Japan, China, and Indonesia. It's often used for high-temperature cooking methods like deep frying and stir frying.

RBO contains saturated, polyunsaturated, and monounsaturated fatty acids, as well as phytochemicals like tocopherols, tocotrienols, and phytosterols. RBO is said to have a range of health benefits, including lowering cholesterol, reducing blood pressure, and preventing colorectal cancer. One study found that oil pulling with RBO reduced bad breath in pregnant women.

MATERIALS AND METHODS:

FTIR: -

Fourier transform infrared (FTIR) is a method of infrared spectroscopy that uses an interferometer to produce an infrared absorption spectrum of a sample. Different chemical structures produce different spectral fingerprints, so FTIR is a useful tool for identifying chemical bonds in a molecule. FTIR is accurate, precise, fast, sensitive, and easy to use. It's also non-destructive to the sample.

Fourier transform infrared spectroscopy (FT-IR) spectroscopy is used to generate bacterial spectral scans based on the molecular composition of a sample. Basically, infrared spectroscopy consists of the infrared source, the sample, and the detector. When IR is absorbed or transmitted through the sample to the detector, it generates a "scan" or "fingerprint" profile. A library of spectral scans can be generated for different bacterial species and strains, which can be used for future comparison. This method requires transfer of cells (biomass) from the growth media to an IR-reflecting substrate for spectral collection.

AAS:-

Atomic absorption spectroscopy (AAS) is a technique that measures the concentration of metals and metalloids in a sample. It's a common tool in analytical chemistry because it's relatively inexpensive, easy to use, and offers high sensitivity and low interference. AAS works by converting a sample into free atoms and then measuring how much electromagnetic radiation they absorb. Atoms absorb light at specific wavelengths, so when a sample is exposed to a specific wavelength of light, the energy is absorbed by the atoms.

The quantity of interest in atomic absorption measurements is the amount of light at the resonant wavelength which is absorbed as the light passes through a cloud of atoms. As the number of atoms in the light path increases, the amount of light absorbed increases in a predictable way. By measuring the amount of light absorbed, a quantitative determination of the amount of analyte element present can be made. The use of special light sources and careful selection of wavelength allow the specific quantitative determination of individual elements in the presence of others.

PROCEDURE:

IR SPECTROSCOPY FOR DETECTING FUNCTIONAL GROUPS IN OILS:

Switch on the IR instrument and allow to stabilize.

- Collect the oil samples.
- Click on background spectrum.
- Dropper is used to place a sample on NaCl lens.
- Probe is twisted to lock.
- IR spectrum is recorded.
- Functional groups is recorded with peaks

- Chloroform is used to clean the probe
- Turn off the instrument
- Analyse the spectrum for unknown sample.

AAS FOR DETECTING HEAVY METALS IN OILS:-

To detect the presence of heavy metals in oils

Digestion method:

a. Diluent Preparation:

Take 69.75ml of Nitric acid into 800ml of water and dilute to 1000ml

b. Flask Preparation:

Wash all the glassware with soap & tap water and add 10% Nitric acid & wash it. Finally, wash with Deionized water and keep in a hot air oven

c. Sample Preparation:

Take 2gms of oil sample in a Digestion flask and one empty digestion flask for Blank, Place a few glass beads into both flasks as anti-bumping granules. Add 20ml 65 % Nitric acid and 10ml 70 % Perchloric acid and place aside

d. Sample Digestion:

Digest the sample in the fume hood at room temperature for 24 hours.

The brown-red color appears due to the fume of HNO_3 formed, then shake the flask

