In The Name of Allah Most Merciful Most Compassionate



Sudan University of Science and Technology

### **College of Education**



**Department of Chemistry** 

# Extraction And Detection Of Basil Oil

## A Thesis Submitted In Partial Fulfillment Of The Requirement Of B.SC Degree In Chemistry

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الايـــة

## قَالَتَعَالَىٰ:

﴿ فِيهَا فَكِمَةٌ وَٱلنَّخْلُ ذَاتُ ٱلْأَكْمَامِ ١

وَٱلْحَبُّ ذُو ٱلْعَصْفِ وَٱلرَّيْحَانُ () }

صرق الله العظيم.

سورة الرغمن (11-12)

#### **DEDICATION**

To those who give me the sense of life and search for a world full of peace, tolerance, and mutual respect (My Mother and Father) To those who live the life with me whenever and whatever I go (My Friends). To those who prepare my life (My Relatives). To those who are a world in which I live (My Teacher)

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#### Abstract

Extraction of essential oil of basil by steam distillation. The percentage of the oil in the sample was formed total (0.20%). And calculated the acid value = 2.805, the saponification value =5.61, and the peroxide value =zero. The major constituents of the oil using gas chromatography.  $\alpha$ -pinene, linalool,limonene,  $\beta$ -pinene, camphor and methyl cinnamate.

#### المستخلص

إستخلص الزيت الطيار من الريحان عن طريق التقطير البخاري ووجد أن النسبة المئوية للزنت تساوي 0.20% وتم حساب رقم الحموضة = 2.805 ورقم التصبن = 6.611 ورقم البيروكسيد = 0 وتحديد المكونات الاساسية له بواسطة التصبن = 6.611 مجاز كروماتغرافيا الغاز , مواسطة β-pinene, linalool,limonene, β-pinene, and methyl cinnamate.

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#### Chapter One

#### 1. Introduction

#### 1.1 - Oils:

an oil is any neutral, non polar chemical substance. That is a viscous liquid at ambient temperatures and both hydrophobic (immiscible water literally <sub>((</sub>water fearing<sub>))</sub>) and lipophilic miscible with other oils literally <sub>((</sub>fat loving<sub>))</sub>.

Oils have a high carbon and hydrogen content and are usually flammable and slippery.

The several definition of oil includes classes of chemical compound that may be other wise unrelated in structure properties and uses. Oils may be animal, vegetable or petrochemical origin and uses. Oils may be volatile or non. Volatile. They are used for food, fuel and the manufacture of paints, plastics and other materials.

Specially prepared oils are used in some religious ceremonies as purifying agent.

Vegetable oils is obtained by pressing the seed and then extracting the oil with steam or water. The high protein residue that remains after oil extraction (oil cake) is a voluble for livestock.

Oils are classified in terms of drying, semi drying, and non. Drying oils. With this terminology is based on the degree of instauration of the oil. They are differentiated by properties such as freezing point (fp), Iodine value (Iv), saponification value (Sv)

Drying oil are highly un saturated materials. Typical iodine values are 140 to 180. Semi- drying oils are similar to drying oils but contain less un saturation. Typical iodine values are 100601,40 non- drying oils usually contains mono- unsaturated compounds. Typical iodine value are less than 100

#### 1-1-1 Vegetable Oils:-

production of vegetable oils from plants such as olive, soybean, sun flower coconut, and others. Oils, fats, food is the third essential.

Component of the two in 1823 AD. It also allowed the progress of scientific knowledge of component of oil extracted and the extent of its impact on human health.

#### 1-1-2 Types Of Vegetable Oils:-

#### 1-1-2-1 Essential oils.

An essential oil is a concentrated by hydrophobic liquid containing volatile aromatic compounds form plants. Essential oils are also known as a volatile oil, ethereal oils, or a ethereal, or a simply as the oil of the plant from which they were extracted such as oil of clove. An oil is "essential" in the sense that it carries a distinctive category for any medical, pharmacological or culinary purpose.

Essential oils are generally extracted by distillation. Steam distillation is often used. Other processes include expression or solvent extraction.

