

# Pharmaceutical Research: Recent Advances and Trends

Vol. 9

*Edited by Prof. Kevin Fernandez*



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# **Pharmaceutical Research: Recent Advances and Trends**

**Vol. 9**

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**Prof. Kevin Fernandez**

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## ABOUT THE EDITOR



**Prof. Kevin Fernandez**  
AIMST University, Malaysia.

I, Prof (Air Cmdre) Kevin Fernandez VSM, am a medical graduate from Armed Forces Medical College Pune. (1966-1970). After my graduation I joined the Indian Air Force as a Medical Officer in Jan 1971. While working in the Indian Force, I completed my DPH (1976), DIH (1977), MD (PSM)1979, MNAMS (1981), CERT in APPLIED NUTRITION from NIN Hyderabad (1982). I served the Indian Air Force for 37 years, during which time I held several academic, clinical, and administrative roles. In recognition of my meritorious services the President of India awarded the Vishista Sewa Medal (VSM) on 26 Jan 2007. From 2007 to 2016, I was Head of Community Medicine Department at SKN Medical college, Pune. Setting up a full-fledged department from scratch, Urban and Rural Training centers and Post graduation course as well as facing multiple MCI inspections were challenges, I faced successfully. During this period, I was Examiner both for UG/PG examinations of Pune, Rajiv Gandhi, and Sardar Patil universities. I was appointed Reviewer of MJAFI, MJ of D Y Patil University and Chairman of IEC of AFMC, Pune. I have been awarded two Fellowships, FISCD and FIPHA. Since 2017 to date, I am Head of Community Medicine at Faculty of Medicine, AIMST University, Malaysia. Now at 75, with over 50 years of experience, I try to impart knowledge to young International Medical students. I am actively involved in New Program Initiatives here (Bachelor of Bio Medical Sciences and Master of Health Care Management). I am a firm believer of all round development of students during their medical studies, not just academics. I am a Distinguished Toast Master (DTM). Toastmasters International is a non-Profit international organization which promotes public speaking and leadership skills to youngsters. My areas of interest are Nutrition, occupational Health and Communication skills.

## **PREFACE**

*This book covers key areas of pharmaceutical research. The contributions by the authors include HPLC methods, electrospray ionization, solid-phase extraction, liquid-solid extraction, caffeine analysis, near-infrared spectroscopy, Aegle marmelos polysaccharide, natural matrix former, advanced drug delivery systems, DSC spectra, UV spectrophotometric method, Maidenhair fern, androgenic alopecia, nephrolithiasis, NP-mediated drug delivery, multidrug resistance, rheumatoid arthritis, epidermal growth factor receptor 2, somatostatin receptors, lipid nanoparticles, hematopoietic stem and progenitor cells, neurofibrillary tangles, oral floating beads, non-steroidal anti-inflammatory drugs, gastric residence times, floating drug dosage systems, colloidal barrier systems, alginate beads, emulsion gelation method, febuxostat, hyperuricemia, gouty arthritis, accurate drug measurement, liquid chromatography, mass spectrometry, personalized medicine, therapeutic drug monitoring, Bayesian estimation, point-of-care testing, expanded monitoring targets, pharmacogenomics integration, paraffin oil adulteration, FTIR spectroscopy, Beer-Lambert law. This book contains various materials suitable for students, researchers, and academicians in the fields of pharmaceutical research.*

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# Detection of Paraffin Oil Adulteration in Edible Oils Using FTIR Spectroscopy

