PRODUCTION AND CHARACTERIZATION OF EXTRACTION OIL FROM NATURAL SPICES: A COMPARISON STUDY WITH FUNCTIONAL GROUP CONTENT OF *ZEA MAY* AND *ELAEIS GUINEENSIS JACQ. OIL*

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ABTRACT

Research in finding more sources for specialty oils have been actively conducted nowadays. In this research four spices selected as a raw materials which is *Pimpinella Anisum,* S*esamum indicum, Syzygium aromaticum*, and *Cuminum cyminum*. This spices was selected because of it is easily find in our local market. This research is to analyze the specialty oil produces and also the functional group in each of the sample. This analysis was conducted in Universiti Malaysia Pahang (UMP) laboratory using the Fourier Transform Infrared Transmitter (FTIR) unit. Most important step in this research is the analysis and comparison with the commodity oil such as palm oil and maize oil. This is because from the comparison the new sources of oil can be recognize and this oil will be the alternative to the commodity oil. Based on the result of this research, *sesamum indicum* produces the highest percentage of specialty oil (46.06%) (average), compare to other spices and the lowest percentage is cuminuim cyminum (10.78%) (average). Comparisons for the functional group indicate that sesamum indicum specialty oil gives nearly similar IR spectrum with the palm oil and maize oil. The market value for this specialty oil is increasing in many part of the world as for the increasing in its application, the development of this specialty oil must be considered because of its potential to be commercialized.

ABSTRAK

Penyelidikan untuk mencari sumber baru terutama dari minyak istemewa semakin banyak dilakukan pada masa ini. Di dalam kajian ini, empat bahan asas telah digunakan seperti jintan manis, bijan hitam, bunga cengkih, dan juga jintan putih. Bahan asas ini dipilih berdasarkan keadaannya yang mudah di jumpai dipasaran tempatan. Kajian ini juga adalah untuk menganalisa kandungan minyak istimewa yang terhasil dan kumpulan berfungsi minyak yang terhasil. Analisa ini dilakukan di makmal Universiti Malaysia Pahang dengan menggunakan Fourier Transforms Infrared Transmiter (FTIR) unit. Perkara yang paling penting di dalam kajian ini adalah mengenal pasti ciri-ciri, menganalisa dan juga membandingkan minyak istimewa dengan minyak komersil yang lain. Ini bertujuan bagi mengenal pasti kewujudan minyak alternatif lain sebagai sumber kepada minyak komersil yang ada sekarang. Berdasarkan daripada keputusan kajian ini, minyak bijan hitam menghasilkan peratus kandungan minyak yang tertinggi (46.06%) berbanding dengan minyak yang lain. Manakala minyak jintan putih menghasilkan peratus kandungan minyak yang terendah (10.78%). Perbandingan kumpulan berfungsi pula menunjukkan minyak bijan hitam memberikan IR spektrum yang hampir sama dengan minyak kelapa sawit dan juga hampir sama dengan minyak jagung. Nilai pasaran untuk minyak istemewa sekarang meningkat di seluruh dunia selaras dengan aplikasi minyak istimewa yang pelbagai. Pembagunan dalam penyelidikan minyak istimewa haruslah di teruskan kerana minyak istimewa mempunyai potensi yang besar untuk dikomersilkan.

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CHAPTER 1

INTRODUCTION

1.1 Background of research

Research in specialty oil are very important now days because specialty oil can be alternative way to replace synthetic chemical in many field of industries such as cosmetic, medicine, and also health. Although it is only can be produce in a small amount compare to other commodity oil such as palm oil, soy bean oil, and maize oil but specialty oil have higher market price due to their limited usage in certain field such as medicine and health.

In this research, specialty oil is extracted from spices such as *Pimpinella Anisum,* S*esamum indicum, Syzygium aromaticum*, and *Cuminum cyminum.* This specialty oil is not suitable for cooking oil because it produced in small amount only.

1.2 Problem statement

Specialty oil researches are very important now days because specialty oil can be alternative way to replace synthetic chemical in many field of industries such as cosmetic, medicine, and also health.

Besides the usages in the cosmetic, medicine and health industries, the specialty oil also have a potential as the storage sources to the common commodity oil such as palm oil, soy bean oil, and maize oil. Nowadays prices of commodity oil are increase. This is due to the high demand of the raw material (commodity oil) in order to produce biodiesel. Research on other storage sources must be continued to get diverse sources.

1.3 Objective of research

The objectives of this research are:

i. To produce specialty oil from spices (*Pimpinella Anisum,* S*esamum indicum, Syzygium aromaticum*, and *Cuminum cyminum*).
ii. To characterize the extraction oil by their physical characteristic.
iii. To identify the functional group of each specialty oil.
iv. To compare the functional group specialty oil with the commodity oil (palm oil and maize oil).

1.4 Scope of Research

i. Produce specialty oil from the suitable and potentials spices (*Pimpinella Anisum,* S*esamum indicum, Syzygium aromaticum*, and *Cuminum cyminum*) using Soxhlect Extraction unit based on solid-liquid extraction principle.
ii. Analyze functional group of the specialty oil by using Fourier Transforms Infrared Transmitter (FTIR) Unit.
iii. Compare the functional group of specialty oil with commodity oil (palm oil and maize oil).

CHAPTER 2

LITERATURE REVIEW

2.1 Specialty oil

Specialty oil has different property with the commodity oil which is used as cooking oil. Specialty oil usually obtain in small amount but with higher selling price. It is difficult to obtain and are used only in a certain industry. In this research, spices that will be used are *Pimpinella Anisum, sesamun indicum, syzygium aromaticum* and *cuminium cyminum.*

Specialty oil is usually applied in pharmaceuticals, health, and cosmetic industries. This specialty oil also can be used as an alternative to the oil that is produce from the animal fat.

2.2 Functional group

In organic chemistry, functional groups are specific groups of atoms within molecules that are responsible for the characteristic chemical reactions of those molecules. The same functional group will undergo the same or similar chemical reaction(s) regardless of the size of the molecule it is a part of. However, its relative reactivity can be modified by nearby functional groups.

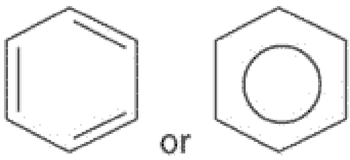
Combining the names of functional groups with the names of the parent alkanes generates a powerful systematic nomenclature for naming organic compounds. The non-hydrogen atoms of functional groups are always associated with each other and with the rest of the molecule by covalent bonds. When the group of atoms is associated with the rest of the molecule primarily by ionic forces, the group is referred to more properly as a polyatomic ion or complex ion. And all of these are called radicals, by a meaning of the term *radical* that predate the free radical. The first carbon atom after the carbon that attaches to the functional group is called the alpha carbon.

Functional groups are attached to the carbon backbone of organic molecules. They determine the characteristics and chemical reactivity of molecules. Functional groups are far less stable than the carbon backbone and are likely to participate in chemical reactions. Functional groups that vary based upon the number and orders of π bonds impart different chemistry.

The basic organic structure is carbons and hydrogen's, all singly bonded to each other. These are called alkanes. Any variation from that basic structure is called a functional group. Knowing the types of functional groups is basic to an understanding of organic chemistry. A list of functional groups is shown in Table 2.1. In this table, ***R*** represents a group of carbons and hydrogens not relevant to the functional group. The R groups do not have to be the same.

Table 2.1: General list of functional group

Name	**Condensed Formula**	**Description**
alkene	$R_2C=CR_2$	contains a C=C double bond
alkyne	$RC\equiv CR$	contains a C≡C triple bond
alcohol	ROH	contains O singly bonded to a C and a H
ether	ROR	contains O singly bonded to two C
aldehyde	RCHO	contains C doubly bonded to O and singly to H

ketone	RCOR	contains C doubly bonded to O and singly to two C
hemiacetal	ROCOHR	contains C singly bonded to O of ether and of alcohol
carboxylic acid	RCOOH	contains C doubly bonded to O and singly to O of OH
amine		
primary	RNH_2	contains N singly bonded to one C and two H
secondary	R_2NH	contains N singly bonded to two C and one H
tertiary	R_3N	contains N singly bonded to three C
aromatic		contains a flat six-membered ring

2.4 *Pimpinella Anisum*

Anise (*Pimpinella anisum*) is a grassy annual plant with white flowers and small green to yellow seeds grows in Turkey, Iran, India, Egypt, and many other warm region throughout the world. Anise is primarily grown for its fruit, commercially called "seed" that is used as flavoring. In Iranian folk medicine, the plant and especially its seed essential oil have been used for the treatment of some diseases including seizures and epilepsy. Anise seed oil also is used in food processing to impart flavor to cakes and alcoholic beverages (Abbas Besharati,2004).

It is an herbaceous annual plant growing to 1m tall. The leaves at the base of the plant are simple, 2-5 cm long and shallowly lobed, while leaves higher on the stems are feathery pinnate, divided into numerous leaflets. The flowers are white, 3 mm diameter, produced in dense umbels. The fruit is an oblong dry schizocarp, 3-5 mm long.

Its fruit, known as aniseed, is one of the oldest spices. The seed is ground-grey to greyish-brown in colour, oval in shape and 3.2 to 4.8 mm in length. It has an agreeable odors and a pleasant taste. The anise plant grows up to a height of 75 cm. It requires sunshine and warmth and does not grow satisfactorily in the tropical lowlands.

Scientific Classification:
Kingdom: *Plantae*
Division: *Magnoliophyta*
Class: *Magnoliopsida*
Order: *Apiales*
Family: *Apiaceae*
Genus: *Pimpinella*
Spices: *Pimpinella Anisum*

Figure 2.1: Dried anis seeds.

2.5 S*esamum indicum*

Sesame (*Sesamum indicum*) is recognized as one of the oldest crops in the world. Archeological records indicate that it has been used in India for more than 5000 years and is recorded as a crop in Babylon and Assyria some 4000 years ago. The crop has since spread over many parts of the world including the East African region where it is grown mainly for grains and oil extraction. The oil is very stable due to the presence of a number of antioxidants such as sesamin, sesamolin and sesamol (Suja et al., 2004). Therefore, it has a long shelf life and can be blended with less stable vegetable oils to improve their stability and longevity (Chung *et al*.,2004; Suja *et al*., 2004).

Sesame is an annual self pollinating plant with an erect, pubescent, branching stem, and 0.60 to 1.20 m tall. The leaves are ovate to lanceolate or oblong while the lower leaves are trilobed and sometimes ternate and the upper leaves are undivided, irregularly serrate and pointed (Felter and Lloyd,1898). The older cultivars have smooth and flat leaves while the no shattering cultivars have cupped leaves with leaf like outgrowths on their lower side. Some cultivars have many branches, while others are relatively unbranched. The flowers are tubular, pendulant, bell shaped, and two lipped with a pale purple or rose to white color and 1.9 to 2.5 cm long. In addition, the flowers are borne on short glandular pedicels (Felter and Lloyd,1898). One flower is produced at each leaf axil and the lower flowers usually bloom 2 to 3 months after planting with continuous blooming until the uppermost flowers are open. The fruit is an oblong, mucronate, pubescent capsule containing numerous small, oval, and yellow, white, red, brown, or black seeds (Felter and Lloyd,1898)

Scientific classification: Kingdom: *Plantae* Division: *Magnoliophyta* Class: *Magnoliopsida* Order: *Lamiales* Family: *Pedaliaceace* Genus: *Sesamum* Species: *Sesamum Indicum.*	

Figure 2.2: Black and white sesame seeds.

2.6 *Syzygium aromaticum*

Aroma chemical present in natural leaves and flowers have been widely used in aroma therapy since ancient times, suggesting that they have some beneficial health effects in addition to their pleasant odors. Until recently, aroma chemicals have been studied mainly from the aspects of aroma chemical, such as antioxidant activities (Lee and Shibamoto, 2001)

Syzygium aromaticum are also known as cloves are the sources of anti-microbial agents against oral bacteria that are commonly associated with dental caries and periodontal disease (Cai and Wu, 1996). In Korea, cloves have been successfully used for asthma and various allergic disorders by oral administration (*Kim et al*., 1998)

The clove tree is an evergreen which grows to a height ranging from 10 to 20 m, having large oval leaves and crimson flowers in numerous groups of terminal clusters. The flower buds are at first of a pale color and gradually become green, after which they develop into a bright red, when they are ready for collecting. Cloves are harvested when 1.5 to 2 cm long, and consist of a long calyx, terminating in four spreading sepals, and four unopened petals which form a small ball in the centre.

<u>Scientific classification</u>
Kingdom: *Plantae*
Division: *Magnoliophyta*
Class: *Magnoliopsida*
Order: *Myrtales*
Family: *Myrtaceace*
Genus: *Syzygium*
Species: *Syzygium aromaticem*

Figure 2.3: Dried clove

2.7 *Cuminum cyminum*

It is an herbaceous annual plant, with a slender branched stem 20 to 30 cm tall. The leaves are 5 to 10 cm long, pinnate or bipinnate, thread-like leaflets. The flowers are small, white or pink, and borne in umbels. The fruit is a laterally fusiform or ovoid achene 4 to 5 mm long, containing a single seed (www.blogtoplist.com/rss/cumin.html). Cumin seeds are similar to fennel seeds in appearance, but are smaller and darker in colour. Cumin seeds are used as a spice for their distinctive aroma, popular in North African, Middle Eastern, Western Chinese, Indian, Cuban and Mexican cuisine. Cumin can be used to season many dishes, as it draws out their natural sweetness. It is traditionally added to curries, enchiladas, tacos, and other Middle-eastern, Indian, Cuban and Mexican-style foods. It can also be added to salsa to give it extra flavour. Cumin has also been used on meat in addition to other common seasonings.

Scientific classification
Kingdom: *Plantae*
Division: *Magnoliophyta*
Class: *Magnoliopsida*
Order: *Apiales*
Family: *Apiaceae*
Genus: *Cuminum*
Species: *Cuminum. cyminum*

Figure 2.4: Cumin seed

2.8 Extraction

There are various type of separation process such as distillation, drying, adsorption, filtration and extraction. Extraction is one of the process that being use to extract the specialty oil from fruit, seeds and many more. This is because there are found chemical compound inside the mixture in solid phase. Then, to get the compound inside the solid phase material, it needs solvent as a medium to get the specialty oil inside the material. The process is also known as solvent extraction (liquid-solid extraction) (Ahmad, 1995).