They are used in perfumes, cosmetics, soaps and other processes for flavoring food and drink and for adding scents to incense and household cleaning products.

Essential oils have been used medicinally in history. Medical applications proposed by those who sell medicinal oil range from skin treatments to remedies for cancer and often are based solely on historical accounts of use of essential oils for these purposes. Claims for the efficacy of medical treatment, and treatment of cancers in particular, are now subject to regulation in most countries.

As the use of essential oils has declined in evidence. Based medicine, one must consult older text books for much information on their use. Modern works are less inclined to generalize, rather than refer to (essential oils) as a class at all they prefer to discuss specific compounds, such as methyl salicy-late, rather that oil winter green.

Interest in essential oils are revived in recent decades with the popularity of aromatherapy, a branch of alter nattier medicine that claims that essential oil and other aromatic compounds have curative effects. Oil are volatilized or diluted in a carrier oil and used in massage, diffused in the air by a nebulizer heated over a candle flame, or burned as in cense.

The ear liest recorded mention of the techniques and methods used to produce essential oils are beloved to be that ibn- albaitar (1188-1248) an andalusian physician pharmacist and chemist.

#### 1-1-2-2 Volatile Oils:

Is mixtures of hydrocarbon and oxygenated compounds derived from these hydrocarbon.

The odour and taste (oxygenated constituents) which are to some extent soluble in water but more soluble in alcohol.

Volatile and essential oils, as their name implies, are volatile in steam. They differ entirely in both chemical and physical properties from fixed oils. practically all volatile oils consist of chemical mixtures that are often quit complex. They vary widely in chemical composition.

The disadvantage of volatile oil is technical constrains, high cost of initial invest mint. While the ability to obtain extracts (volatile oil) of composition very close to that of the natural products

The possibility of adjusting the selectivity, viscosity, by fine tuning the temperature and pressure (simultaneous extraction and fraction) and the absence of hydrolysis or rearrangement

The uses of volatile oils are decaffeinated coffee prepare hops extract (for the brewery industry), remove nicotine from tobacco, preparation of spice extract (ginger, paprika, celery) flavors (black tea, oak wood smoke) and plant oils (pure and free of the terpenes) which add nothing to the small and are oxidizable.

As a rule, volatile oils immiscible with water, put they are sufficiently soluble to impart their odor to water (the aromatic water).

#### **1.2 Method Of Extractions:**

#### $1.2.1 \Rightarrow$ Mechanical Extraction:

Oils can also be removed via mechanical extraction, termed (crushing) or (pressing) this method is typically used to produce the more traditional oils (e. g, olive) and its preferred by most <sup>(()</sup> health- food<sup>))</sup> customers in the united state and in Europe. There are several different types of mechanical extraction. Expeller-pressing extraction is common though the screw press, rampress and Ghana (powered mortar and pestle) are also used. Oil seed presses are commonly used in developing countries, among people for whom other extraction methods would be prohibitively expensive. The Ghana is primarily used in India-the amount of oil extracted using these methods very widely.

#### **1.2.2** ⇒ Hydrogenation:

Oils may be partially hydrogenated to produce various ingredient oils. lightly hydrogenated oils have very similar physical characteristics to regular soy oil but are more resistant to becoming rancid. Margarine oils need to be mostly solid at 90 F(32 C) so that the margarine does not melt in warm rooms, yet it need to be completely liquid at 98 F(37 C) so that it dose not leave a (lardy) taste in the mouth.

Hardening vegetable oil is done by raising a blend of vegetable oil and a catalyst in near- vacuum to very high temperatures, and introducing hydrogen. This causes the carbon atom of the oil to break double- bonds with other carbons, each carbon forming anew single- bond with a hydrogen atom. Adding these hydrogen atoms to the oil makes it more solid raises the smoke point, and makes the oil more stable. Hydrogenated vegetable oils differ in two major ways from other oils which are equally saturated.

During hydrogenation it is easier for hydrogen to come into contact with the fatty acids on the end of the triglyceride, and less easy for them to come into contact with the center fatty acid. This makes the resulting fat more brittle than a tropical oil, soy margarines are less "spreadable".