Andrei A. Bunaciu <sup>a\*</sup> and Hassan Y. Aboul-Enein <sup>b\*</sup>

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## ABSTRACT

Edible oils are commonly used as salad, cooking or frying, or in fabricating food products. From the standpoint of nutrition, they are major, but there has been concern over their purity from ancient times. Adulterating high-priced oil with low-priced oil is a significant problem due to its increased demand in domestic and foreign markets. Customers' health may suffer as a result. Therefore, authentication and adulteration prevention are urgently needed for customers' benefit. The application of mid-infrared or near-infrared spectroscopy of molecular vibrations has become more prevalent in the characterization of various chemicals, such as edible oil, intending to track alterations and identify counterfeit modifications. A novel method to assess the alteration of rapeseed oil (RSO), sunflower oil (SFO), maize germ oil (CGO), and extra virgin olive oil (EVOO) with mineral oil, paraffin oil (PO), was established. Using ATR spectra, a Fourier transform infrared (FT-IR) spectrometric technique was created to evaluate edible oil adulteration quickly. The findings show that the suggested approach effectively detects paraffin oil in adulterated EVOO, SFO, RSO and CGO.

*Keywords:* FTIR spectroscopy; edible oils analysis; adulteration food; paraffin oil adulteration.

## 1. INTRODUCTION

"Edible oils" (also named "cooking oils") are a class of fatty liquids that are obtained through physical extraction from various crops, animal tissues, or microorganisms [1]. The majority of edible oils are produced from seeds, while olive, coconut, and

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<sup>a</sup> S.C. AAB\_IR Research S.R.L., 9-11A Gloriei Street, Bragadiru – Ilfov District-077025, Romania.

<sup>b</sup> Pharmaceutical and Medicinal Chemistry Department, Pharmaceutical and Drug Industries Research Division, National Research Center, Cairo 12622, Egypt.

\*Corresponding authors: E-mail: [aabunaciu@gmail.com](mailto:aabunaciu@gmail.com), [haboulenein@yahoo.com](mailto:haboulenein@yahoo.com);

palm oils are extracted from fruits, according to an investigation into the main producers of oils [2].

Many different types of vegetable oils can be included in a person's diet, making them among the most significant and widely used agricultural products in the world. It is frequently referred to that edible fats and oils are vital parts of the diet for humans. Since the body cannot generate these fatty acids, an adult needs approximately 5 g of unsaturated and linolenic fatty acids daily [3].

The food industry places a great deal of importance on food adulteration and the ensuing assessment of authenticity. This has led to significant concerns among food manufacturers, particularly in the wake of significant scandals like the European scandal involving horse flesh in 2013, and the melamine crisis in China in 2007 [4-7]. A sensitive, precise, and appropriate technique to identify the adulteration is crucial.

According to the ICC Counterfeiting Intelligence Bureau [8], adulteration is a widespread problem that affects a variety of products and is alarmingly expanding to electrical equipment, cigarettes, and even medications. When hazardous substances are offered to uninformed customers, adulteration occurs and the human organism gets incorporated with them.

Vibrational spectroscopy techniques (near-infrared (NIR), mid-infrared (MIR), and Raman) are among the variety of analytical tools used [9-11]. These techniques are currently gaining popularity and are predicated on the idea that food products have a unique "fingerprint" that is derived from a particular composition of characteristics. Additionally, vibrational spectroscopy methods provide quick, nondestructive, and affordable analysis.

Spectrophotometric methods are a method of choice for the targeted identification of altered products due to their speed and little or nonexistent sample preparation requirements. Given the complexity of the material to be studied and the knowledge that the recorded spectra are dependent on contributions from all the elements, interpretation to detect moderate degrees of falsification may be difficult, but not impossible [12-17].

The well-known vegetable oil, *extra-virgin olive oil* (EVOO), is only produced from fruits using mechanical and physical techniques [18]. Ancient Greeks esteemed olive oil as the "elixir of youth and health" and acknowledged its nutritional benefits for ages. The primary fat source utilized in Mediterranean nations is olive oil. As the primary source of fat in the traditional Mediterranean diet, olive oil is a crucial component of the plan. Therefore, one of the most significant characteristics that set the Mediterranean Diet apart from this ancestral dietary pattern is its high-fat content (up to 40% of total energy) [19]. Several techniques for determining olive oil adulteration using infrared spectroscopy are described in the published data [20,21]. Critical parameters for determining the purity of EVOO are presented in different reviews [21-24].