Solvent extraction is usually using in extract the specialty oil from the seeds and fruits. This method is not suitable for the food industry because the solvent is dangerous to the human and it cannot be eat. The advantage of this process is it can extract almost all the oil inside the fruits and seeds and flaking. Absolute n-hexane, a petroleum-derived product, has been extensively used as a solvent for the oil extraction from soya beans and other oilseeds because of its low vaporization temperature (boiling point 63 to 69 ^{o}C), high stability, low corrosiveness, low greasy residual effect, and better odour and flavour productivity for the milled products (Johnson & Lusas, 1983). Extraction produces high-quality oil which requires less refining, produces high quality meal which requires less toasting, uses less energy.

CHAPTER 3

METHODOLOGY

3.1 Introduction

This research is focusing on oil extraction from spices and to identify its potential as the source of specialty oils by analysis their functional group. This is the beginning of the research possibilities of specialty oil production from the selected spices that have a naturally potential. The functional group of the specialty oil will be compared with the functional group contain in commodity oil which are palm oil and maize oil.

The comparison is to recognize the diversification of component in the specialty oil and commodity oil.

3.2 Research Apparatus/Chemical Reagent.

3.2.1 Lab Apparatus

The apparatus used in this experiment:

a) Soxhlet Extraction unit
b) Rotary evaporator
c) Fourier Transform Intrared Transmeter (FTIR)
d) Thimble 26mm x 60mm
e) Measuring cylinder 500ml
f) Conical flask 500ml
g) Analytical balance
h) Water bath

Thimble that used in this research are made from cellulose because its not react with the solvent and solution during the experiment.

3.2.2 Chemical reagent

a) n-Hexane; C_6H_{14}, density 0.659kg/L, 86.18g/mol

3.2.3 Research sample

a) Syzygium aromaticum
b) Sesamun indicum
c) Pimpinella anisum
d) Cuminium cyminum

3.3 Research methodology

The general methodology of this research are illustrate in figure 3.1 below:

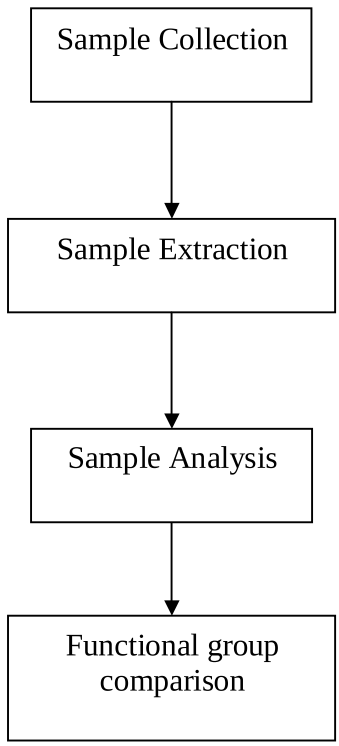


Figure 3.1: General methodology

3.3.1 Sample collection

In this research the four spices was selected based on ability easy to get in local market and frequently used in society. The sample are *Syzygium aromaticum, Sesamun indicum ,Pimpinella anisum,Cuminium cyminum.*

Pimpinella anisum

Sesamum indicum

Cuminium cyminum

Syzygium aromaticum

Figure 3.2: Spices sample used in this research.

3.3.2 Sample Extraction

Sample extraction is an important part of this research process in order to get high yield of specialty oil. First, all the selected spices must be blended in to small size. This is because when the surface area are increase the rate of the extraction process also increase and also easy to put sample in the thimble.

Then, the blended samples are weighted and put into the thimble. For this research, triplicate data are prepared for each spice to get the average of oil present in the sample. Solvent that has been used in this research is n-Hexane. Each experiment, 400mL of n-Hexane will be used and put it into the still pot which at the bottom part of the Soxhlet Extractor Unit.

After sample and the solvent are put into the Soxhlet Extractor Unit, the equipment is switched on and the operations need to be run in 4 hours. During the experiment, hexane will be recycled into the still pot and the specialty oil contain in the sample is extracted by the hexane.

After 4 hours extraction process, the Soxhlet extractor needs to be switch off and let it cool about 30 minutes to cool down the equipment. After that, the solvent with sample oil mixture is taken out from the still pot and put it in the conical flask.

The mixture oil must be separated to get the pure essential oil. To get the essential oil, the mixture will be run on rotary evaporator unit. The temperature of the rotary evaporator was set about 69 to 70^{0}C which is in the range of n-hexane boiling point. The vacuum pump must be switch on and the tap water must be opened during the evaporation in order to make sure the separating process run completely.

Figure 3.3: Soxhlet Extraction Unit.

Figure 3.4: Rotary evaporator unit.

3.3.3 Sample analysis

In this research Fourier Transform Infrared Transmitter (FTIR) Unit is used as the analyzer (model Nicolet Avatar 370) to find the functional group in the specialty oils. The Nicolet Avatar 370 is available in several source-beamsplitter-detector configurations. There are two main considerations when selecting a configuration which is compatibility and spectral range. This analysis was conducted in the Universiti Malaysia Pahang.

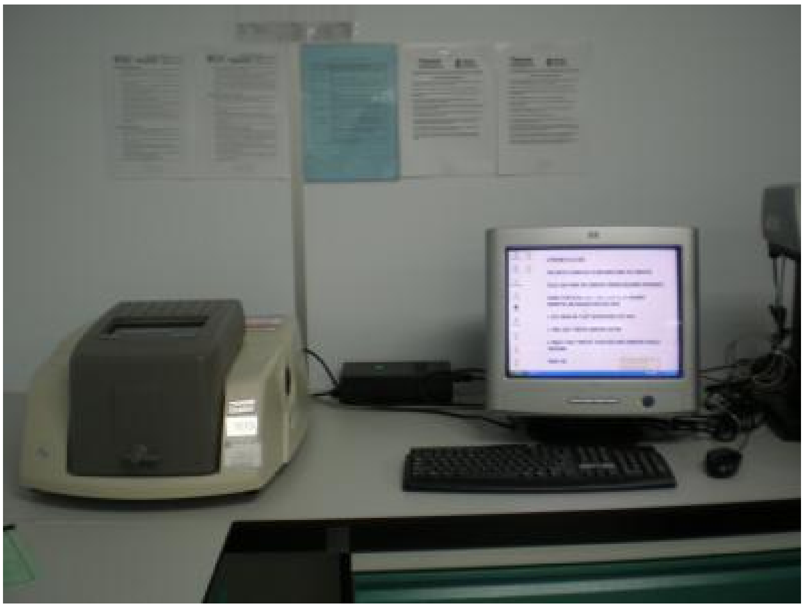


Figure 3.5: Fourier Transmeter Infrared (FTIR) Unit.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

In this research, sample used are *Pimpinella Anisum,* S*esamum indicum, Syzygium aromaticum*, and *Cuminum cyminum* which are in spices category, being sifting by n-Hexane solvent by using Soxhlet Extraction Unit in Universiti Malaysia Pahang laboratory. The sifting process is conduct by following the liquid-solid extraction by using n-Hexane as a solvent. For each spices, three samples will be prepared to get average essential oil present.

After the extraction process, the mixture of n-Hexane and the essential oil will be separated using Rotary Evaporator Unit and analyzed by Fourier Transform Infrared Transmeter (FTIR) Unit.

Finally one sample of the spices will be chosen based on it similarity of functional group with palm oil and maize oil and will be varied in term of weight parameter in order to find the optimum mass value.

4.2 Calculation of specialty oil produces.

Specialty oil produced can be calculated by using this equation:

$$\text{Percent specialty oil} = \frac{\text{Specialty oil weight}}{\text{Sample weight}} \times 100\%$$

4.3 Specialty oil extraction result.

Result of specialty oil extraction for all spices sample illustrated in table below:

Table 4.1: Physical properties of the specialty oils.

Specialty oil	Odor	Colour	Remark
Pimpinella Anisum	Strong odor	Black	Liquid in room temperature.
Sesamum indicum,	Light odor	Light brown	Liquid in room temperature
Syzygium aromaticum	Strong odor	Dark brown	Liquid in room temperature
Cuminum cyminum	Strong odor	Black	Liquid in room temperature

Table 4.2: *Pimpinella Anisum* specialty oil.

Solvent: n-Hexane				
Extraction time: 4 hours				
Operation temperature: 69°C				
Sample	**Sample weight (gram)**	**Solvent Volume (mL)**	**Specialty oil weight (gram)**	**Specialty oil percent (%)**
I	16.00	400	3.48	21.78
II	16.12	400	2.90	18.01
III	16.15	400	3.34	26.93

$$\text{Average percent specialty oil present} = \frac{\text{Total specialty oil weight}}{\text{Total sample weight}} \times 100\%$$

$$= \frac{9.73\text{g}}{48.27\text{g}} \times 100\%$$

$$= 20.16\%$$

Table 4.3: *Sesamun Indicum* specialty oil.

Solvent: n-Hexane				
Extraction time: 4 hours				
Operation temperature: 69°C				
Sample	**Sample weight (gram)**	**Solvent Volume (mL)**	**Specialty oil weight (gram)**	**Specialty oil percent (%)**
I	17.40	400	7.82	44.94
II	17.22	400	7.93	46.05
III	17.51	400	8.26	47.20

$$\text{Average percent specialty oil present} = \frac{\text{Total specialty oil weight}}{\text{Total sample weight}} \times 100\%$$

$$= \frac{24.01g}{52.13g} \times 100\%$$

$$= 46.06\ \%$$

Table 4.4: *Syzygium aromaticum* specialty oil.

Solvent: n-Hexane				
Extraction time: 4 hours				
Operation temperature: 69^{o}C				
Sample	**Sample weight (gram)**	**Solvent Volume (mL)**	**Specialty oil weight (gram)**	**Specialty oil percent (%)**
I	14.00	400	2.62	18.71
II	14.14	400	2.02	14.27
III	14.50	400	2.42	16.69

$$\text{Average percent specialty oil present} = \frac{\text{Total specialty oil weight}}{\text{Total sample weight}} \times 100\%$$

$$= \frac{7.06\ g}{42.64g} \times 100\%$$

$$= 16.55\%$$

Table 4.5: *Cuminum cyminum* specialty oil.

Solvent: n-Hexane				
Extraction time: 4 hours				
Operation temperature: 69^{o}C				
Sample	**Sample weight (gram)**	**Solvent Volume (mL)**	**Specialty oil weight (gram)**	**Specialty oil percent (%)**
I	16.20	400	1.56	9.63
II	16.31	400	2.01	12.32
III	16.1	400	1.67	10.37

$$\text{Average percent specialty oil present} = \frac{\text{Total specialty oil weight}}{\text{Total sample weight}} \times 100\%$$

$$= \frac{5.24\text{ g}}{48.61\text{g}} \times 100\%$$

$$= 10.78\%$$

4.4 Analysis specialty oil result.

Analysis of functional group of the specialty oil is made by using Fourier Transform Transmeter (FTIR) Unit. The result is illustrated as below.

Table 4.6: *Pimpinella Anisum* specialty oil analysis.

No.	**Wave numbers (cm^{-1})**	**Bond**	**Possible functional group**	**Range**
1.	2923.75	C-H O-H	Stretch alkane Carboxylic acid	2850-3000 2500-3400
2.	2852.82	C-H C-H O-H	Stretch alkane Aldehyde Carboxylic acid	2850-3000 2800-2900 2500-3400
3.	1746.31	C=O	Ester	1730-1750
4.	1711.37	C=O C=O	Ketone Carboxylic acid	1705-1725 1700-1725
5.	1259.07	S=O	Alcohol, ether, ester, anhydride, carboxylic acid. Amine Sulfonyl chloride Fluoride	1000-1300 1000-1350 1140-1350 1000-1400
6.	1247.06	S=O	Alcohol, ether, ester, anhydride, carboxylic acid. Amine Sulfonyl chloride Fluoride	1000-1300 1000-1350 1140-1350 1000-1400

7.	1145.31	S=O	Alcohol, ether, ester, anhydride, carboxylic acid. Amine Sulfonyl chloride Fluoride	1000-1300 1000-1350 1140-1350 1000-1400
8.	1094.92	C-X	Alcohol, ether, ester, anhydride, carboxylic acid. Amine Fluoride	1000-1300 1000-1350 1000-1400
9.	1037.62	C-X	Alcohol, ether, ester, anhydride, carboxylic acid. Amine Fluoride	1000-1300 1000-1350 1000-1400
10.	799.08	C-H	Aromatic	690-900
11.	719.44	C-X	Chloride	540-785
12.	704.16	C-X	Chloride	540-785

Table 4.7: *Sesamun Indicum* specialty oil analysis.

No.	**Wave numbers (cm^{-1})**	**Bond**	**Possible functional group**	**Range**
1.	3006.72	C-H O-H	Stretch alkene Carboxylic acid	3000-3100 2500-3400
2.	2924.66	C-H O-H	Stretch alkane Carboxylic acid	2850-3000 2500-3400

3.	2854.31	C-H C-H O-H	Alkane Aldehyde Carboxylic acid	2850-3000 2800-2900 2500-3400
4.	1745.68	C=O	Ester	1730-1750
5.	1465.14	C-H	Bent Alkane	1465
6.	1238.64	C-N S=O C-X	Amine Sulfonyl chloride Fluoride	1000-1350 1140-1350 1000-1400
7.	1161.28	O-H C-N S=O C-X	Alcohol, ether, ester, anhydride, carboxylic acid. Amine Sulfonyl chloride Fluoride	1000-1300 1000-1350 1140-1350 1000-1400
8.	1099.10	O-H C-X C-N	Alcohol, ether, ester, anhydride, carboxylic acid. Fluoride Amine	1000-1300 1000-1400 1000-1350
9.	807.99	C-H	Bent aromatic	690-900
10.	722.83	C-H	Bent aromatic	690-900

Table 4.8: *Syzygium aromaticum* specialty oil analysis.