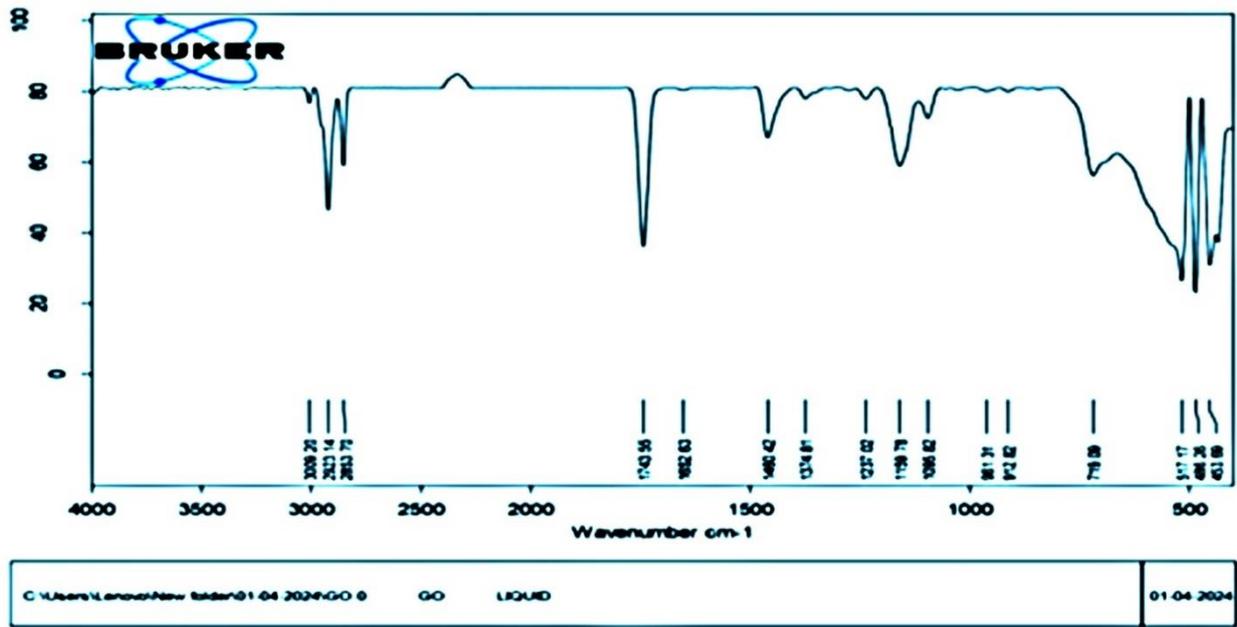
Place the flask on the heating mantle of the digestion at 120°C for 30 minutes until the brown-red fume is removed gradually from both the flask.

e. Sample Analysis:

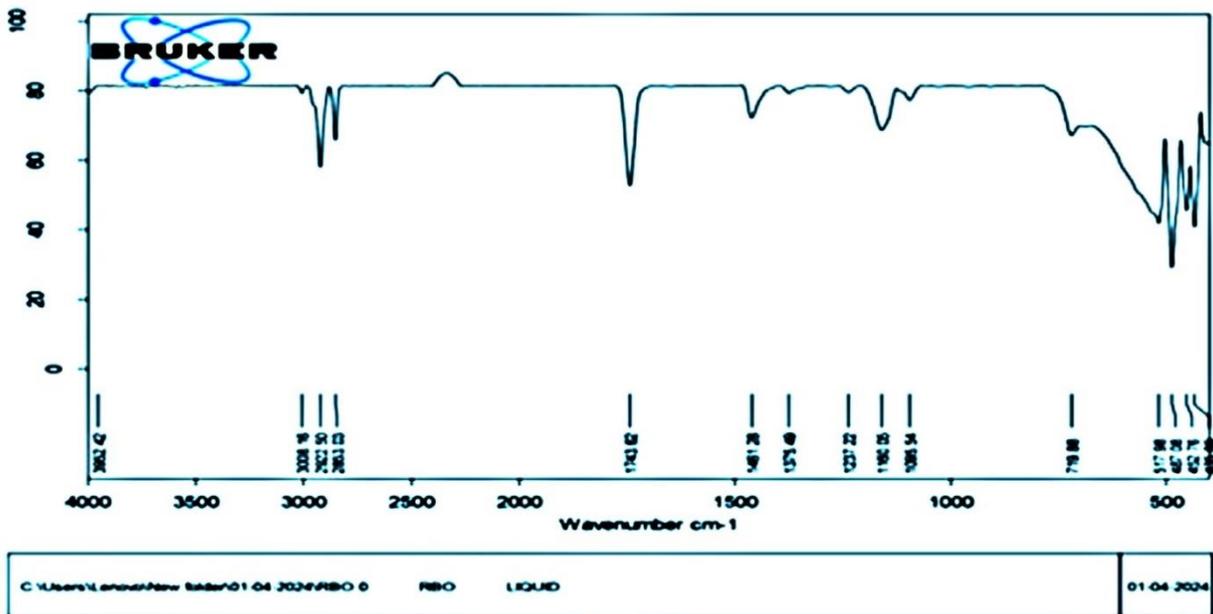
Wash each of the flasks with distilled water then with the help of a funnel, do the filtration with filter paper. Then take the sample for AAS.