*Sunflowers*, (SFO, *Helianthus annuus L.*) are cultivated worldwide because of their great adaptability, high mechanization, and cheap labor requirements. Sunflower oil is one of the most widely used edible oils, which can be produced by mechanically crushing sunflower seeds. The oil is mostly used for shortening and cooking, as well as for making margarine and salad dressing [25]. Traditional sunflower oil has a low SFA (saturated fatty acids) concentration and a high PUFA (polyunsaturated fatty acids) content, primarily LA (linoleic acid). However, there are now types with mid- and high-oleic content. Tocopherols and plant sterols make up sunflower's nonglyceridic composition [26]. According to Raß [27] and Rauf [28], sunflower plant seed is regarded as one of the most significant varieties within the oilseed family because of its high oil content, ranging from 36 to 50%. Sunflower oil (SFO) is one of the most important edible oils, together with soybean and palm oils [28]. To identify when sunflower oil has been adulterated with other oils, several reviews are presented [29-31].

A by-product of milling corn oil is *corn germ oil* (CGO). Most corn collected serves as feed, but since additional bioethanol is produced, a growing amount of maize is being milled [32]. As the corn processing business has grown, oil has emerged as one of the key byproducts of deep-processing corn. The lipid concentration of corn germ typically ranges from 18% to 41%, making up over 80% of the total lipid content of maize. Thus, corn germ oil, sometimes referred to as commercial corn oil, is mostly derived from maize germ. One of the nutritious vegetable oils is corn germ oil, which has a high percentage of unsaturated fatty acids ( $\geq 80\%$ ), particularly linoleic acid ( $\geq 50\%$ ), and a low level of trans fatty acids ( $< 0.3\%$ ) [33].

Research in this field offers several techniques for determining corn germ oil content (CGO), including a pertinent review for discovering if corn germ oil has been adulterated [15,34].

*Rapeseed oil*, (RSO, Brassica Napus) is the most important oilseed crop and a major source of edible oils (EOs) in China. The third most popular cooking oil worldwide is rapeseed oil. It is highly valued for its nutritional richness because of its high level of unsaturated fatty acids, particularly polyunsaturated fatty acids. The benefits of eating a diet high in unsaturated fatty acids are becoming more and more clear. The nutritional components of rapeseed oil and the precise effectiveness of the nutrients are yet unknown, although its intake is widespread throughout the world [35]. Worldwide cultivation of rapeseed is practised because it is regarded as the most important oil crop following soybeans [36]. To identify when rapeseed oil has been adulterated, several reviews are presented [34, 37-41].

Mineral oils are widely used in a variety of consumer goods, including medications and cosmetics [42]. To hydrate, smooth, and protect the skin and hair, paraffin oil is frequently found in body oils, moisturizers, lip balms, and hair care products. It is regarded as safe for use in skincare and cosmetic applications. Both mineral oil saturated hydrocarbons (MOSH) and mineral oil aromatic hydrocarbons (MAAH) make up mineral oils like paraffinum liquidum or white oil, PO. Critical parameters for determining the purity of PO are presented in different reviews [29, 43-46].

The goal of one of our previous works was to use Lambert-Beer's law to reduce the need for sample preparation before FTIR analysis and so to realize a new spectrophotometric method for the speedy and more precise determination of two varieties of commercial products including corn germ oil (CGO) and extra virgin olive oil (EVOO) laced with paraffin oil (PO) [47]. Now in this chapter, we extended the experiments performed before by adding some other edible oils, rapeseed oil (RSO) and sunflower oil (SFO).