The other difference is that trans fatty acids (often called trans fat) are formed in the hydrogenation reactor, and may amount to as much as 40 percent by weight of a partially hydrogenated oil with their higher amount of trans fatty acids are increasingly thought to be unhealthy.

#### **1-2-3 Solvent Extraction :**

Most flowers contain too little volatile oil to undergo expression, their chemical components are too delicate and easily denatured by the high heat used in steam distillation.

Instead a solvent such as hexane or super critical carbon dioxide is used to extract the oil. Extracts from hexane and other hydrophobic solvent are called concretes. Which are a mixture of essential oil. Waxes, resins and other lipophilic (oil soluble) plant material "although highly fragrant concretes contain large quantities of non fragrant waxes and resins. Often, another solvent, such as ethyl alcohol which is more polar in nature is used to extract the fragrant oil from the concrete the alcohol is removed by evaporation leaving behind the absolute.

#### **1-2-4 Distillation:**

Most oils are distilled in a single process one exception is yang (canang odorata), which takes 22 hours to complete through a fractional distillation there condensed water is referred to hydrosol, hydrolate herbal distillate or plant water essence, which may be sold as another fragrant product. Popular hydrosols include rose water lavender water, lemon balm, clary sage and orange blossom water. The use of herbal distillates in cosmetics some plant hydrosols have unpleasant smells and are there fore not sold.

#### **1-2-4-1 Simple Distillation:**

In simple distillation. The vapor is immediately channeled into a condenser. Consequently, the distillate is not pure but rather its composition is identical to the composition of the vapors at the given temperature and pressure. That concentration follows raoults law.

An result, simple distillation is effective only one when the liquid boiling point differ greatly (rule of thumb is 25c) or then separation liquid from non volatile solid or oils for these cases. The vapor pressures of the components are usually sufficiently different that the distillate may be sufficiently pure for its intended purpose.

#### 1-2-4-2 Steam Distillation :

Like vacuum distillation, steam distillation is a method for distilling compounds which are heat sensitive. The temperature of the steam is easier to control than the surface of a heating element, and allows a high rate of heat transfer without heating at a very high temperature. This process involves bubbling steam through a heated mixture of the raw material. By raoults law, some of the target compound will vaporize (inaccordance with its partial pressure). The vapor mixture is cooled and condensed, usually yielding a layer of oil and a layer of water.

Steam distillation of various aromatic herbs and flowers can result in two products. An essential oils as wall as a watery herbal distillate. The essential oils are often used in perfumery and aromatherapy while the watery distillates have many applications aromatherapy. Food processing and skin care.

#### 1-2-4-3 molecular distillation:

Is vacuum below the pressure of 0.0torr. 0.01torr is one order of magnitude a bore high vacuum, where fluids are in the free molecular flow regime, i.e the mean free path of molecules is, comparable to the size of the equipment. The gaseous phase no longer exerts significant pressure on the substance to be evaporated, and consequently rate of evaporation no longer depends, on pressure. That is, because the continuum assumptions of fluid dynamics no longer apply, mass transport is governed by molecular dynamics rather than fluid dynamics thus, a short bath between the hot surface and the cold surface in necessary, typically by suspending a hot plate covered with a film of feed next to a cold plate with a line of sight in between. Molecular distillation is used industrially for purification of oils.

#### **1-2-4-4 Multi– Effect Distillation:**

The goal of multi- effect distillation is to increase the energy efficiency of the process, for use in distillation or in some cases one stage in the production of ultrapure water. The number of effects is proportional to the w.h/m<sup>s</sup> of water recovered figure, and refers to the volume of water recovered per unit of energy compared with single- effect distillation. One effect is roughly 636 kw.h/m<sup>3</sup>.

Multi- stage flash distillation can achieve more than 20 effects with thermal energy input, as mentioned in the article.

Vapor compression evaporation commercial larg- scale units can achieve around 72 effects with electrical energy in put according to a manufacturers. There are many other types of multi effect distillation (MED) in which multiple chambers, with intervening heat exchangers, are employed.