No.	Wave numbers (cm^{-1})	Bond	Possible functional group	Range
1.	2928.83	C-H O-H	Stretch alkane Carboxylic acid	2850-3000 2500-3400
2.	1638.16	C=O N-H C=C	Amide Bent Amide Alkene	1630-1680 1550-1640 1600-1680
3.	1605.98	C=C N-H	Alkene Bent Amide	1600-1680 1550-1640
4.	1368.11	S=O C-X	Sulfonyl chloride Fluoride	1300-1373 1000-1400
5.	1267.24	C-O C-N S=O C-X	Alcohol, ether, ester, anhydride, carboxylic acid Amine Sulfonyl chloride Fluoride	1000-1300 1000-1350 1140-1350 1000-1400
6.	1233.77	C-O C-N S=O C-X	Alcohol, ether, ester, anhydride, carboxylic acid Amine Sulfonyl chloride Fluoride	1000-1300 1000-1350 1140-1350 1000-1400
7.	1205.97	C-O C-N S=O	Alcohol, ether, ester, anhydride, carboxylic acid Amine Sulfonyl chloride	1000-1300 1000-1350 1140-1350

		C-X	Fluoride	1000-1400
8.	1149.40	C-O C-N S=O C-X	Alcohol, ether, ester, anhydride, carboxylic acid Amine Sulfonyl chloride Fluoride	1000-1300 1000-1350 1140-1350 1000-1400
9.	1122.19	C-O C-N S=O C-X	Alcohol, ether, ester, anhydride, carboxylic acid Amine Sulfonyl chloride Fluoride	1000-1300 1000-1350 1140-1350 1000-1400
10.	1035.66	C-O C-N C-X	Alcohol, ether, ester, anhydride, carboxylic acid Amine Fluoride	1000-1300 1000-1350 1000-1400
11.	849.51	C-H	Bent Aromatic	690-900
12.	817.06	C-H	Bent Aromatic	690-900
13.	794.36	C-H	Bent Aromatic	690-900
14.	746.07	C-H	Bent Aromatic	690-900

Table 4.9: *Cuminum cyminum* specialty oil analysis.

No.	**Wave numbers (cm^{-1})**	**Bond**	**Possible functional group**	**Range**
1.	2955.89	C-H O-H	Stretch alkane Carboxylic acid	2850-3000 2500-3400
2.	2924.29	C-H O-H	Stretch alkane Carboxylic acid	2850-3000 2500-3400
3.	2854.33	C-H O-H	Stretch alkane Carboxylic acid	2850-3000 2500-3400
4.	1745.03	C=O	Ester	1730-1750
5.	1706.36	C=O C=O	Ketone Carboxylic acid	1705-1725 1700-1726
6.	1464.77	C-H	Bent Alkane	1465
7.	1211.59	C-O C-N S=O C-X	Alcohol, ether, ester, anhydride, carboxylic acid Amine Sulfonyl chloride Fluoride	1000-1300 1000-1350 1140-1350 1000-1400
8.	1168.69	C-O C-N S=O C-X	Alcohol, ether, ester, anhydride, carboxylic acid Amine Sulfonyl chloride Fluoride	1000-1300 1000-1350 1140-1350 1000-1400
9.	1055.21	C-O	Alcohol, ether, ester, anhydride, carboxylic acid	1000-1300

		C-N C-X	Amine Fluoride	1000-1350 1000-1400
10.	840.03	C-H	Bent Aromatic	690-900
11.	828.70	C-H	Bent Aromatic	690-900
12.	815.26	C-H	Bent Aromatic	690-900
13.	722.67	C-H C-X	Bent Aromatic Chloride	690-900 540-785

4.5 Analysis of commercial oil

Analysis of functional group of in the commercial oil is made by using Fourier Transform Infrared Transmeter (FTIR) Unit. The result illustrated below:

Table 4.10: Cooking oil (palm oil) analysis.

No.	Wave numbers (cm^{-1})	Bond	Possible functional group	Range
1.	2923.09	C-H O-H	Stretch Alkane Carboxylic acid	2850-3000 2500-3400
2.	2853.09	C-H O-H	Stretch Alkane Carboxylic acid	2850-3000 2500-3400
3.	1745.30	C=O	Ester	1730-1750
4.	1465.50	C-H	Bent Alkane	1465
5.	1162.81	C-O	Alcohol, ether, ester, anhydride,	1000-1300

			carboxylic acid	
		C-N	Amine	1000-1350
		S=O	Sulfonyl chloride	1140-1350
		C-X	Fluoride	1000-1400
6.	117.05	C-O	Alcohol, ether, ester, anhydride, carboxylic acid	1000-1300
		C-N	Amine	1000-1350
		S=O	Sulfonyl chloride	1140-1350
		C-X	Fluoride	1000-1400
7.	721.79	C-H C-X	Bent Aromatic Chloride	690-900 540-785

Table 4.11: Maize oil analysis

No.	Wave numbers (cm^{-1})	Bond	Possible functional group	Range
1.	3008.71	C-H O-H	Stretch Alkene Carboxylic acid	3000-3100 2500-3400
2.	2925.53	C-H O-H	Stretch Alkane Carboxylic acid	2850-3000 2500-3400
3.	2854.35	C-H O-H	Stretch Alkane Carboxylic acid	2850-3000 2500-3400
4.	1745.55	C=O	Ester	1730-1750
5.	1465.50	C-H	Bent Alkane	1465
6.	1275.16	C-O	Alcohol, ether, ester, anhydride, carboxylic acid	1000-1300
		C-N	Amine	1000-1350

		S=O C-X	Sulfonyl chloride Fluoride	1140-1350 1000-1400
7.	1241.81	C-O C-N S=O C-X	Alcohol, ether, ester, anhydride, carboxylic acid Amine Sulfonyl chloride Fluoride	1000-1300 1000-1350 1140-1350 1000-1400
8.	1163.76	C-O C-N S=O C-X	Alcohol, ether, ester, anhydride, carboxylic acid Amine Sulfonyl chloride Fluoride	1000-1300 1000-1350 1140-1350 1000-1400
9.	1100.80	C-O C-N C-X	Alcohol, ether, ester, anhydride, carboxylic acid Amine Fluoride	1000-1300 1000-1350 1000-1400
10.	727.46	C-H C-X	Bent Aromatic Chloride	690-900 540-785

4.6 Subtraction with commodity oil

The subtraction with commodity oil is done to identify the different and similarity of the functional group in the specialty oil and the commodity oil which is palm oil and maize oil