IR SPECTROSCOPY RESULTS

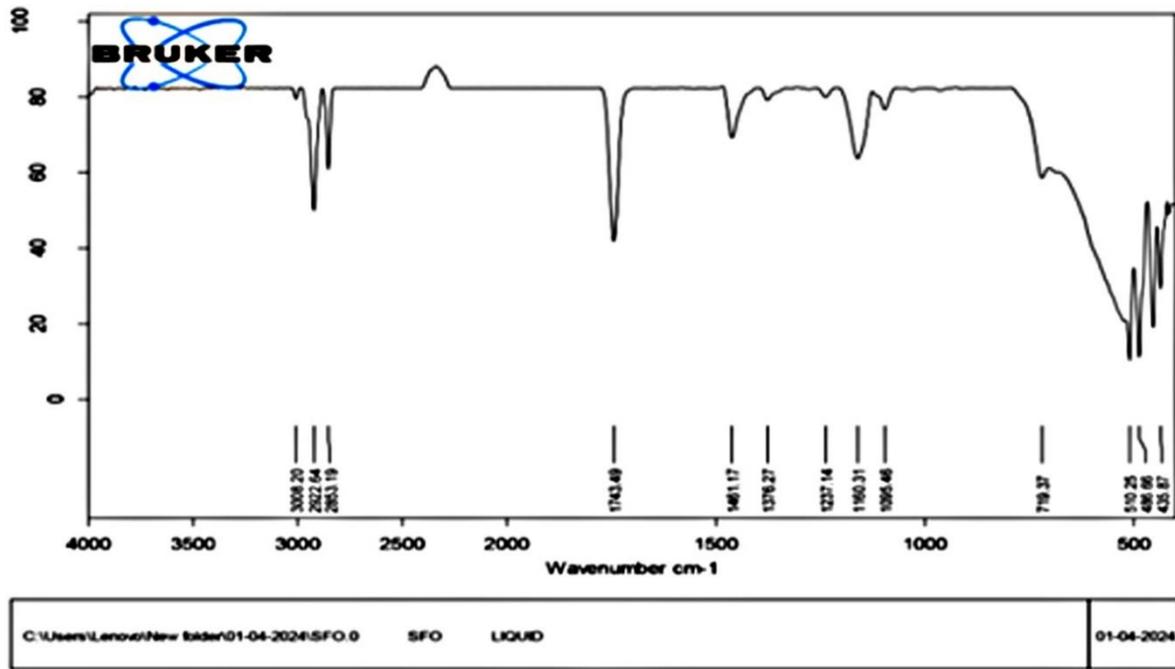
IR Spectroscopy of Groundnut oil



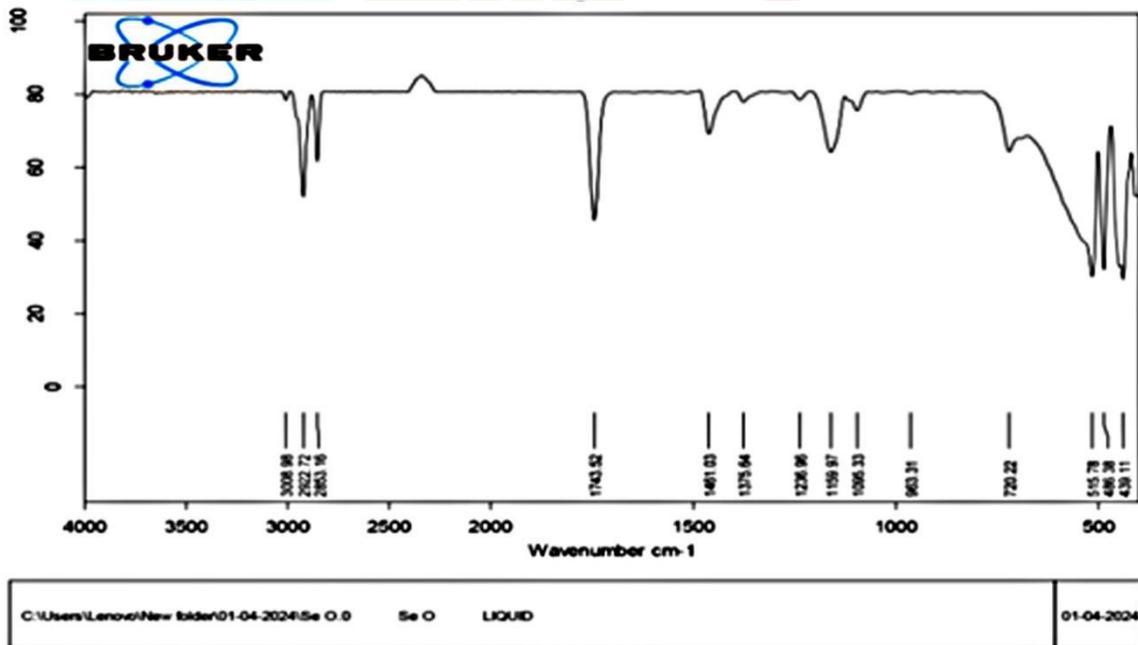
IR IR Spectroscopy of Rice bran oil



IR Spectroscopy of Sunflower oil



IR Spectroscopy of Sesame oil



IR Frequency Ranges:

IR Frequency Range of Groundnut oil

S.No	Wave number	Functiona l Group	Absorption Intensity	Class of Compounds	Mode of Vibration
01	3009	=C-H(cis)	Medium	Alkenes	Stretching
02	2923	C-H(CH ₂)	Very Strong	Alkyl	Stretching (Asymmetrical)
03	2853	C-H(CH ₂)	Strong	Aromatic	Stretching (Symmetrical)
04	1743	C=O(ester group)	Strong	Carbonyl	Stretching
05	1460	CH(CH ₂)-C-H(CH ₃).	Medium	Alkanes	Bending / Deformation
06	1374	C-H(CH ₃)	Medium	Alkanes	Bending (Symmetrical)
07	1240	C-H(CH ₂)	Medium	Ethers	Bending
08	1160	C-H(CH ₂)	Strong	Ethers	Bending
09	1117	C-O(ester group)	Medium	Ethers	Stretching