#### **1-2-4-5 Continuous Distillation:**

Continuous distillation an ongoing distillation in which a liquid mixture is continuously (without interruption) fed into the process and separated fractions are removed continuously as output steams as time passes during the operation. Continuous distillation produces at least two output fractions, including at least one volatile distillation fraction, which has boiled and been separately captured as a vapor condensed to a liquid. There is always a bottoms (or residue) fraction, which is the least volatile residue that has not been separated captured as u condensed vapor.

Continuous distillation differs from batch distillation in the respect that concentrations should not change over time continuous distillation can be run at a steady state for an arbitrary amount f time for any source material of specific composition. The main variable that affect the purity of products in continuous distillation are the reflux ration and the number of theoretical equilibrium stages (practically, the number of trays or the height of packing). Reflux is a flow from the condenser back to the column, which generates are cycle that allows a better separation with a given number of trays. Equilibrium stages are ideal steps where compositions achieve vapor liquid equilibrium repeating the separation process and allowing better separation given are flux ration. A column with a high reflux ration may have fewer stages, but it refluxes alarg amount of liquid giving a wide column with alarg holdup. Conversely, a column with a low reflux ratio must have alarg number of stages. Thus requiring a taller column.

#### 1-3 Basil Oil:

The basil is extracted from a herd known as ocimum basilicum.

The herb belongs to the lamiaceae family. The plant is an annual herb that grows up to one meter in height. The flower color rang from white to pink depending on the species of plant.

The leaves are religiously important In India and are considered a holy plant. The basil oil is highly use in aromatherapy as it awakens the mind to clarity of thought and acts as a mental stimulant.

#### **1-3-1 Basil Oil Description:**

General Name: Basil Oil Botanical

Name: Ocimum basilicum

Basil oil is also known as sweet basil oil, basil essential oil or oil of ocimum basilicum.

Buy pure and natural basil oil from our online store and take advantage of the discount offers.

The information provided on this website may not be accurate and complete and you ate also recommended to follow expert's guidance for the most effective use of pure basil oil.

# 1-4Theoratical-back ground of the physicochemical proportion of the basil oil

#### 1-4-1 Saponification Value: (SV)

Is a measure of the free acid and saponifiable ester groups. It is expressed as the number of milligrams of potassium hydroxide required to neutralize the free acid.

#### 1.4.2. Acid value (Av)

The milligrams of potassium hydroxide that needed to neutralize the amount of free acid present in one gram of oil

#### 1.4.3 Peroxide value (Pv)

the number of equivalents of peroxide per kilogram of oil

#### 1.4.4 iodine value (iv)

The iodine is the mass of iodine in grams that is consumed by 100 grams of a chemical substance

#### 1.5 physical properties

The oil has water viscosity and is pale greenish yellow in color.

#### **1.6 Toxicity of basil oil:**

A study of the essential oil showed antifungal and insectrepelling properties. A similar study reported in 2009 has confirmed that extracts from the plant are very toxic to mosquitoes. However the plant is not toxic to rats. Little in formation is available about any potential toxicity in humans.

#### 1.7 Uses:

- potential health effects:
- Invitro- anti oxidant
- antiviral- anti crobial
- supplementary treatment of stress asthma and diabetes in India
- a good tonic for the treatment of nervous disorder
- stress related headaches migraines and allergies.
- used to dear the mind and valve

- vomiting and cramp. And is also good when used for menstrual problems

#### **1.8 Objective:**

to extract the basil oil

to detect constituents of the basil oil

#### Chapter Two 2-Material And Methods

#### 2.1 Material

Authentic sample of basil oil were collected by Albasatten farm in Khartoum (Almogran) in summer on July 2014

#### 2.2 Methods

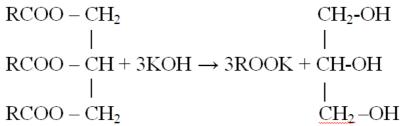
#### 2.2.1 Extraction Of Basil Oil

4000 g of the sample were cut into small pieces and put into around bottom flask in steam distillation system the distillation was heated till boiling at (80-100) for five hours, the oil was collected from the distillate after the separation of the two layers by a glass dropper (the upper layers).