4.6.1 Palm oil (*Elaeis guineensis jacq. oil*)

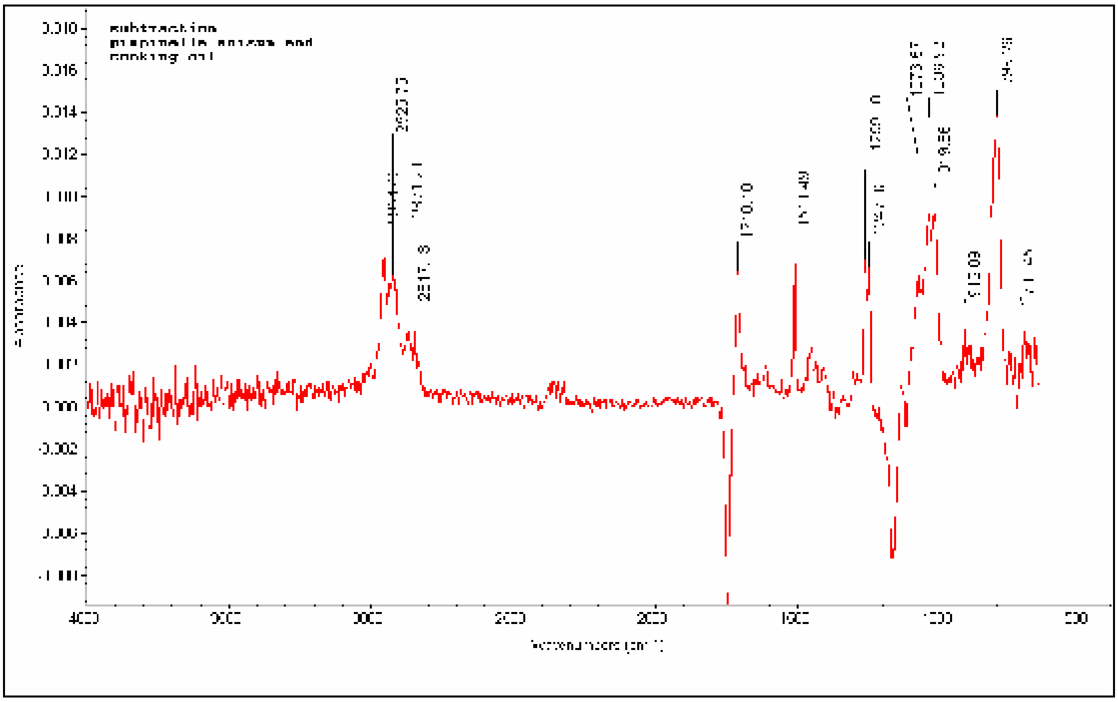


Figure 4.1: Subtraction *pimpinella anisum* with palm oil

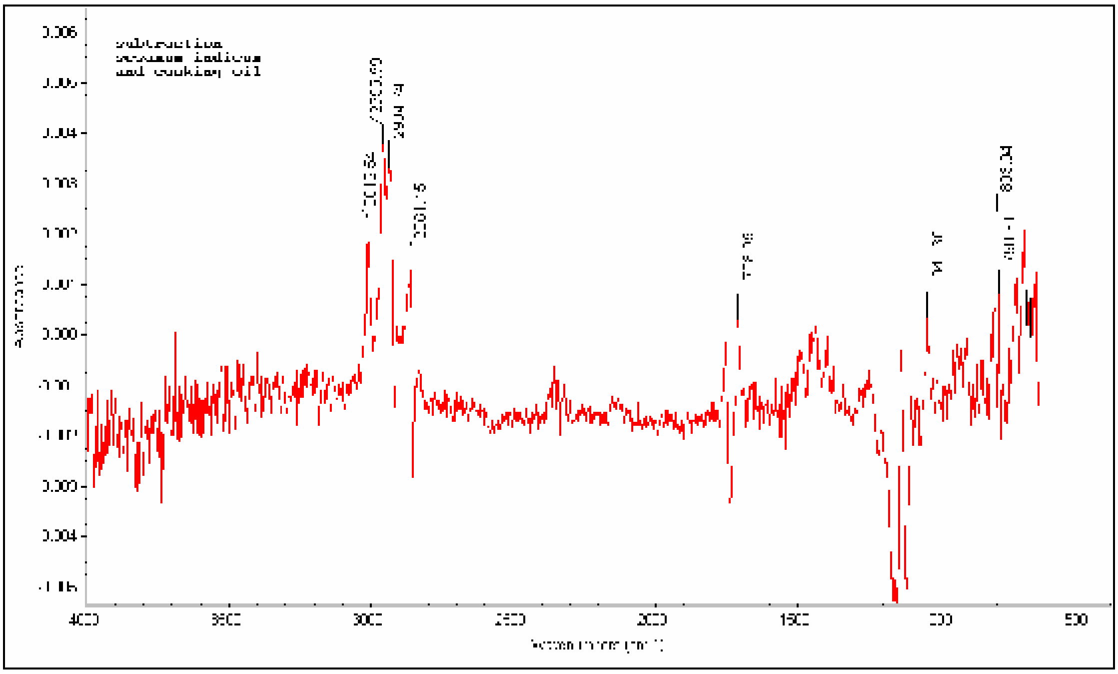


Figure 4.2: Subtraction *sesamum indicum* with palm oil

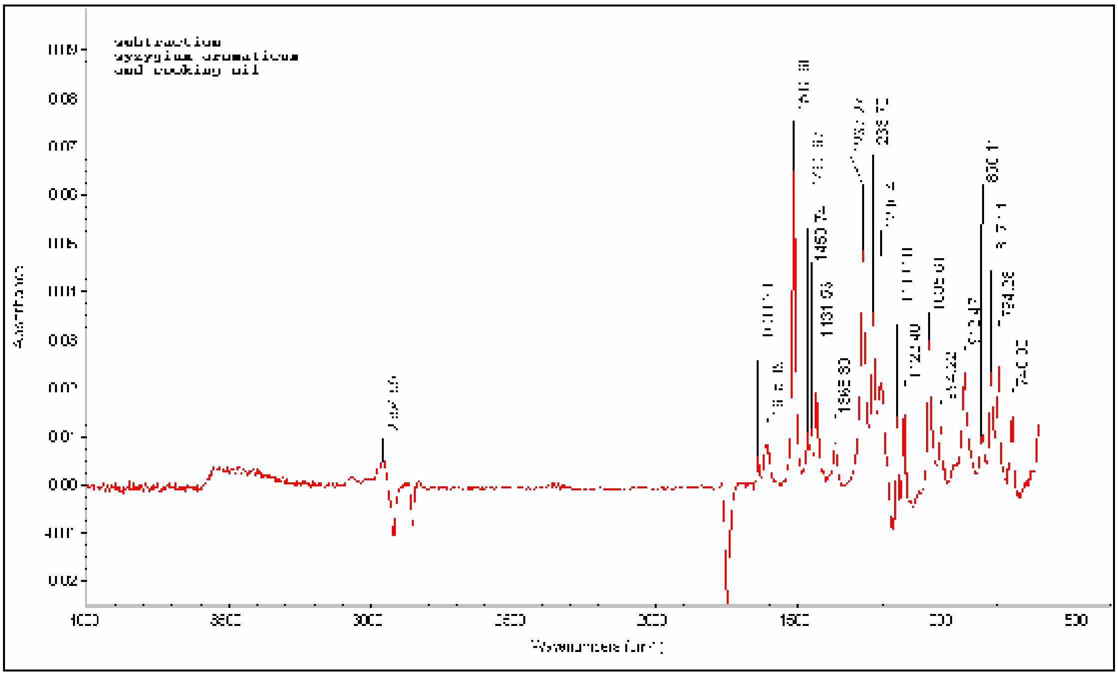


Figure 4.3: Subtraction *syzygium aromaticum* with palm oil

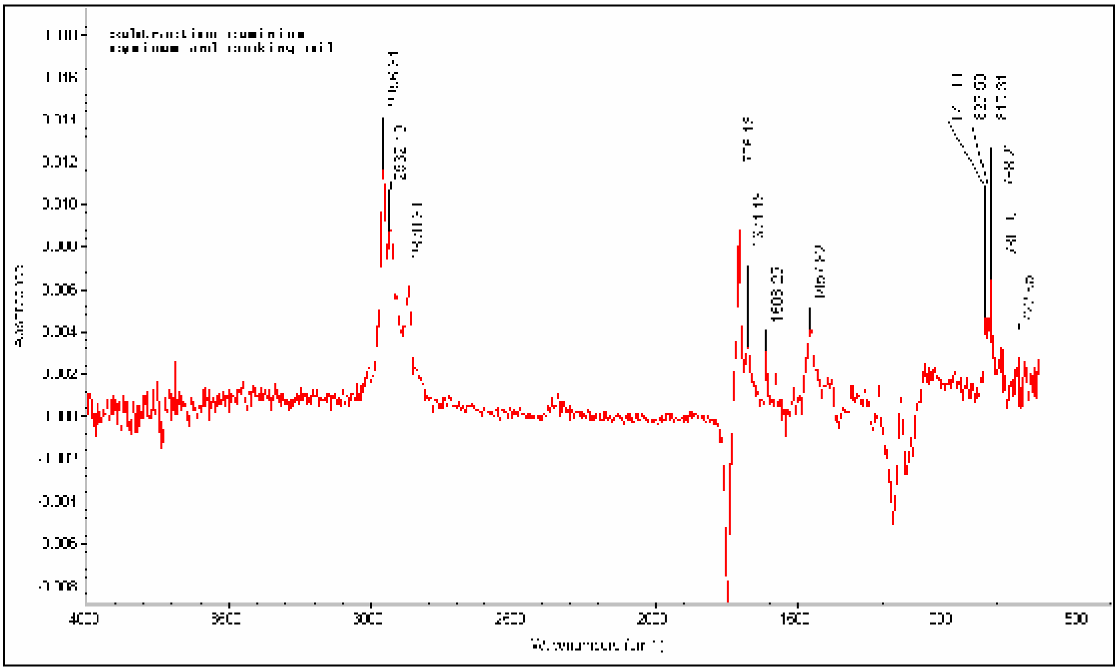


Figure 4.4: Subtraction *cuminium cyminum* with palm oil

4.6.2 Maize oil (*zea may oil*)

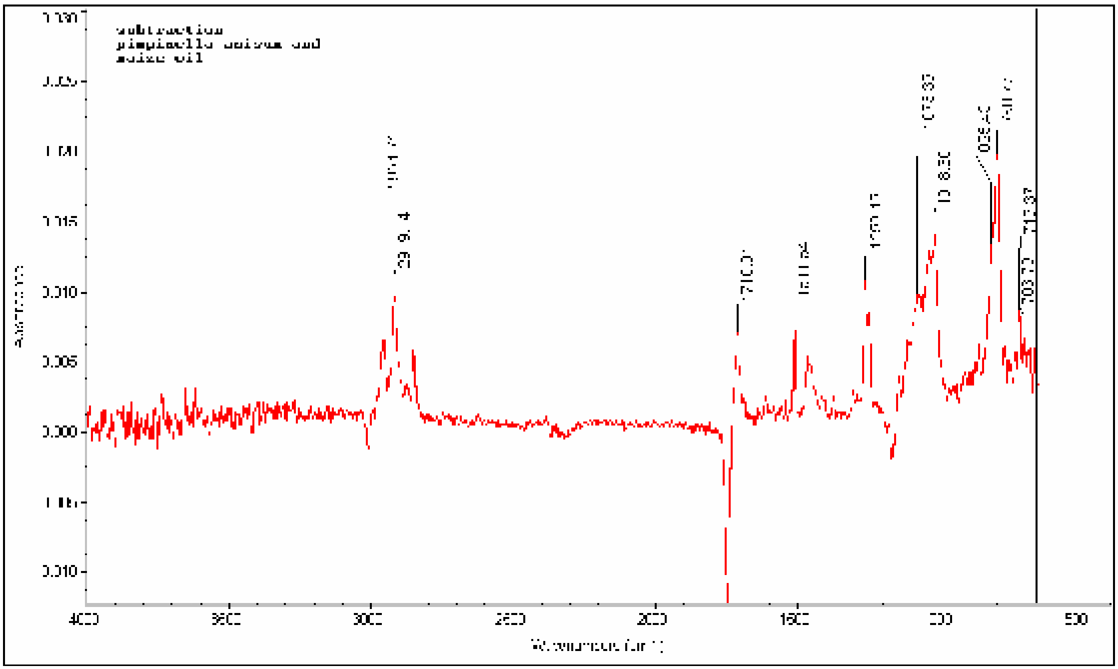


Figure 4.5: Subtraction *pimpinella anisum* with maize oil

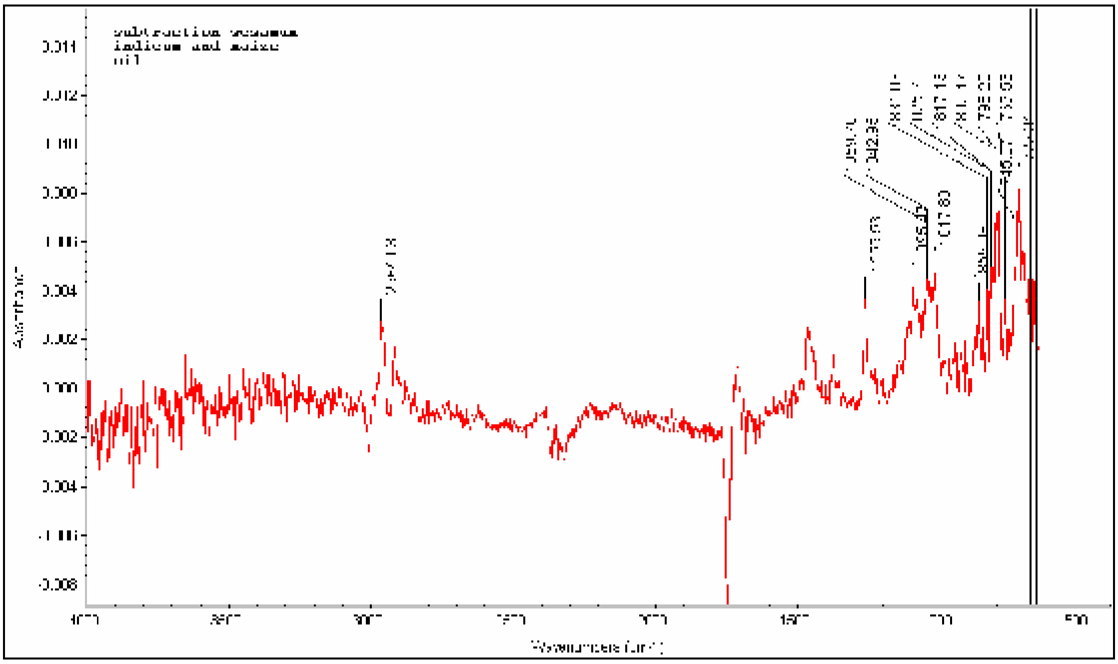


Figure 4.6: Subtraction *sesamum indicum* with maize oil

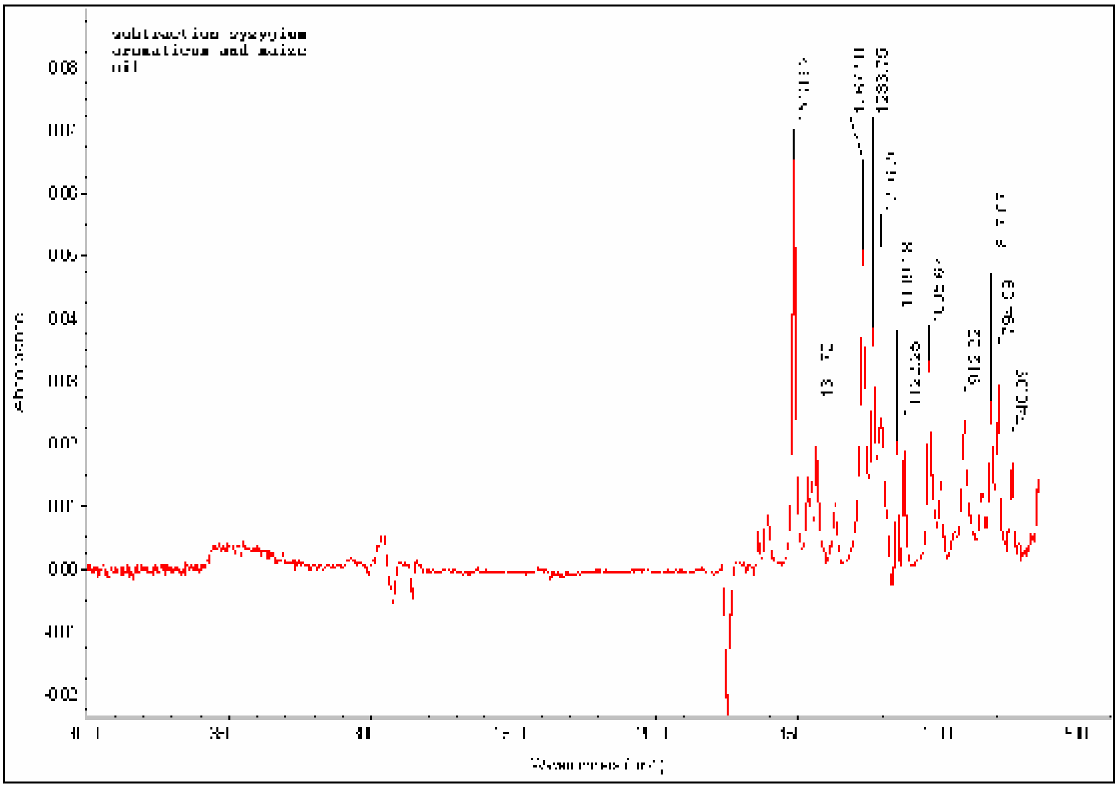


Figure 4.7: Subtraction *syzygium aromaticum* with maize oil

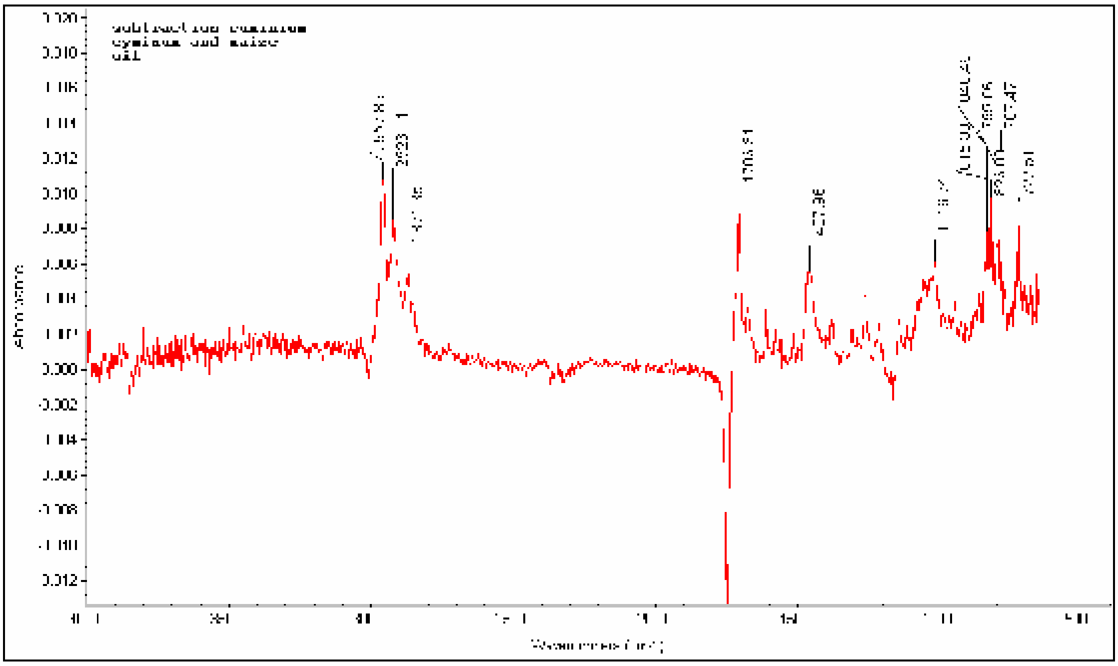


Figure 4.8: Subtraction *cuminium cyminum* with maize oil

4.7 Result variation of *sesamun indicum*.

Table 4.12: Variation of *sesamum indicum* weights.

Sample	Sample weight (gram)	Solvent Volume (mL)	Specialty oil weight (gram)	Specialty oil percent (%)
I	10.00	400	4.67	46.70%
II	12.00	400	5.68	47.33%
III	14.00	400	5.82	41.57%
IV	16.00	400	7.16	44.76%
V	18.00	400	8.42	46.78%
VI	20.00	400	9.28	46.40%

Table 4.13: Comparison s*esamum indicum* functional group.

Wave length (cm^{-1})	Intensity					
	20 gram	**18 gram**	**16 gram**	**14 gram**	**12 gram**	**10 gram**
2923	0.0361	0.0347	0.0347	0.0351	0.031	0.0323
2853	0.0230	0.0223	0.0231	0.0218	0.0199	0.0208
1745	0.0427	0.0426	0.0157	0.0158	0.0399	0.0159
1463	0.0129	0.0125	0.0127	0.0129	0.0115	0.0132

1239	0.0127	0.0128	0.0121	0.0130	0.0114	-
1162	0.0269	0.0269	0.0116	-	0.0249	0.0125
1092	0.0144	0.0138	-	-	0.0135	-
719	0.0193	0.0167	0.0162	0.0178	0.0168	0.0176
691	0.0131	0.0123	0.0123	0.0147	-	0.0130

4.8 Discussion

In this research, extraction process is conducted by using the Soxhlet Extraction unit with n-Hexane as a solvent medium to extract essential oil from the spices followed by liquid-solid extraction principle. N-hexane is selected because it has low boiling point temperature, high stability, low corrosiveness and residual effect. The important of low boiling point characteristic is it can avoid the chemical compound in the spices from damage or overheated.

4.8.1 Discussion of specialty oil

From the table, *sesamum indicum* produce highly yield of specialty oil which is about 46.06% (average) followed by *pimpinella anisum* (20.16%), *syzygium aromaticum* (16.55%), and *cuminum cyminum* (10.78%). The differences between all the specialty oil also can be seen by comparing each sample colour. For *pimpinella anisum* and *cuminium cyminum*, the essential oil produced is black in colour while the *sesamum indicum* and *syzygium aromaticum* have light brown and dark brown colour respecttively. All the spices essential oil has pleasant odour.

According to the percentage of essential oil produce, there have small different in each sample of spices. The different rate because of some error that cannot avoid when producing the essential oil such as:

a) All the spices are not fresh because the sample was buying from the local market. Usually is very hard to preserve spices in fresh condition. The spices need to be dry before can being pack and sell into market.
b) Specialty oil produces being adsorbed by the thimble when extraction process held and will decrease the essential weights.
c) Specialty oil produce maybe have some n-Hexane dissolved which is not fully evaporate.
d) Specialty oil produce is not fully extract from the sample.
e) When using the Rotary Evaporator Unit, the essential oil produce are stick to the flask and cannot be 100% collected.