## **2. MATERIALS AND METHODS**

### **2.1 Apparatus**

The Spectrum1000 FT-IR spectrometer (Perkin Elmer Co., Beaconsfield, Bucks, UK) fitted with Spectrum for Windows v.5.01 was used to collect the data. QUANT+ expert v.4.51 and Spectrum Beer's law v.10.7.2.1360 (Perkin Elmer Co. UK) were the commercial programs utilized to produce an analysis for the main component analysis.

### **2.2 Reagents and Materials**

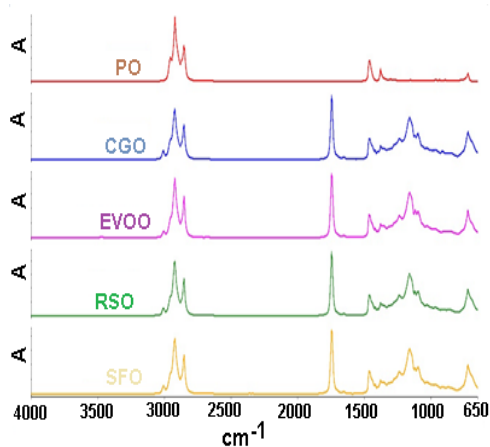
The commercial products were: extra virgin olive oil (EVOO) from Primadonna, Italy (a blend of olives from the European Union and those not), corn germ oil (CGO) from Italy (obtained solely by physical means, pressing without the use of solvents), sunflower oil (SFO) from Floriol, Romania, rapeseed oil (RSO), and paraffin oil (PO) from Vitalia Pharma Ploiesti district, Romania. All of these products were purchased from Romanian retail stores. The solvents, namely acetone and hexane, were procured from Aldrich Chemical Co. (Milwaukee, WI) and were of reagent grade.

### **2.3 Recommended Procedures**

After all samples were homogenized, a tiny quantity of each was applied to the ATR crystal to ensure complete coverage. Before the first measurement day for each sample, the absorbance spectrum was measured against an ATR cell that had been dried up and empty. Averaging 16 scans at a resolution of 4 or 8  $\text{cm}^{-1}$ , the spectra were scanned in triplicate at room temperature spanning the wavenumber interval of 4000–650  $\text{cm}^{-1}$ . The spectra were then averaged before calculations. Following the acquisition of each spectrum, cellulose tissue soaked in hexane was used to clean the ATR crystal, which was subsequently rinsed with acetone. Paraffin oil stock solutions cover the detection range of 0.0, 1.0, 2.0, 3.0, 4.0, 5.0, 6.0, 7.0, 8.0, 9.0, 10.0, 20.0, 30.0, 40.0, 50.0, 60.0, 70.0, 80.0, 90.0 and 100.0% (v/v) in edible oils of EVOO, SFO, RSO, and CGO were made, respectively. In a 100 ml volumetric flask, the volumes of paraffin oil stock solutions were added to the mark with edible oils under the study to obtain the solutions used for calibration.

### 3. RESULTS AND DISCUSSION

Fig. 1 compares the FTIR spectra of paraffin (PO), corn germ (CGO), olive (EVOO), rapeseed (RSO), and sunflower oil (SFO) [47].



**Fig. 1. ATR spectra of extra virgin olive oil (EVOO), corn germ oil (CGO), rapeseed oil (RSO), sunflower oil (SFO), and paraffin oil (PO)**

This figure shows that the spectra of EVOO, SFO, RSO, and CGO do not differ significantly.

The single biggest distinction is the location of the peak attributed to the *cis* double bond [48], which can be observed at around 3004.3  $\text{cm}^{-1}$  in the EVOO spectrum, 3007.5  $\text{cm}^{-1}$  for CGO samples, and 3006.3  $\text{cm}^{-1}$  for SFO and RSO, as can be seen in detail in Fig. 2 [47].

To get a better idea of how much EVOO, SFO, RSO, and CGO were present, the peak at 1743  $\text{cm}^{-1}$  was used. This peak is related to the stretching vibrations of the ester carbonyl band, which can be seen in more detail in Fig. 3 [47].