Percentage of oil = (weight of oil / weight of sample)  $\times$  100

**2.3** Theoratical-back ground of the physicochemical proportion of the basil oil

2.3.1 Saponification Value (Sv)



Ig of oil was weighted in conical flask 12.5ml of alcoholic potassium hydroxide (0.5M) was added reflux on water bath about 45 minute, cooled and titrate against hydrochloric acid solution (0.5M) until the end point using phenolphthalein indicator ( $v_1$ ) repeated the titration without the sample ( $v_2$ )

 $SV = \frac{(v2 - v1) \times 28.05}{w}$ 

#### 2.3.2 A Cid Value (Av)

 $RCOOH + KOH \rightarrow RCOOK + H_2O$ 

Ig of oil was weighted in conical flask, 2ml of ethanol and ether was added and shaked well and titrated against standard hydroxide solution ( $v_1$ ).

The titration repeated without the sample  $(v_2)$ 

 $Av = \frac{(v2 - v1) \times 5.61}{w}$ 

#### 2.3.3 Peroxide Value (PV)

0.5g of oil was weighted in a conical flask, 6ml of mixture of acetic acid chloroform was added by measuring cylinder shaked well, 2 ml of for potassium iodide solution was added shaked well for minute and titrated against standard potassium thiosulfate solution using starch as indicator.

## $P_V = \frac{v \times n \times 1000}{w}$

#### 2.4 Gas Chromatograph (GC)

Is a commonly used analytic technique in many research and industrial laboratories for quality control as well as identification and quantitation of compounds in mixture GC is also a frequently used in many environmental and forensic laboratories because it allows for the detection of very small quantities.

Abroad variety of samples can be analyzed as long as the compounds are sufficiently thermally stable and volatile.

How does gas chromatography work???

Like for all other chromatographic techniques, a mobile and stationary phase are required for this technique the mobile phase (= carrier gas ) is comprised of an inert gas i, e, helium, argon, or nitrogen, the stationary phase consists of a packed column where the packing or solid support its lf acts as stationary phase, or is coated with the liquid stationary pause (= high boiling polymer). Most analytical gas chromatographs use capillary columns, where the stationary phase coats the wall of a small. Diameter tube directly (i. e. 0.25m film in a 0.32mm tube).

The separation of compound is based on the different strengths of interaction of the compounds with the stationary phase (likedissolves- like- rule).

The stronger the interacts with the stationary phase, and the more time it takes to migrate through the column (= longer

retention time). In the example above, compound  $\times$  interacts stronger with the stationary phase, and therefore behind compound in its movement through the column as a result, compound has a much shorter retention time than compound x. Which factors influence the separation of the components?

#### **2.4.1 Boiling Point**

The boiling point of a compound is often related to its polarity. The lower the boiling points is the shorter retention time usually is because the compound will spent main time in the gas phase. That is one of the main reasons why low boiling solvent (i.e- diethyl ether, dichloromethane) are used as solvents to dissolve the sample- the temperature of the column does net have to be above the boiling point because every compound has a non – zero vapor pressure at any given temperature, even solids that is the reason why we can smell compounds like camphor (0.065 mm Hg, 25c°), isoborneol (0.035mm Hg/25c°) naphthalene (0.84mm Hg/25c°), etc.

However their vapor pressures are fairly low compared to liquids (i,e water (25mm Hg /25c°), ethyl acetate (95 mm Hg /25c°), diethyl ether (25mm Hg /25c°).

## **2-4-2** The Polarity Of Components Versus The Polarity Of Stationary Phase On Column:

If the polarity of the stationary phase and compound are similar, the retention time increases because the compound interacts stronger with the stationary phase.

As aresult, polar compound lave long retention times on polar stationary phase and shorter retention times on non – polar columns using the same temperature.

Chiral stationary phase that are bused on amino a cid derivatives, cyclodextrins and chiral silanes are capable of separating enatimers because one enatiomer interacts slightly stronger phase, often due to steric effects or other very specific interaction.