4.8.2 Discussion of analysis specialty oil.

The specialty oils produce then will be analyzed by Fourier Transform Infrared Transmitter (FTIR) Unit to determine their functional group. Comparison also made to recognize the different between functional group in *Pimpinella Anisum,* S*esamum indicum, Syzygium aromaticum*, and *Cuminum cyminum* with the commercial oil. Commercial oil that have been chosen are palm oil and maize oil in order to made comparatives purposed.

This comparative is made to find the chemical properties which are different or same between the commercial oil and the essential oils. Every differentials of functional group in the specialty oil will bring different usages especially in industries.

a) *Pimpinella anisum*

- From table, there consist 12 significant peaks at wave numbers of 2923, 2852, 1746, 1711, 1259, 1247, 1145, 1094, 1037, 799, and 719, 704 indicating possible functional group such as alkane, carboxylic acid, aldehyde, ester, ketone, sulfonyl chloride, fluoride, alcohol, ether, anhydride, amine, aromatic, and chloride.

b) Sesamum indicum

- From table, there consist 10 significant peaks at wave number of 3006, 2924, 2854, 1745, 1465, 1238, 1161, 1099, 807, and 722 indicating possible functional group such as alkene, carboxylic acid, bent alkane, aldehyde, ester, amine, sulfonyl chloride, fluoride, alcohol, ether, anhydride, and aromatic.

c) Syzygium aromaticum

- From table, there consist 14 significant peaks at wave number of 2928, 1638, 1605, 1368, 1267, 1233, 1205, 1149, 1122, 1035, 849, 817, 794, 746 indicating possible functional group such as alkane, carboxylic acids, amide, bent amide, alkene, aromatic, sulfonyl chloride, alcohol, ether, ester, anhydride, and fluoride.

d) Cuminium cyminum

- From table, there consist 13 significant peaks at wave number of 2955, 2924, 2854, 1745, 1706, 1464, 1211, 1168, 1055, 840, 828, 815 and 722 indicating possible functional group such as alkane, carboxylic acid, ester, ketone, alcohol, ether, anhydride, amine, sulfonyl chloride, fluoride, aromatic and chloride.

It is showed that the main functional groups of spices are alkene, carboxylic acid, alkane, aldehyde, ester, amine, sulfonyl chloride, fluoride, alcohol, ether, anhydride, ketone, chloride, amide and aromatic. The different between four specialty chosen is cuminium cyminum does not have aldehyde, stretch alkene, alcohol, amide, bent amide and alkene functional group. Syzygium aromaticum does not have alkane, aldehyde, ester, ketone, stretch alkene, bent alkene, and chloride functional group. Sesamum indicum does not have ketone, amide, bent amide, and alkene functional group and finally pimpinella anisum do not have stretch alkene, bent alkane, amide, bent amide, and alkene functional group.

The commodity oil analysis showed that palm oil consist 8 significant peaks at wave number 2923, 2853, 1745, 1465, 1377, 1162, 1117, and 721 indicating possible functional group such as alkane, carboxylic acid, ester, alcohol, ether, ester, anhydride, amine, sulfonyl chloride, fluoride, aromatic, chloride.

While maize oil have 10 significant peaks at wave number 3008, 2925, 2854, 1745, 1465, 1275, 1241, 1163, 1100, and 727 indicating possible functional group such as alkene, carboxylic, alkane, ester, alcohol, ether, ester, anhydride, amine, sulfonyl chloride, fluoride, aromatic, and chloride functional group.

4.8.3 Discussion subtraction with commodity oil

a) Palm oil

As shown in figure 4.1, subtraction between *pimpinella anisum* and palm oil produce two significant peaks near 1745 and 1160 due to over subtraction of ester (C=O) and sulfonyl chloride(S=O), fluoride (C-X), alcohol, ether, ester, carboxylic acid (C-O) respectively. For the positive peaks, the strongest absorption band was observed at 799 which is bent aromatic compound (C-H). Positive at wave numbers 2954, 2925, 2871, 2847, is stretch alkane (C-H) and carboxylic acid (O-H). For wave number in range 1140-1350 the functional group is sulfonyl chloride (S=O) and 1000-1400 is fluoride (C-X).

Subtraction for *sesamum indicum*, figure 4.2 showed that two significant negative peaks near 1160 and 1120 due to over subtraction of alcohol, ether, ester, and carboxylic acid (C-O), amine (C-N), sulfonyl chloride (S=O), fluoride (C-X). The strongest absorption band was observed at 3012, bent alkene (C-H), 2955, 2934 which is bent alkane (C-H), and carboxylic acid (O-H).

Figures 4.3 showed that subtraction of *syzygium aromaticum* have one significant negative peak near 1750 due over the subtraction. The negative functional group is ester (C=O), the strongest absorption band was observe at peak 1513 and other is bent aromatic functional group (C-H) which is in 690-900 wave number range, alcohol, ether, ester, anhydride, carboxylic acid (C-O) in range 1000-1300,amine (C-N) range in 1000-1350, sulfonyl chloride (S=O) range in 1140-1350, and fluoride (C-X) range in1000-1400.

Subtraction for *cuminium cyminum* illustrate in figure 4.4 showed two negative peaks near 1750 due over subtraction of ester (C=O) and 1170 due over subtraction of fluoride (C-X), sulfonyl chloride (S=O), amine (C-N), alcohol, ether, ester, anhydride, carboxylic acid (C-O). While the stronger absorption band is 2956 for stretch alkane (C-H), carboxylic acid (O-H). Other positive peak was 1706, ketone (C=O), carboxylic acid (C=O) .

b) Maize oil.

As shown in figure 4.5 subtraction between *pimpinella anisum* and maize oil produce one significant peaks near 1745 and due to over subtraction of ester (C=O). For the positive peaks, the strongest absorption band was observed at 789 which is bent aromatic compound (C-H). Positive at wave numbers 2954, 2919, is stretch alkane (C-H) and carboxylic acid (O-H). For wave number in range 1140-1350 the functional group is sulfonyl chloride (S=O) and 1000-1400 is fluoride (C-X). Peak at 1710 is ketone (C=O), carboxylic acid (C=O).

Subtraction for *sesamum indicum*, figure 4.6 showed that one significant negative peaks near 1740 due to over subtraction aldehyde (C=O) and ester (C=O). The strongest absorption band was observed at 722, for bent aromatic (C-H), and chloride (C-X).

Figures 4.7 showed that subtraction of *syzygium aromaticum* have one significant negative peak near 1750 due over the subtraction. The negative functional group is ester (C=O), the strongest absorption band was observe at peak 1513 and other is bent aromatic functional group (C-H) which is in 690-900 wave number range, alcohol, ether, ester, anhydride, carboxylic acid (C-O) in range 1000-1300,amine (C-N) range in 1000-1350, sulfonyl chloride (S=O) range in 1140-1350, and fluoride (C-X) range in1000-1400. As can be seen that this subtraction most likely the syzygium aromaticum subtraction with palm oil.

Subtraction for *cuminium cyminum* illustrate in figure 4.7 showed one negative peaks near 1750 due over subtraction of ester (C=O) and. While the stronger absorption band is 2957, 2923, 2871 for stretch alkane (C-H), carboxylic acid (O-H). Other positive peak was 1706, ketone (C=O), carboxylic acid (C=O).

4.8.4 Discussion of optimal value of *sesamum indicum.*

The objective due to the subtraction of the specialty oil and the commodity oil is to find the similarity of the functional group of specialty oil and the commodity oil. From all graphs that gained from the analysis, the *sesamum indicum* have almost the similarity to the palm oil and maize oil. The different of the absorbance value also in small number range about -0.008 and 0.008. From this similarity, *sesamum indicum* had been chosen to be varied in term of weight to know the optimum value of the specialty oil produce.

Table 4.12 showed specialty oil that produces from 10 gram, 12 gram, 14 gram, 16 gram, 18 gram, and 20 gram has a range of 41.57%- 47.33%. The highest specialty oil produce is 12 gram sample and the lowest is 14 gram sample. The error in producing specialty oil had been discussed before and from the table, *sesamum indicum* produce oil about 45.59% (average).

Analysis from the 10 gram, 12 gram, 14 gram, 16 gram, 18 gram, 20 gram indicate from Table 4.13 showed that all the specialty oil produce have nearly similar wave length number and nearly similar absorbance intensity. In comparing the specialty oil, the optimum value for the ***sesamum indicum*** is 20 gram which is the higher absorbance intensity.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusions

In this research they are several conclusion can be made from the extraction and analysis result of spices sample. In extraction experiment, the highest yield of specialty oil that produced is S*esamum Indicum* which is (46.06%)(average) followed by *Pimpinella Anisum* (20.16%), *Syzygium Aromaticum* (16.55%), and *Cuminum Cyminum* (10.78%). *Pimpinella anisum*, *syzygium aromaticum*, and *cuminium cyminum* had strong odor while *sesamum indicum* had light odor. *Pimpinella anisum* and *cuminium cyminum* have black in colour while *sesamum indicum* and *syzygium aromaticum* have light brown and dark brown respectively. All the specialty oils are liquid in room temperature.

From the observation of the subtraction wavelengths result that produce after the analysis using Fourier Transforms Infrared (FTIR) Unit, the *Sesamum indicum* specialty oil is the one have nearly same chemical component with the commodity oil such as maize oil and cooking oil.

After the variation of weight parameter, sesamum indicum produce specialty oil in range 41.57% to 47.33%.The highest was 12 gram and the lowest was 14 gram sample. The analysis of all the sesamum indicum specialty oil give nearly similar wave length number and absorbance intensity. In comparing the specialty oil, the optimum value for the *sesamum indicum* is 20 gram which is the higher absorbance intensity.

Liquid-solid extraction method that used is proven as the good method in extracting specialty oil. N-hexane is a good solvent as the extractor solvent which is remove nearly all the specialty oil in the sample by comparing each percentage of specialty oil samples.

5.2 Recommendation

This research only covers for four spices only and for completion, research must be done to other spices. This is because in our country there are many sources that can produce oil especially specialty oil.

Other research also can be done for complete analysis of chemical characteristic such as research on triglycerides and fatty acid.