10	1095	C-O(ester group)	Medium	Ethers	Stretching
11	722	-(CH ₂ N)-HC=CH	Medium weak	Aromatic	Bending & out-of-plane vibration

IR Frequency Range of Rice bran oil

S.NO	Wave number	Functiona l Group	Absorption Intensity	Class of Compounds	Mode of Vibration
01	3952	O-H	Low	Phenolic	Stretching
02	3008	=C-H(cis)	Medium	Alkenes	Stretching
03	2923	C-H(CH ₂)	Very Strong	Alkyl	Stretching (Asymmetrical)
04	2853	C-H(CH ₂)	Very Strong	Aromatic	Stretching (Symmetrical)
05	1743	C=O(ester group)	Very Strong	Carbonyl	Stretching
06	1460	CH(CH ₂)-C-H(CH ₃).	Medium	Alkanes	Bending / Deformation
07	1374	C-H(CH ₃)	Medium	Alkanes	Bending (Symmetrical)
08	1240	C-H(CH ₂)	Medium	Ethers	Bending
09	1160	C-H(CH ₂)	Strong	Ethers	Bending
10	1119	C-O(ester group)	Medium	Ethers	Stretching
11	1096	C-O(ester group)	Medium	Ethers	Stretching
12	722	-(CH ₂ N)-HC=CH	Medium weak	Aromatic	Bending & out-of-plane vibration