To get a better idea of how much PO is present, the peak at 2954  $\text{cm}^{-1}$  was used for quantitative determination, the distinctive peak attributed to the methyl asymmetrical stretching vibration [49], as shown in detail in Fig. 3.

The distinctive peak in the PO spectrum, located at 2954  $\text{cm}^{-1}$ , was assigned to the methyl asymmetric stretching vibration [49].

The peak located at approximately 1743  $\text{cm}^{-1}$  served to determine the presence of the other edible oils more quantitatively. This peak was linked to the stretching vibrations of the ester carbonyl band [49].

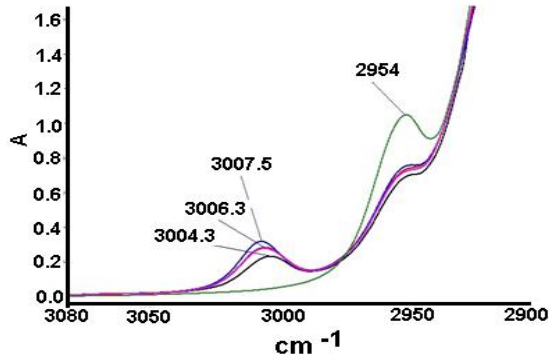
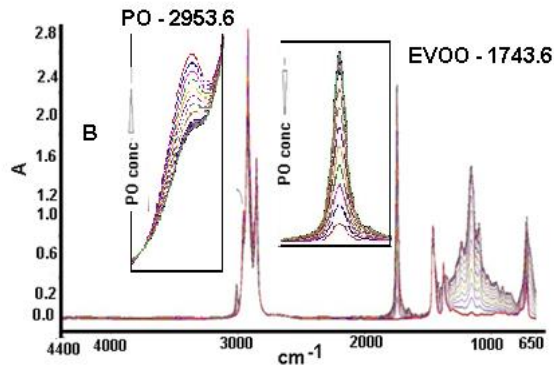
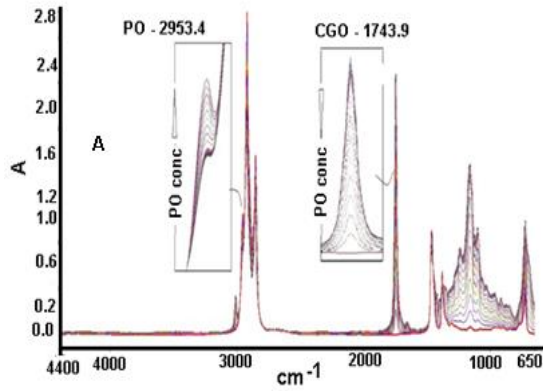
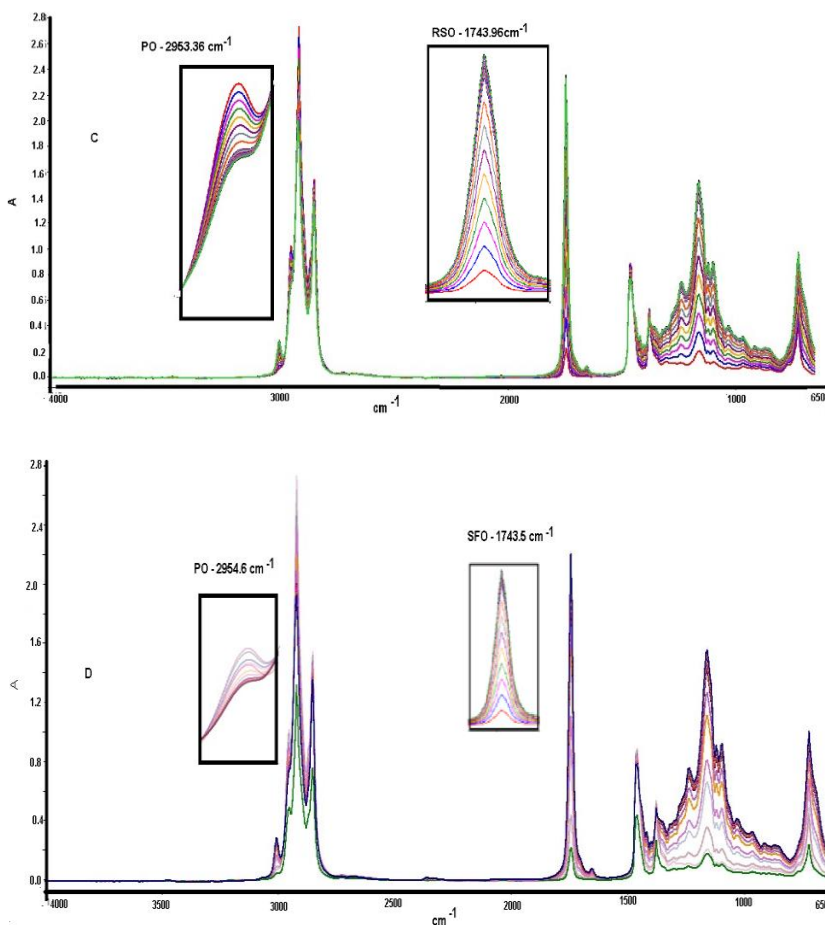