For instance a cyclodextrin column is used in the determination of the enantiomeric excess in the chiral epoxidation experiment.

#### **2-4-3 Column Temperature:**

A excessively high column temperature results in very short retention time but also in very poor separation because all components mainly stay in the gas phase.

However, in order for the separation to occur the components need to be able to interact with the stationary phase, the retention time will decrease.

At the some time, the quality of the separation deteriorates, because the differences in retention times are not as pronound any more.

The best separations are usually observed for temperature gradients, because the differences in polarity and in boiling points are used here.

#### **2-4-4 Carrier Gas Flow Rate:**

A high flow rate reduces retention times, but a poor separation would be observed as well.

Like above, the components have very little time to interact with the stationary phase and are just being pushed through the column.

#### 2-4-5 Column Length:

A longer column generally improves the separation the trade – off is that the retention time increase proportionally to the column length and a significant peak broadening will be observed as well because of increased longitudinal diffusion in side the column.

One has to keep in mind that the gas molecules are not only traveling in one direction but also sideways and back wards.

This broadening is inversely proportional to the flow rate broadening is also observed because of the finite rate of mass transfer between the phase and because the molecules are taking different paths through the column.

#### 2-4-6 Amount Of Material Injected:

Ideally the peaks in the chromatogram display a symmetric shape, if too much of the sample is injected.

The peaks show a significant tailing.

Which causes a poorer separation most detector are relatively sensitive and do not need a lot of material in order to produce a detectable signal.

Strictly speaking under standard conditions only 1-2% of the compound injected into the injection port passes through the column because most GC instruments are operated in split – mode to prevent overloading of the column and the detector.

The split less mode will only be used if the sample is extremely low in concentration in terms of the analyte.

#### **Chapter Three**

3-1 Rsult of oil analysis

The weight of sample 4000g

The weight of oil 8g

The percentage of oil = weight of oil /weight of sample  $\times$  100

 $= 8/4000 \times 100 = 0.20\%$ 

- 3.2 Result of chemical properties:
- 3.2.1 Saponification value

 $SV = \frac{(V2 - V1) \times 28.05}{W} = \frac{0.2 \times 28.05}{1} = 5.61$ 

3.2.2 Acid value

Av = 
$$\frac{(V2-V1)\times 5.61}{W} = \frac{0.5\times 5.61}{1} = 2.805$$

3-2-3 peroxide value =

$$\frac{\nu \times N \times 1000}{W} = \text{m.eq/kg} = \text{zero}$$

Number	Retention Time	Area	Area %	Name
1.	1.901	1059937	8.57	<i>α</i> -pinene
2.	2.549	310855	2.5	-
3.	2.982	2480587	20.06	linalool
4.	3.286	619913	5.01	Limonene
5.	3.744	264734	2.14	$\beta$ -pinene
6.	3.875	466453	3.77	Camphor
7.	4.218	7160122	57.92	Methyl
				cinnamate

3-3 the major compound of oil in gas chromatography

Percent of compound = peak area / total area  $\times$  100

Total area = 12362601

 $\alpha$  = pinene % = 1059937 / 12362601 × 100 = 8.57% Linalool % = 2480587 / 12362601 × 100 = 20.06%  $\beta$ - pinene % 264734 / 12362601 × 100 = 2.14% *Camphor* % = 466453 / 12362601 × 100 = 3.77% Methyl cinnamate % = 7160122 / 12362601 × 100 = 57.92%

#### **3-4 Discussion :**

In this study a volatile oil was extracted from basil oil by steam distillation techniques the study covered the percentage yield, and studied GC. The major constituents of the oil using GC,  $\alpha$ -pinene, linalool, limonene  $\beta$ -pipene, camphor, methyl cinnamate.

Linalool is an organic compound with the chemical formula  $C_{10}$  H<sub>18</sub> O and chemical structure.



It is he colorless linalool occurs in nature (over species of plant plant produce linalool). Is used as ascent in 60-80% of perfumed hygiene products inter mediate.

Methyl cinnamate is an organic compound is the cemical formula  $C_{10}\,H_{10}\,O_2$ 

it is found naturally in a variety of plants.

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