IR Frequency Range of Sunflower oil

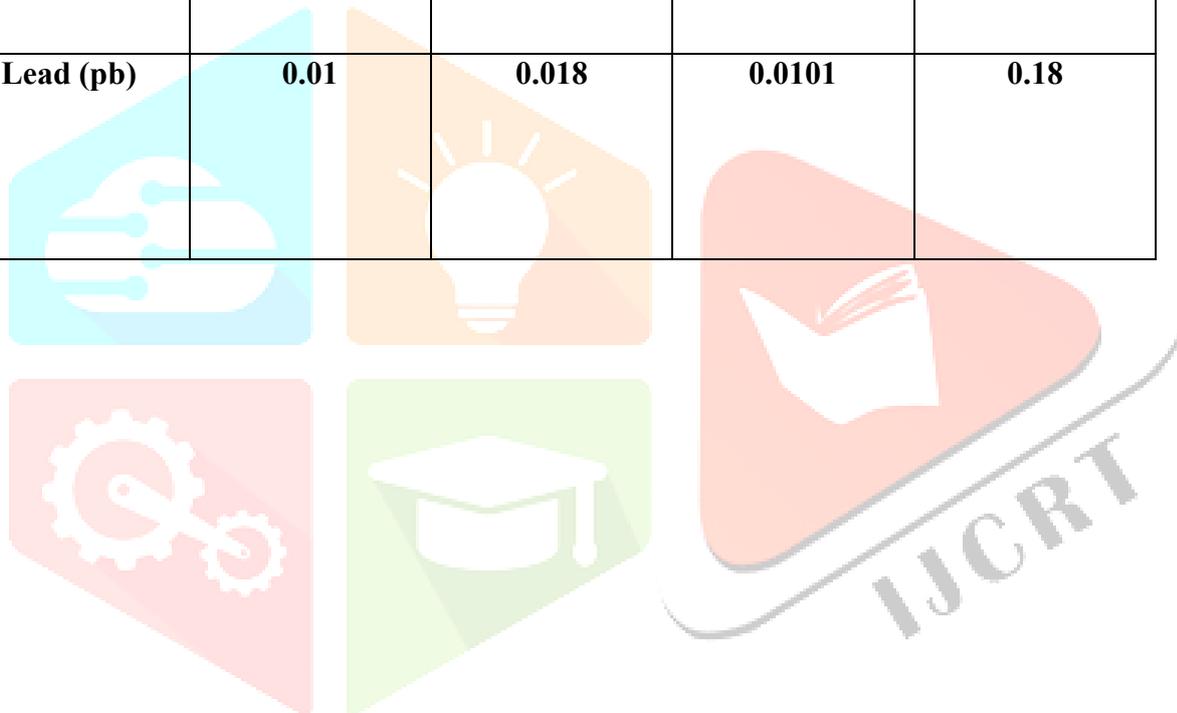
S.No .	Wave number	Functiona l Group	Absorption Intensity	Class of Compounds	Mode of Vibration
01	3008	=C-H(cis)	Medium	Alkenes	Stretching
02	2922	C-H(CH ₂)	Very Strong	Alkyl	Stretching (Asymmetrical)
03	2853	C-H(CH ₂)	Very Strong	Aromatic	Stretching (Symmetrical)
04	1743	C=O(ester group)	Very Strong	Carbonyl	Stretching
05	1461	CH(CH ₂)-C-H(CH ₃).	Medium	Alkanes	Bending / Deformation
06	1378	C-H(CH ₃)	Medium	Alkanes	Bending (Symmetrical)
07	1237	C-H(CH ₂)	Medium	Ethers	Bending
08	1160	C-H(CH ₂)	Strong	Ethers	Bending
09	1095	C-O(ester group)	Medium	Ethers	Stretching
10	722	C-O(ester group)	Medium weak	Aromatic	Bending & out-of-plane vibration
11	452	-(CH ₂ N)-HC=CH	Medium	Alkanes	Bending (Symmetrical)

IR Frequency Range of Sesame oil

S.No .	Wave number	Functiona l Group	Absorption Intensity	Class of Compounds	Mode of Vibration
01	3008	=C-H(cis)	Medium	Alkenes	Stretching
02	2923	-C- H(CH ₂)	Very Strong	Alkyl	Stretching (Asymmetrical)
03	2853	-C- H(CH ₂)	Very Strong	Aromatic	Stretching (Symmetrical)
04	1743	-C=O(ester group)	Very Strong	Carbonyl	Stretching
05	1461	-C- H(CH ₂) -C- H(CH ₃)	Medium	Alkanes	Bending / Deformation
06	1375	-C- H(CH ₃)	Medium	Alkanes	Bending (Symmetrical)
07	1237	-C- H(CH ₂)	Medium	Ethers	Bending
08	1160	-C- H(CH ₂)	Strong	Ethers	Bending
09	1095	-C-O(ester group)	Medium	Ethers	Stretching
10	722	-(CH ₂ N)- HC=CH	Medium weak	Aromatic	Bending & out-of-plane vibration
11	487	-C- H(CH ₃)	Medium	Alkanes	Bending (Symmetrical)
12	452	-C- H(CH ₃)	Medium	Alkanes	Bending (Symmetrical)