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APPENDIX A

Figure A1 : *Cuminium cyminum* specialty oil

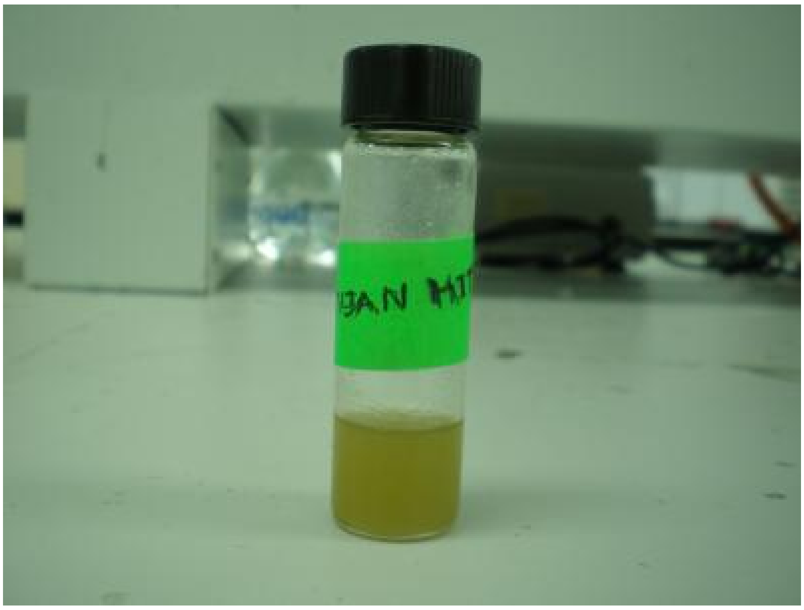

Figure A2: *Sesamum indicum* specialty oil

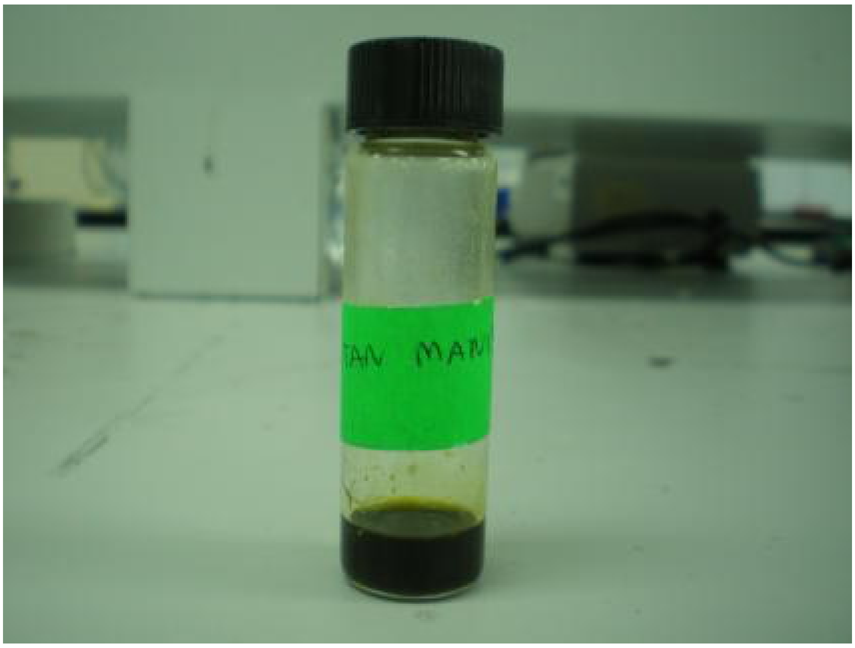

Figure A3: *Pimpinella anisum* specialty oil

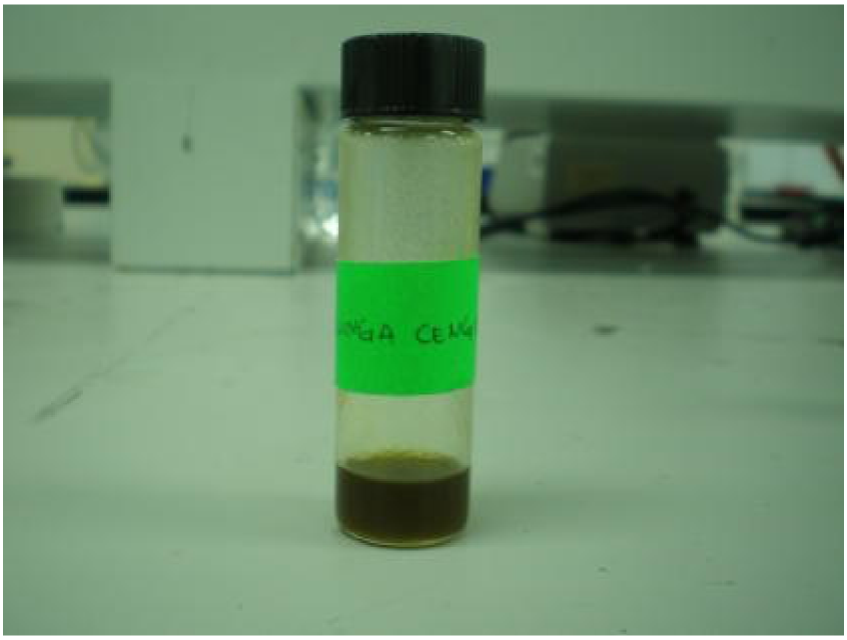

Figure A4: *Syzygium aromaticum* specialty oil

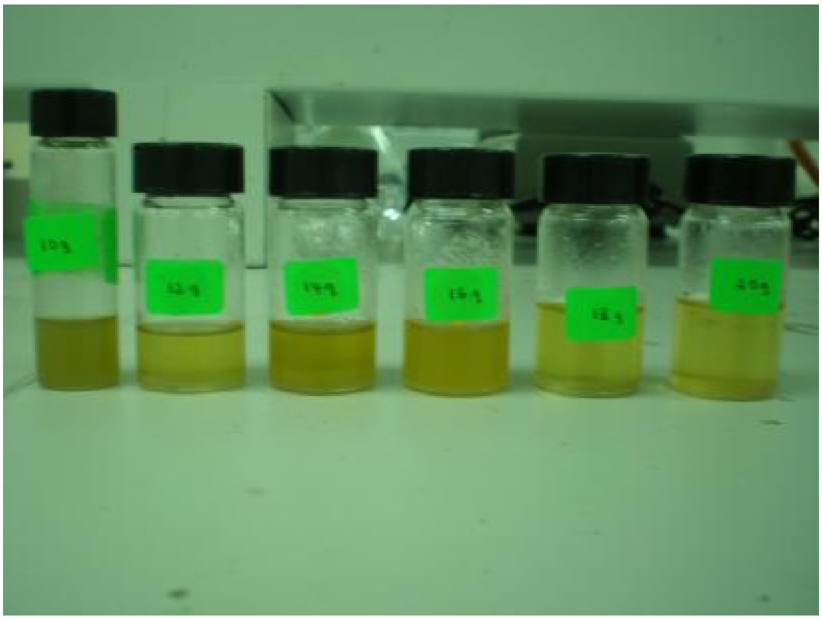

Figure A5: Weight variation of *sesamum indicum* specialty oil

APPENDIX B

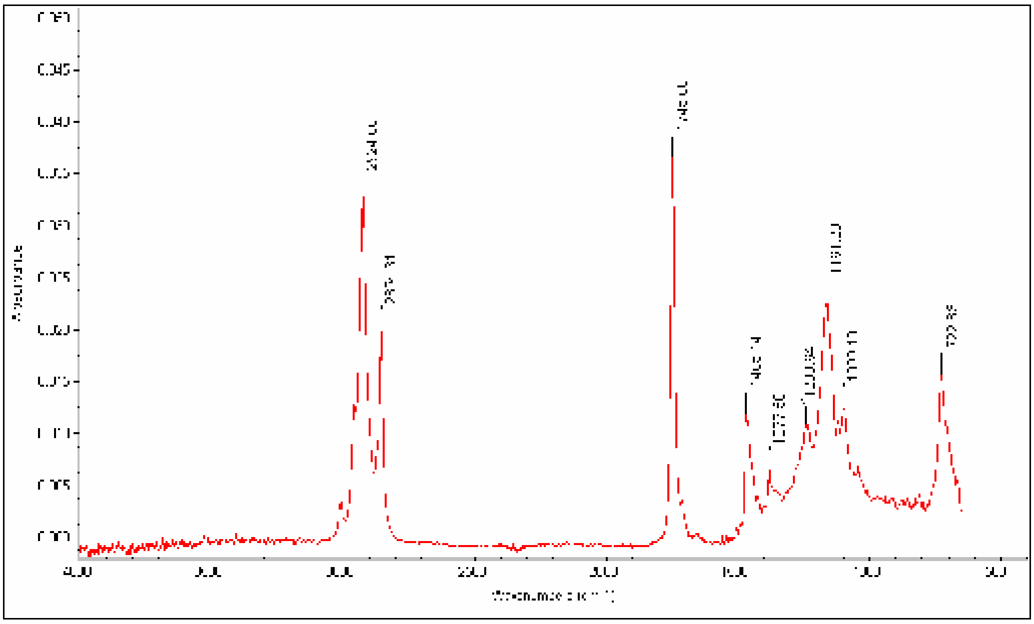

Figure B1: *Sesamum indicum* analysis graph

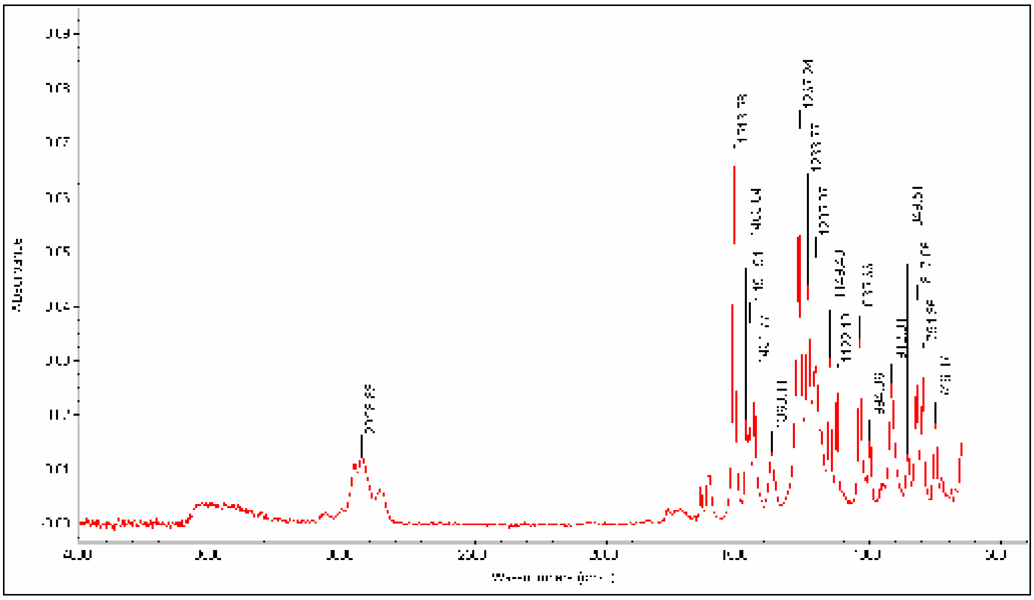

Figure B2: *Syzygium aromaticum* analysis graph

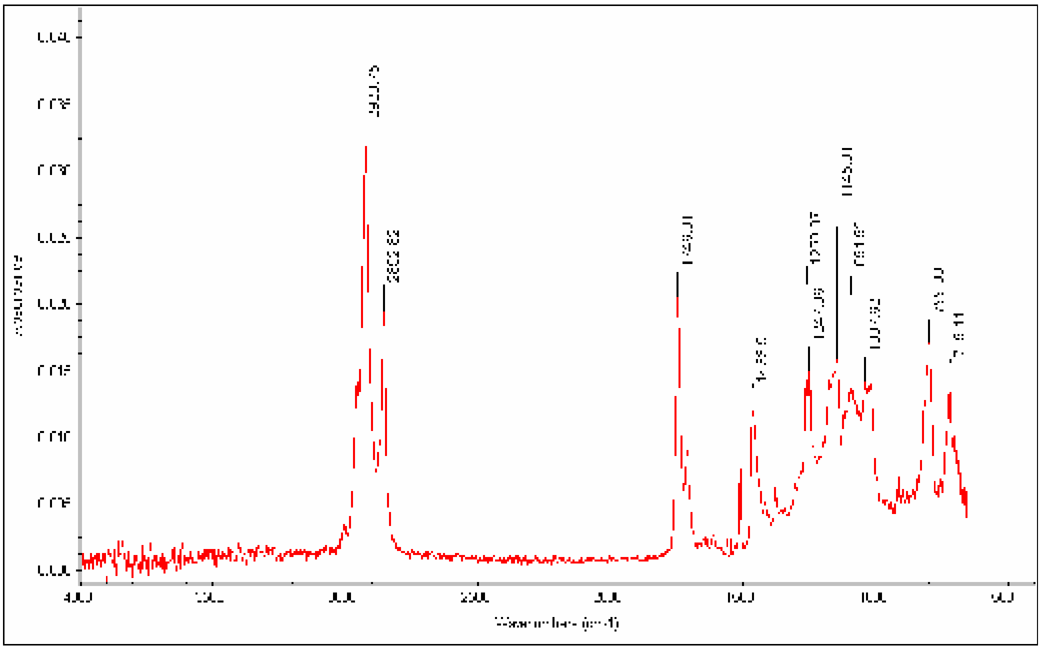

Figure B3: *Pimpinella anisum* analysis graph