Fig. 2. FTIR peaks used for the quantitative determination of EVOO (purple line), SFO and RSO (red line), and CGO (black line) against PO (green line)





**Fig. 3. FT-IR spectra regions for adulterating edible oils with paraffin oil**

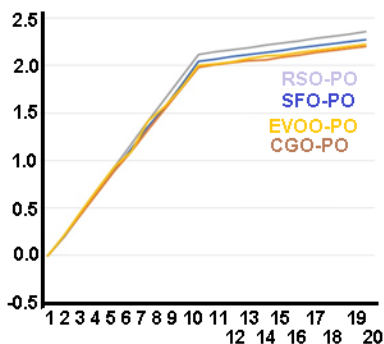
- A – CGO – corn germ oil;*
- B – EVOO – extra virgin olive oil;*
- C – RSO – rapeseed oil;*
- D – SFO – sunflower oil*

We obtained calibration curves for the quantitative determination of the adulteration by using the standards we had prepared, as mentioned in the Recommended procedures.

Two linear domains, each with its linear equation and slopes that change with concentration, may be distinguished from the calibration graphs: one for

adulteration, which has a range of 1–10%, and another for adulteration, which has a range of 10–100%.

Table 1 displays the calibration graph results for the binary mixes, measurements being performed at 1743.6 cm<sup>-1</sup>.



**Fig. 4. Calibration graph for Paraffin oil determination in mixtures with edible oils at 1743.6 cm<sup>-1</sup>**

**Table 1. Calibration results at 1743.6 cm<sup>-1</sup>**

Parameter	Binary mixtures, 0-10% (v/v)			
	CGO-PO	EVOO-PO	SFO-PO	RSO-PO
Slope	0.0228	0.0216	0.0235	0.0223
Intercept	-00075	-0.0367	-0.0141	-0.0105
R <sup>2</sup>	0.9998	0.9951	0.9998	0.9983
Parameter	Binary mixtures, 10-100% (v/v)			
	CGO-PO	EVOO-PO	SFO-PO	RSO-PO
Slope	0.0221	0.0221	0.0237	0.0228
Intercept	1.7645	1.8025	1.7038	1.8108
R <sup>2</sup>	0.9999	0.9999	0.9999	0.9964

This table highlights the similarities in the results; thus, we recommend applying this method - especially when determining the mixture's principal component, EVOO, RSO, SFO, or CGO - because of the smaller RSD value (< 1.0%). Even though the RSD value is a little higher, the results for the quantity of paraffin oil determination are also good.