AAS:

Element	SFO (mg/ ml)	GO (mg/ ml)	RBO (mg/ ml)	Se o (mg/ ml)
Arsenic (Ar)	0.0012	0.0010	0.0019	0.0009
Cadmium (cd)	0.017	0.019	0.017	0.0087
Lead (pb)	0.01	0.018	0.0101	0.18

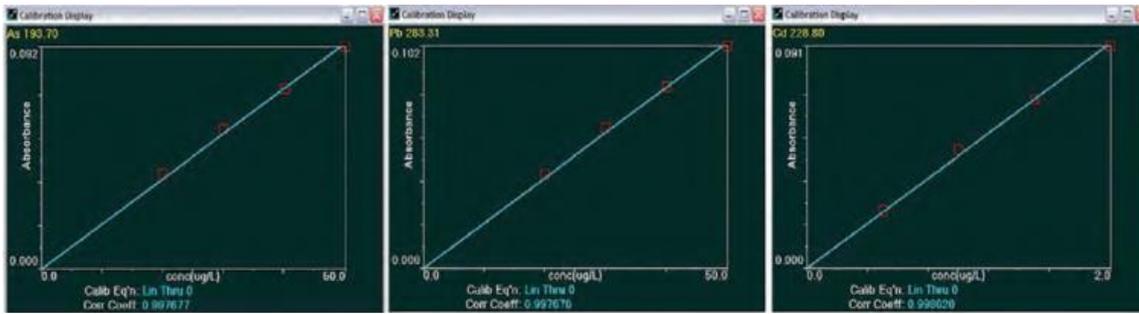


S.No.	Analyte	As	Ps	Cb
01	Wavenumber	193.7	283.3	228.8
02	Width of Slit {nm}	0.6	0.6	0.6
03	Type of Lamp	EDL	EDL	HCL
04	Sign	Area Peak	Area Peak	Area Peak
05	Time (sec)	2	2	2
06	Sample Volume (µl)	10	10	10
07	Diluent (µl)	5	5	4
08	Modifier Matrix	4 + 0.4 µg Mg	4 + 0.4 µg Mg	4Pd + 0.4 µg Mg
09	Matrix Volume (µg)	4	4	4
10	Temp of Injection (°C)	85	85	85
11	Pipet (%)	50	50	50
12	Equation of Calibration	Linear Zero	Linear Zero	Linear Zero
13	Concentration at Standard (µg/L)	0,10,15,20,25,30	0,10,15,20,25,30	0,0.2,0.4,0.6,0.8,1.0
14	Q.C Concentration (µg/L)	5	5	5
15	Spike Conc. (µg/L)	8	8	6
16	Drying 01	108 1 15	108 1 15	108 1 15
17	Drying 02	130 08 08	130 08 08	130 08 08
18	Drying 03	400 09 18	400 09 18	400 09 18
19	Pyrolysis	1000 10 15	1000 10 15	1000 10 15
20	Atomization	2200 0 3	2000 0 3	1900 0 2
21	Clean Out	2000 2 4	2000 2 4	2000 2 4