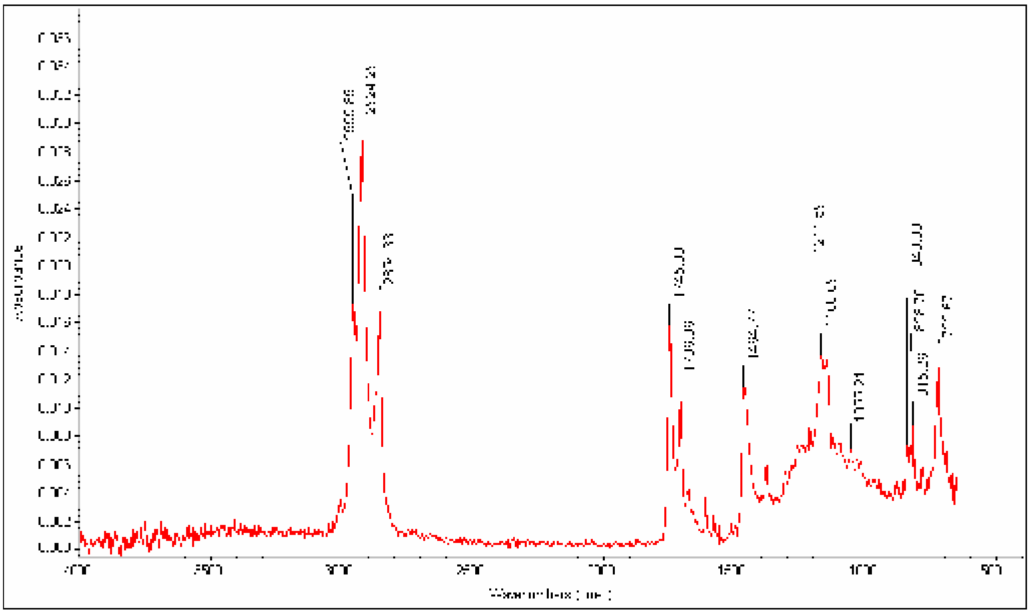

Figure B4: *Cuminium cyminum* analysis graph

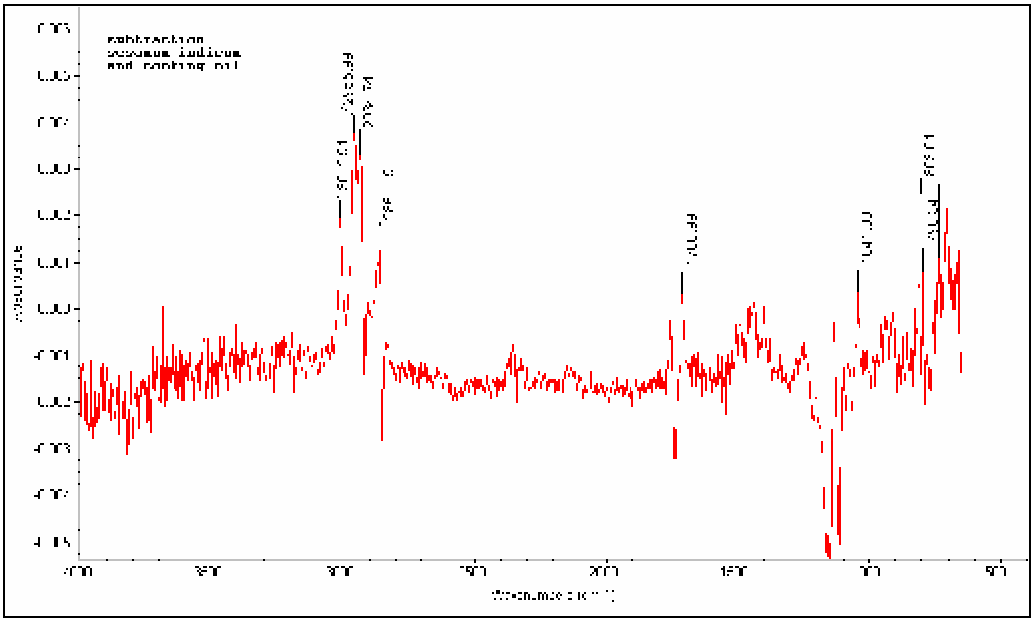

Figure B5: Subtraction *sesamum indicum* with palm oil

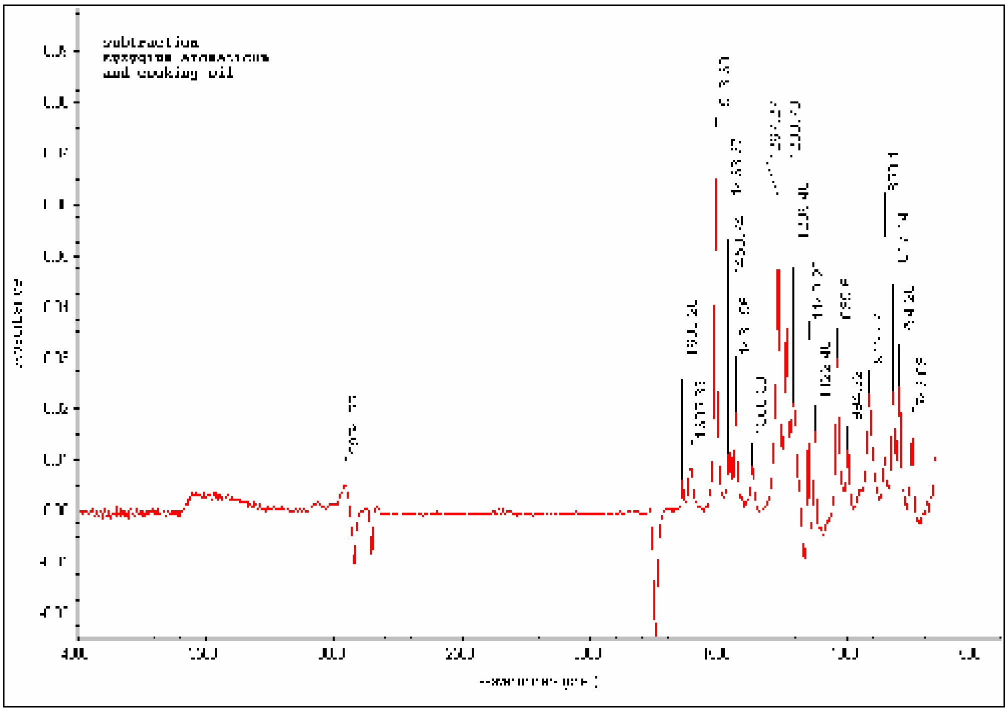

Figure B6: Suctraction *syzygium aromaticum* with palm oil

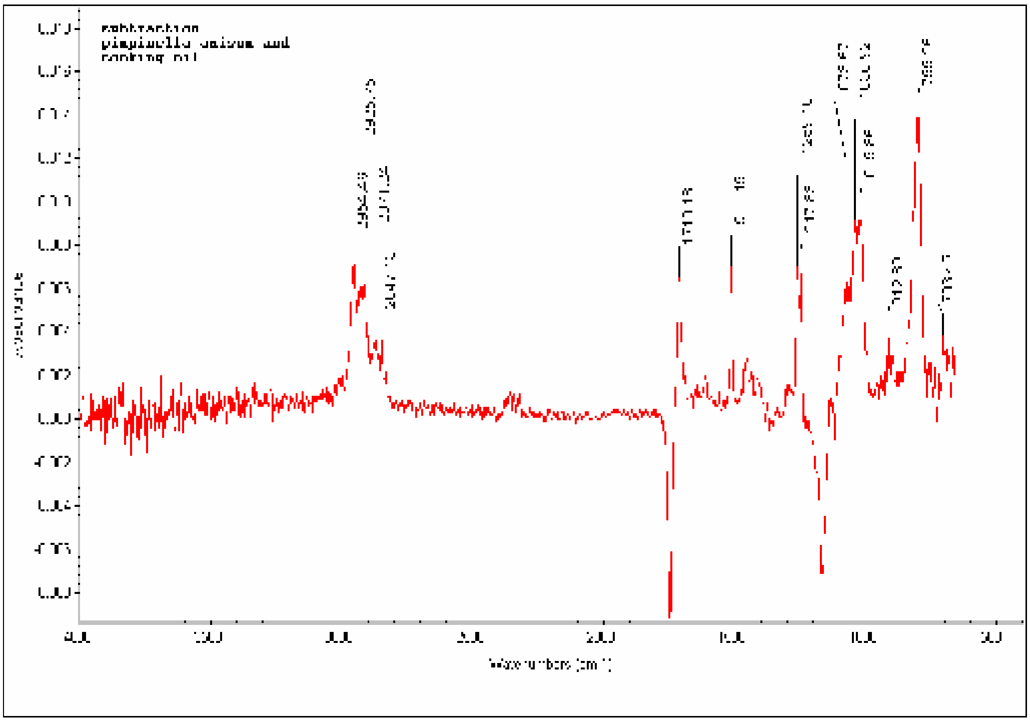

Figure B7: Subtraction *pimpinella anisum* with palm oil

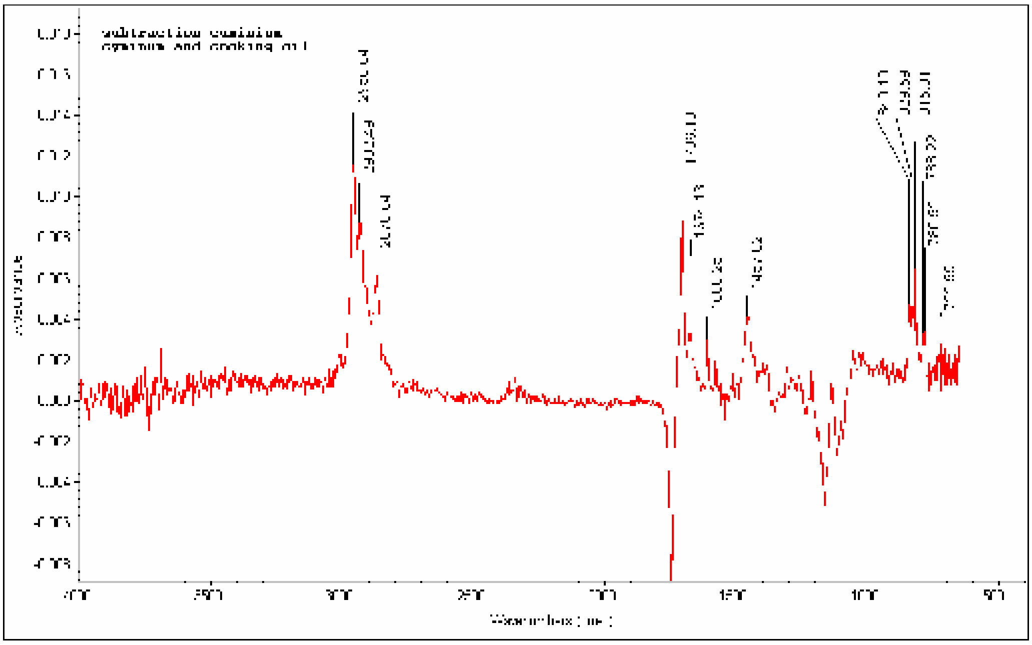

Figure B8: Subtraction *cuminium cyminum* with palm oil

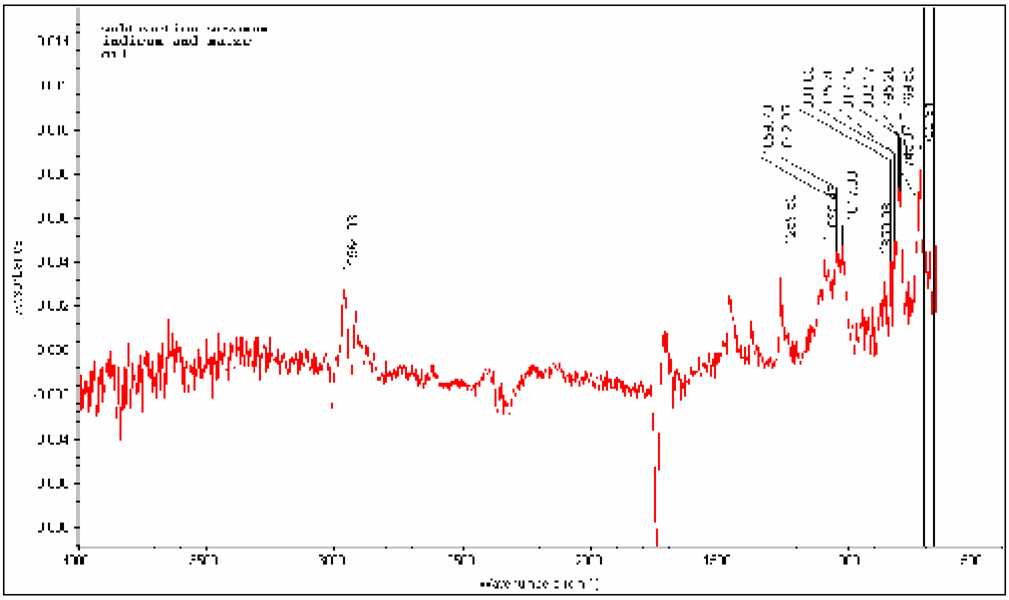

Figure B9: Subtraction *sesamum indicum* with maize oil

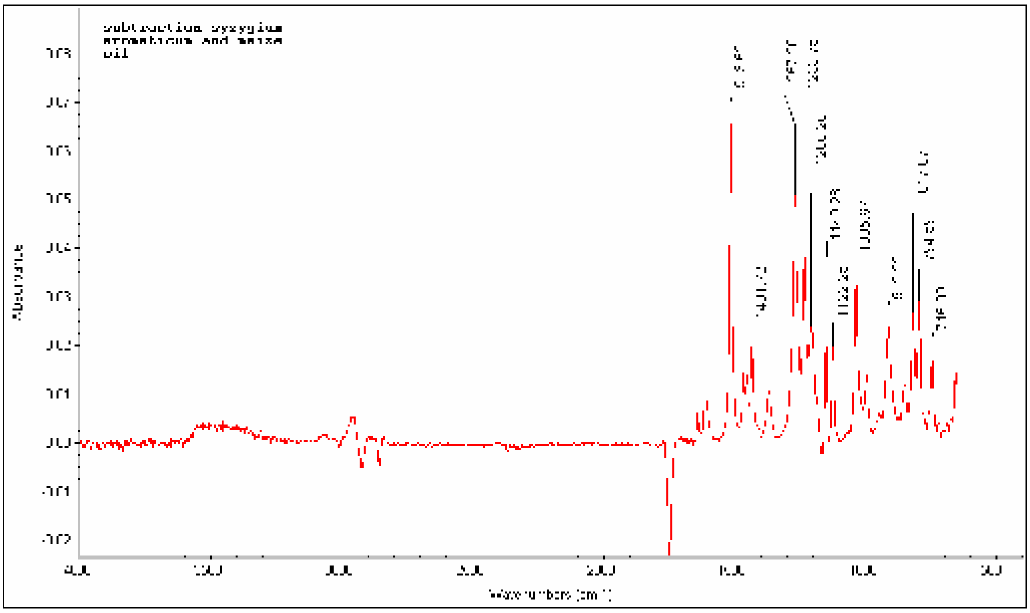

Figure B10: Subtraction *syzygium aromaticum* with maize oil

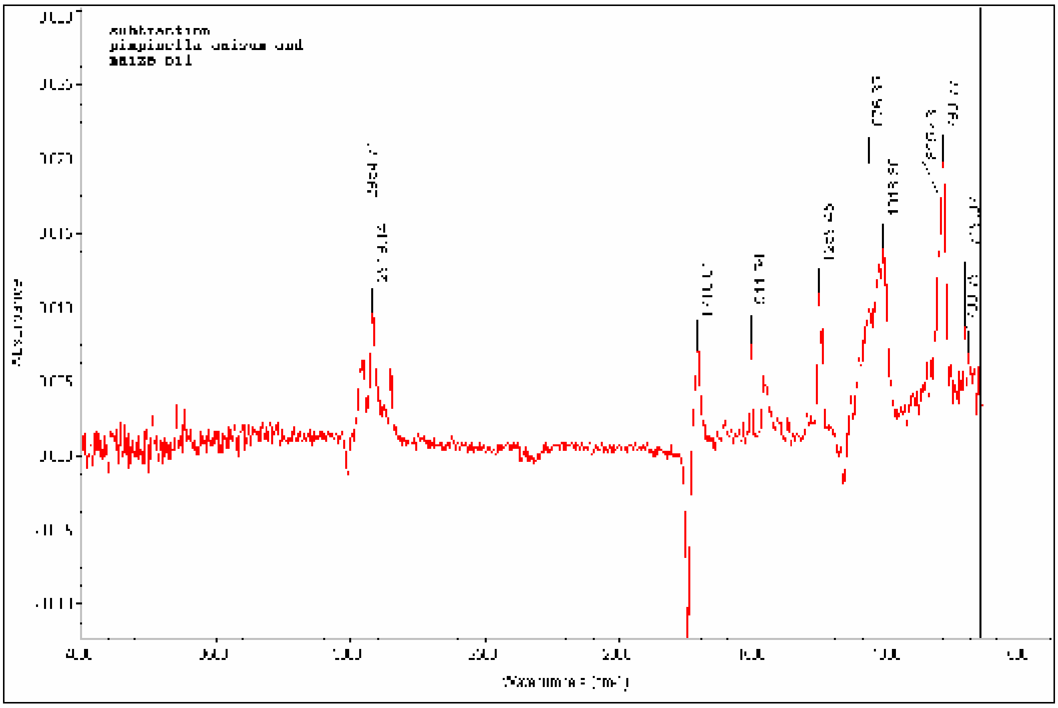