The two variants were employed for the quantitative assessments of edible oil adulteration with mineral oil (paraffin oil). Table 2 shows the averages of five determinations for the same samples that we prepared, with the following amounts of adulterant: 3.5 (3.5% PO + 96.5% EVOO, 3.5% PO + 96.5% RSO, 3.5% PO + 96.5% SFO and respectively 3.5% PO + 96.5% CGO), 15 (15.0% PO + 85.0%

EVOO, 15.0% PO + 85.0% RSO, 15.0% PO + 85.0% SFO, and respectively 15.0% PO + 85.0% CGO), and 25 (25.0% PO + 75.0% EVOO, 25.0% PO + 75.0% RSO, 25.0% PO + 75.0% SFO and respectively 25.0% PO + 75.0% CGO).

**Table 2. Quantitative assessment of paraffin oil adulteration in different edible oils**

Taken (%)	Found at 1743 cm <sup>-1</sup>		Found at 2956 cm <sup>-1</sup>	
	%	RSD (%)	%	RSD (%)
<b>Edible oil</b>	<b>EVOO</b>			
3.5	96.53	0.32	3.44	0.88
15.0	85.10	0.24	15.08	1.15
25.0	74.76	0.48	25.34	1.78
<b>Edible oil</b>	<b>SFO</b>			
3.5	96.48	0.48	3.52	0.79
15.0	85.06	0.33	14.96	1.09
25.0	75.09	0.29	24.93	1.35
<b>Edible oil</b>	<b>RSO</b>			
3.5	96.49	0.32	3.53	0.85
15.0	85.08	0.27	15.05	1.15
25.0	74.85	0.33	25.22	0.99
<b>Edible oil</b>	<b>CGO</b>			
3.5	96.51	0.33	3.57	0.70
15.0	84.64	0.27	15.58	1.06
25.0	74.88	0.59	25.56	1.42

As can be seen in Table 2, the results are very similar, and so, we suggest the use of this method, especially for the determination of the major component of the mixture, EVOO or CGO, because of the smaller value of RSD (< 1.0%). Also, the results are good for determining paraffin oil quantity, even if the RSD value is a little higher.

#### 4. CONCLUSION

Because of the unique fingerprint of the spectra, vibrational spectroscopy has been used in recent years to control the quality of edible oils. Oil characterization and adulteration detection are made possible by FTIR, which is widely used in laboratories and industry. Information regarding the composition, place of origin, processing, and storage is provided by this method.

It is evident that when mixed with mineral oil, PO, FT-IR spectrometry may directly determine the contents of EVOO, SFO, RSO, and CGO in a variety of mixes. Compared to chromatographic procedures, the suggested method is straightforward, accurate, and doesn't require a lot of time thanks to the commercial software that uses the Beer-Lambert law. Including sample preparation and spectrum capture, quantification could be completed in five to ten minutes.

Our suggested quantification techniques are accurate, exact, specific, and labor-saving (fast and easy to execute), which makes them suitable as screening tests in the routine analysis of edible oils adulterated with paraffin oil, according to the validation results.

## **DISCLAIMER (ARTIFICIAL INTELLIGENCE)**

Authors hereby declare that NO generative AI technologies such as Large Language Models (ChatGPT, COPILOT, etc.) and text-to-image generators have been used during the writing or editing of this manuscript.

## **ETHICAL APPROVAL**

This article does not contain any studies with human participants or animals performed by any of the authors.

## **COMPETING INTERESTS**

Authors have declared that no competing interests exist.

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**Biography of author(s)**



**Andrei A. Bunaciu**

S.C. AAB\_IR Research S.R.L., 9-11A Gloriei Street, Bragadiru – Ilfov District-077025, Romania.