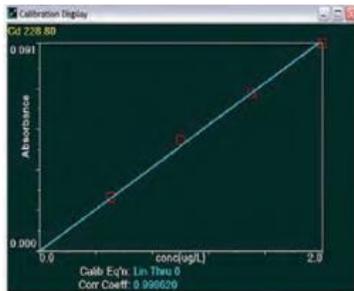
Groundnut oil

Sesame oil

Sunflower oil



Rice bran oil

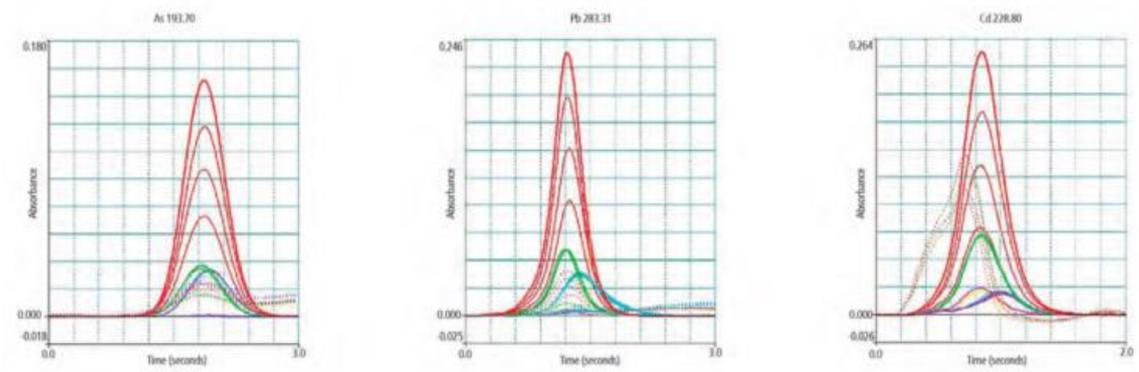


Calibration curve for Oil

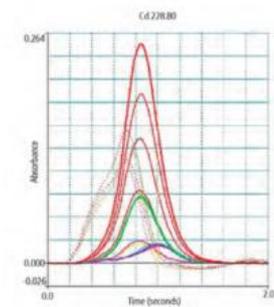
Groundnut oil

Sesame oil

Sunflower oil



Rice bran oil



Recovery percentage Peaks

Recoveries of Oils

S.No			% Recovery	
	Analyte	As	Pb	Cd
1	Q.C 1	103	109	108
2	Q.C 2	98.6	111	110
3	Q.C 3	105	108	111
4	Spike recovery of Groundnut Oil	94.1	105	108
5	Spike recovery of Sesame Oil	94.5	93.6	111
6	Spike recovery of Sunflower Oil	98.6	93.8	108
7	Spike recovery of Rice bran Oil	95.6	96.3	110

CONCLUSION

Presence of Phenolic group in Rice bran oil along with Olefins, FFA, Ester Carbonyl of Triglycerides compare to other oils. Alkanes are less in Groundnut oils when compare to Sesame-oil Triglycerols low concentration in Sunflower oil Carbonyl groups are present in all oils that is low level of MUFA and PUFA. Medium absorption is seen in alkanes.

SFO was analyzed using frequency regions of 3025–3000 and 1400–985 cm^{-1} . FTIR technique can be considered as green analytical tools for classification and quantification of Unsaturation of oils as adulterants.

To analyse TAGs profile of fatty acids in four different oils by using FTIR. The analysis confirms the presence of functional groups in the Peanut, Sunflower, Rice bran oil and Sesame oils all absorptions in the region 722-3000 cm^{-1} corresponding to C-H, H-C-H, -CH₃, C-O and O-C=O, C=C with well resolved stretching frequencies was noticed. The absence of doublet of doublet or multiplet peaks and appearance in shielded region in the span of 5.30-0.8 ppm reveals the double bonds both in mono and poly unsaturated fatty acids retains in Z or cis conformation. The slight variation of peak values in FTIR between saturated and unsaturated fatty acids. The used methodology is an interesting and quite valuable for the authentication of edible oils both in food industry, quality control as well as human health. Its main advantage is simplicity, speed, less time consume high-sensitivity and no require for sample Pre-treatment. The results found that % Recovery id more in Sesame oil when compared to other oils.

Heavy metals are not detected in oils but after the spike recovery the Heavy metals are detected, after expiry don't consume oils they are harmful.

ABBREVIATION USED:

FT-IR: Fourier Transform Infrared,

AAS: Atomic Absorption Spectroscopy,

As- Arensic

Pb- Lead,

Cd- Cadmium

SeO -Sesame Oil

SFO-Sunflower Oil

GO-Groundnut Oil

RBO - Rice Bran Oil

L- Liter

µm- Micrometer

µl- Microliter

Mm - Millimeter

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