Figure B11: Subtraction *pimpinella anisum* with maize oil

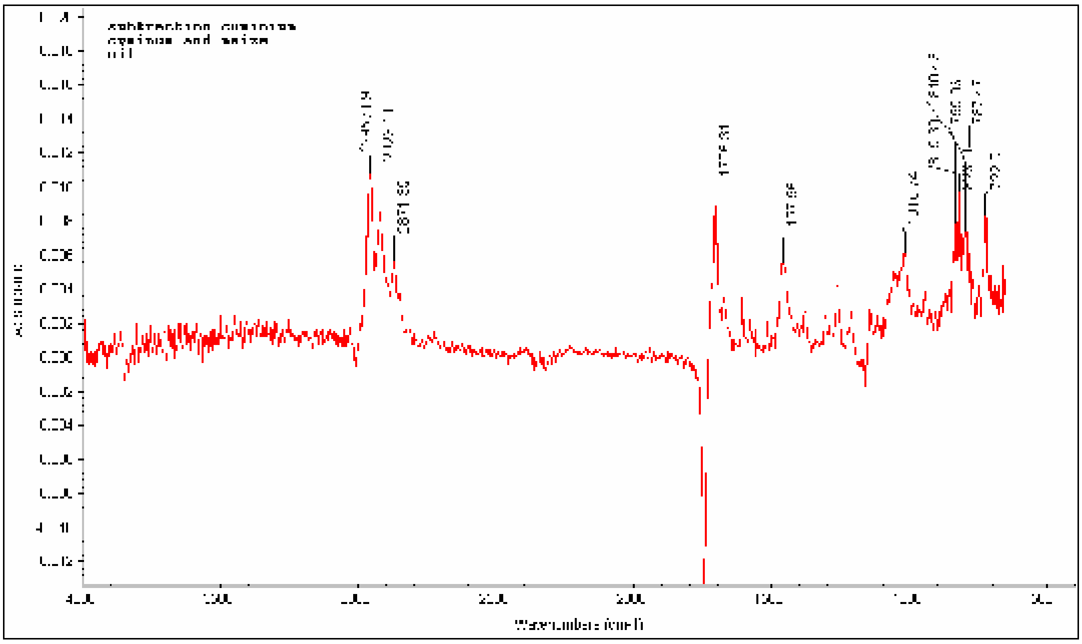

Figure B12: Subtraction *cuminium cyminum* with maize oil

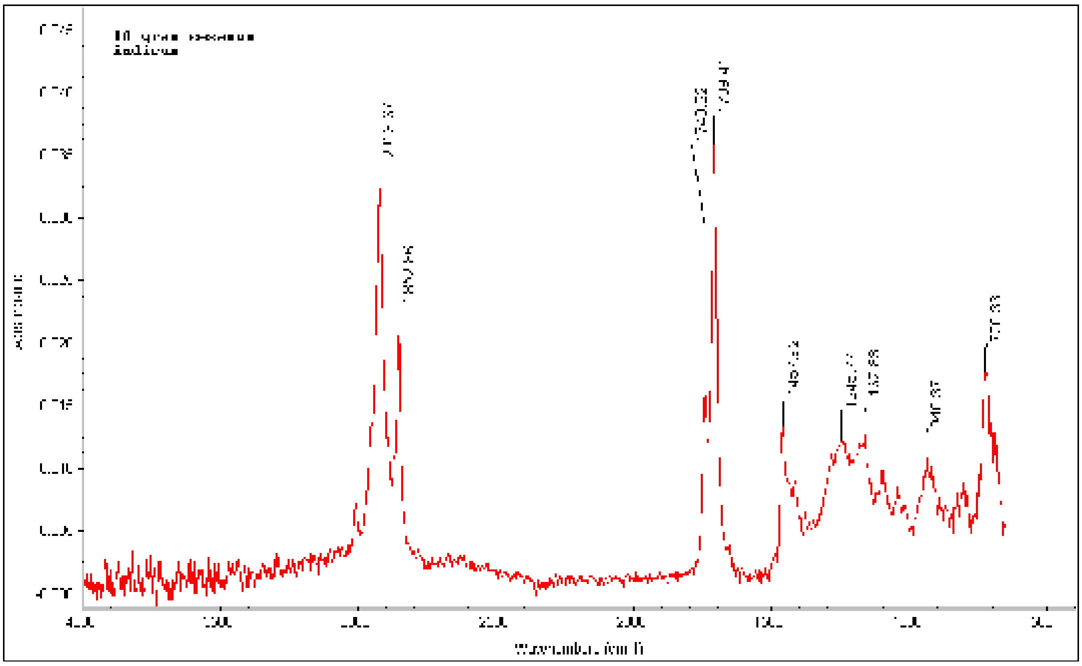

Figure B13: *Sesamum indicum* (10 gram) graph analysis.

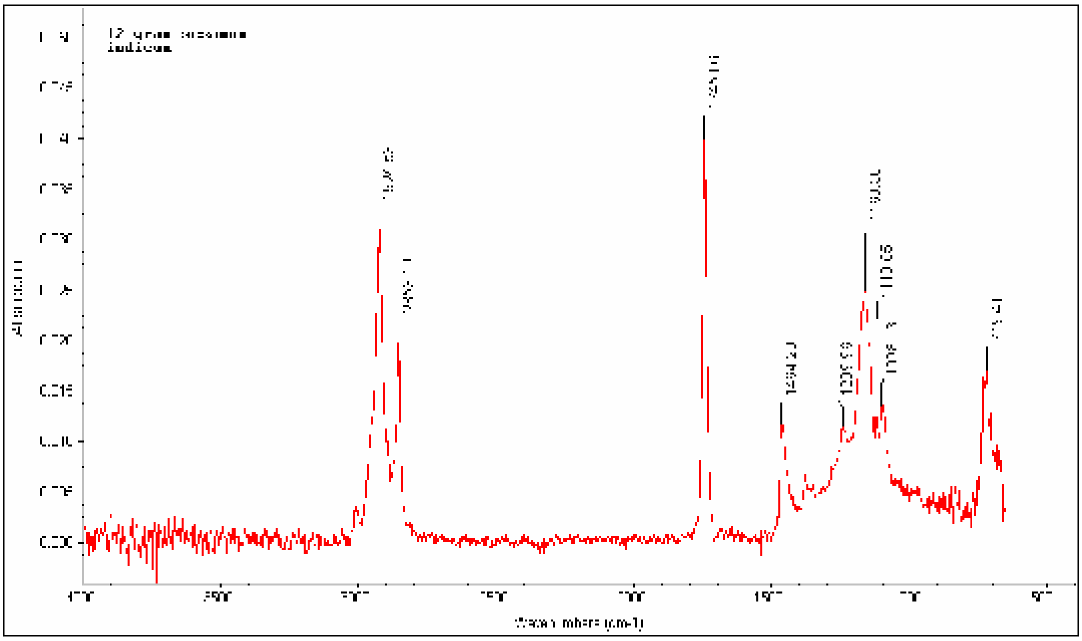

Figure B14: S*esamum indicum* (12 gram) graph analysis

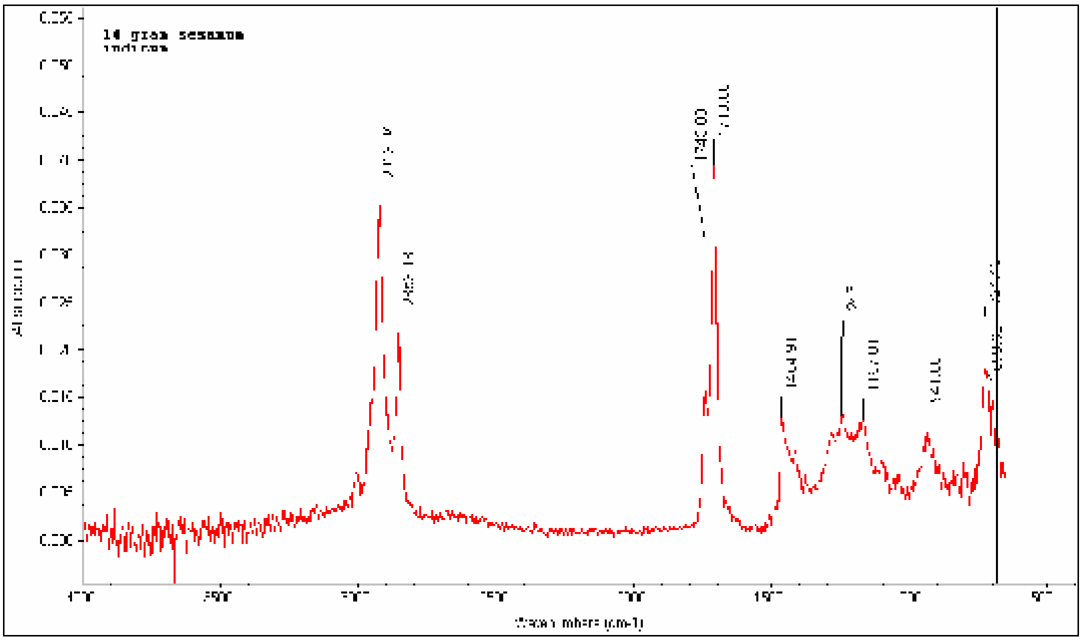

Figure B15: *Sesamum indicum* (14 gram) graph analysis

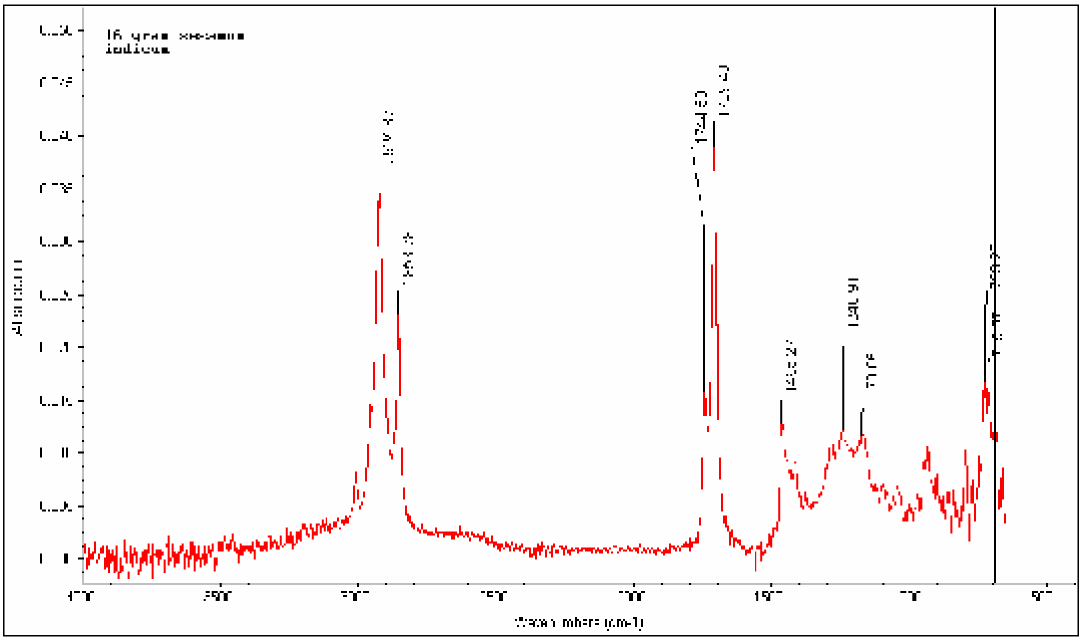

Figure B16: *Sesamum indicum* (16 gram) graph analysis

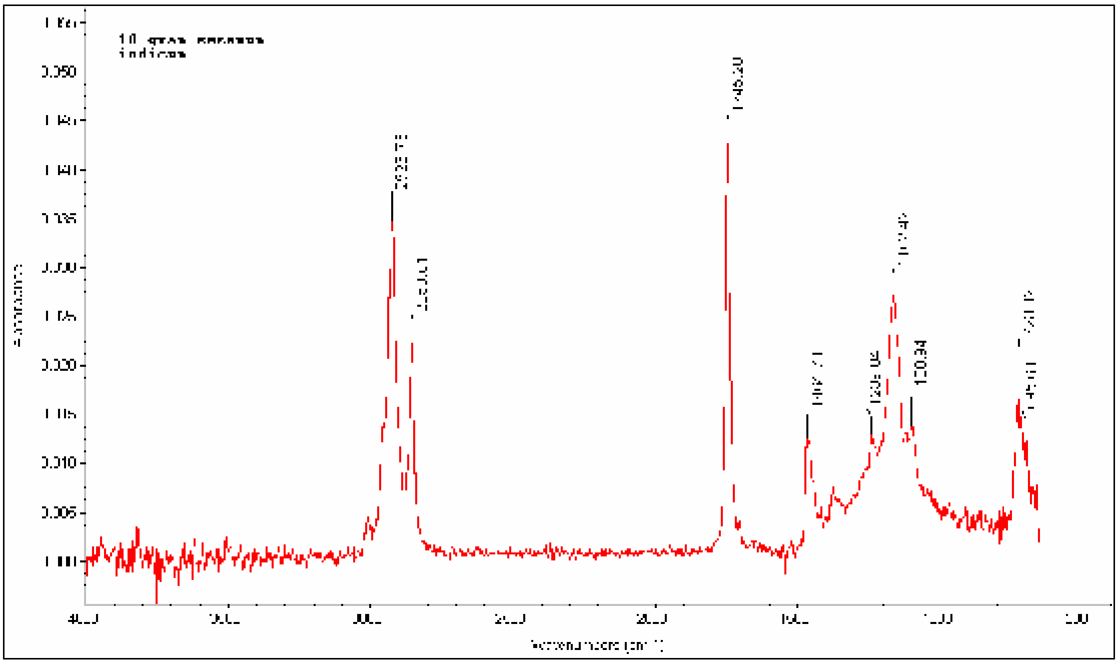

Figure B17: *Sesamum indicum* (18 gram) graph analysis

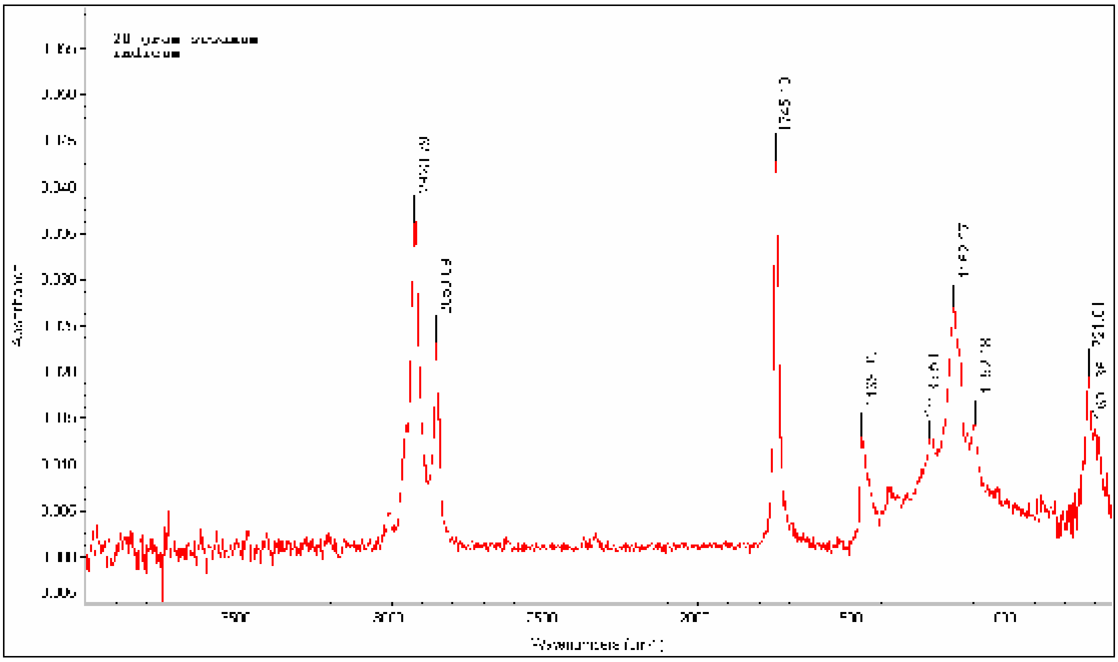

Figure B18: *Sesamum indicum* (20 gram) graph analysis