He graduated from the Politehnica University of Bucharest in 1979. He received his PhD from the Politehnica University of Bucharest, Faculty of Chemistry, Department of Analytical Chemistry in ion-selective membrane electrodes applied in pharmaceutical analysis in 1990. For several years he worked as a senior researcher at the Institute of Chemical and Pharmaceutical Research, Romanian Intelligence Service, and in the Vibrational Spectroscopy Department at SCIENT - Research Center for Instrumental Analysis, Tâncăbești - Snagov. Now he is the C.E.O. and senior researcher at AAB\_IR research company from Bragadiru - ROMANIA. His research interests concern molecular recognition of pharmaceutical and biological compounds, using infrared spectroscopy techniques. He is the author/co-author of 96 publications in biomedical, pharmaceutical, and food analysis, including a book "VIBRATIONAL SPECTROSCOPY APPLICATIONS IN BIOMEDICAL, PHARMACEUTICAL AND FOOD SCIENCES", published in 2020 by Elsevier, Inc., as well as two chapters: "VIBRATIONAL SPECTROSCOPY APPLICATIONS IN DRUG-ANALYSIS", in Encyclopedia of Spectroscopy and Spectrometry, 3rd Edition, Lindon, J.C., Tranter, G.E. and Koppenaal, D.W. (eds.), Oxford; Elsevier, 2017, vol. 4, pp. 575-581, and respectively "RECENT APPLICATIONS OF INFRARED SPECTROSCOPY IN BIOMEDICAL ANALYSIS", in FTIR Spectroscopy: Advances in Research and Applications, edited by Adriana S. Franca and Leandro S. Oliveira, Nova Publishers, NY, USA, 2022, pp. 575-581.



**Professor Hassan Y. Aboul-Enein**

Pharmaceutical and Medicinal Chemistry Department, Pharmaceutical and Drug Industries Research Division, National Research Center, Cairo 12622, Egypt.

He received his first degree from Cairo University in 1964 and then went on to study in the United States, receiving a PhD in Medicinal Chemistry from the University of Mississippi in 1971. In 2000, he was also awarded a DSc from the University of Bucharest, Romania. He was an assistant professor at the University of Alabama in Birmingham and the University of Iowa before joining the pharmaceutical industry in the United States. He has also occupied several posts outside the United States, including at King Saud University and later at King Faisal Specialist Hospital and Research Centre in Riyadh, Saudi Arabia. He was a visiting professor at the University of Sydney, the University of Nebraska, Ain Shams University, University Sains Malaysia, Shandong University China, the University of Sharjah, University Teknologi Malaysia (UTM), University of Bechar, Bechar, Algeria and Mahatma Gandhi University, Kottayam, India. Currently, he is an Emeritus Professor at the National Research Center in Cairo. He is a consultant for several academic institutions in Malaysia, China, the United States, Turkey, Algeria, and Australia. He is also on the WHO-UN Expert Advisory Panel on International Pharmacopoeia and Pharmaceutical Preparations. Moreover, he is on the editorial board of several peer-reviewed journals.

His research interests are in the areas of pharmaceutical and biomedical analysis and he has published, over the course of his long career, more than 1200 scientific papers on pharmaceutical and biomedical analysis, in addition to 10 books and numerous presentations at international scientific conferences. He holds a US Patent and has received various honors and awards.

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## **London Kolkata Tarakeswar**

**India:** Guest House Road, Street no - 1/6, Hooghly, West Bengal, India (Reg. Address),  
Diamond Heritage Building, 16, Strand Road, Kolkata, 700001 West Bengal, India (Corporate Address),  
Tele: +91 7439016438 | +91 9748770553, Email: [director@bookpi.org](mailto:director@bookpi.org),  
**(Headquarters)**

**UK:** 27 Old Gloucester Street London WC1N 3AX, UK,  
Fax: +44 20-3031-1429, Email: [director@bookpi.org](mailto:director@bookpi.org),  
**(Branch office)**