## (1.0)Chapter one

### (1.1)Essential oil

An essential oil is a concentrated hydrophobic liquid containing volatile aroma compounds from plant. Essential oil are also known as volatile oils, ethereal oils, aetherolea, or 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 contains the characteristic fragrance of the plant that it taken from Essential oils do not from distinctive category for any medical, pharmacological, or culinary purpose they are not essential for health.

Essential oils are generally extracted by distillation, often by using steam.

Other processes include expression or solvent extraction. They are used in perfumes, cosmetics, soaps, and other products, 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 purposed by those who sell medicinal oils range from skin treatments to remedies for cancer and often are based solely on historical accounts of use of essential oils for this purposes .Claims for the efficacy of medical treatments, 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 textbooks for much information on their use modern works are less include to generalize ;rather than refer to "essential oils" as a class at all ,they prefer to discuss specific compounds ,such as methyl salicylate, rather than "oil of wintergreen".

## (1.1.1) Clove essential oil

It is antimicrobial, antifungal, antiseptic, antiviral, aphrodisiac and stimulating in natur.

Clove essential oil has long been used in treatment related to dental care, like toothaches and cavities, as well as for infections skin care, stress, headaches, respiratory problems weakness premature ejaculation, cholera and sties.

## (1.1.2)Description

Cloves are the unopened pink flower buds of the evergreen clove tree. The buds are picked by hands when they are pink and dried until they turn brown in color. Cloves are about  $1\2$  inch long and  $1\4$  inch in diameter and with their tapered stem, they resemble tiny nails. In fact, their English name is actually derived from the Latin word clavus, which means nails.

Although cloves have a very hard exterior, their flesh features an oily compound that is essential to their nutritional and flavor profile. Cloves have a warm, sweet and aromatic taste that evokes the sultry tropical climates where they are grown.

# (2.0) Chapter two

### (2.1) Clove

## (2.1.1) scientific classification

Table (2.1) scientific classification

Kingdom	Plant
Order	Myrtales
Family	Myrtaceae
Genus	Syzygium
Species	S. aromaticum

Binomial name = Syzygium aromaticum

Cloves are the aromatic flower buds of a tree in the family Myrtaceae, *Syzygium aromaticum*. They are native to the Maluku Islands in Indonesia, and are commonly used as a spice. Cloves are commercially harvested primarily in Indonesia, India, Madagascar, Zanzibar, Pakistan, Sri Lanka and Tanzania.

The clove tree is an evergreen tree that grows up to 8–12 m tall, with large leaves and sanguine flowers grouped in terminal clusters. The flower buds initially have a pale hue, gradually turn green, and then transition to a bright red when ready for harvest. Cloves are harvested at 1.5–2.0 cm long, and consist of a long calyx that terminates in four spreading sepals, and four unopened petals that form a small central ball.

Indian Ayurvedic medicine, Chinese medicine, and western herbalism and dentistry where the essential oil is used as an anodyne painkiller) for dental emergencies.

Cloves are used as a carminative, to increase hydrochloric acid in the stomach and to improve peristalsis. The essential oil is used in aromatherapy when stimulation and warming are needed, especially for digestive problems. Topical application over the stomach or abdomen are said to warm the digestive tract.

Applied to a cavity in a decayed tooth, it also relieves toothache.

Chinese medicine, cloves or *ding xiang* are considered acrid, warm, and aromatic, entering the kidney, spleen and stomach meridians, and are notable in their ability to warm the middle, direct stomach *qi* downward, to treat hiccough and to fortify the kidney *yang*.

Cloves may be used internally as a tea and topically as oil for hypotonic muscles, including for multiple sclerosis this is also found in Tibetan medicine.

### (2.1.2) Potential medicinal uses

The U.S. Food and Drug Administration (FDA) has reclassified eugenol (one of the chemicals contained in clove oil), downgrading its effectiveness rating. The FDA now believes not enough evidence indicates clove oil or eugenol is effective for toothache pain or a variety of other types of pain

Studies to determine its effectiveness for fever reduction, as a mosquito repellent, and to prevent premature ejaculation have been inconclusive. It remains unproven whether clove may reduce blood sugar levels.

Clove oil is used in preparation of some toothpastes and clovacaine solution, which is a local anesthetic used in oral ulceration and inflammation. Eugenol (or clove oil generally) is mixed with zinc oxide to form a temporary tooth cavity filling.

Clove oil can be used to anesthetize fish, and prolonged exposure to higher doses (the recommended dose is 400 mg/l) is considered a humane means of euthanasia.

### (2.1.3) Adulteration

### **Clove stalks**

Are slender stems of the inflorescence axis that show opposite decussate branching. Externally, they are brownish, rough, and irregularly wrinkled longitudinally with short fracture and dry, woody texture.

### Mother cloves (anthophylli)

The ripe fruits of cloves that are ovoid, brown berries, unioculor and one seeded. This can be detected by the presence of much starch in the seeds.

#### **Brown cloves**

Are expanded flowers from which both corollae and stamens have been detached.

#### **Exhausted cloves**

Have most or all the oil removed by distillation. They yield no oil and are darker in color.

### (2.1.4) **History**

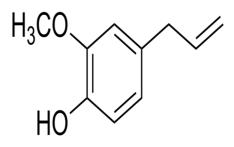
Archeologists have found cloves in a ceramic vessel in Syria, with evidence that dates the find to within a few years of 1721 BCE. In the third century BCE, a Chinese leader in the Han Dynasty required those who addressed him to chew cloves to freshen their breath.

Cloves were traded by Muslim sailors and merchants during the Middle Ages in the profitable Indian Ocean trade, the clove trade is also mentioned by Ibn Battuta and even famous *Arabian Nights* characters such as Sinbad the Sailor are known to have bought and sold cloves from India.

Until modern times, cloves grew only on a few islands in the Maluku Islands (historically called the Spice Islands), including Bacan, Makian, Moti, Ternate, and Tidore. In fact, the clove tree that experts believe is the oldest in the world, named *Afo*, is on Ternate. The tree is between 350 and 400 years old. Tourists are told that seedlings from this very tree were stolen by a Frenchman named

Poivre in 1770, transferred to France, and then later to Zanzibar, which was once the world's largest producer of cloves.

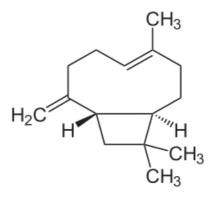
### (2.1.5) Active compounds



### IUPAC: 4-Allyl-2-Methoxyphenol

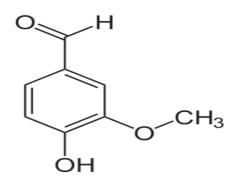
Eugenol comprises 72.90% of the essential oil extracted from cloves, and is the compound most responsible for clove aroma. Other important essential oil constituents of clove oil include 1acetyl eugenol, beta.caryophyllene and vanillin, 1crategolic acid, tannins such as bicornin, gallotannic acid, methyl salicylate (painkiller), the flavonoids eugenin, kaempferol, rhamnetin, and eugenitin, triterpenoids such as oleanolic acid, stigmasterol, and campesterol.

## (2.1.6) Caryophyllene



IUPAC names 4,11,11-trimethyl-8-methylene. bicyclo [7.2 .9]undec-4-ene

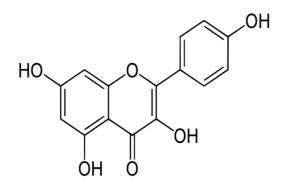
## (2.1.7) Vanillin



IUPAC name

4-Hydroxy-3-methoxybenzaldehyde

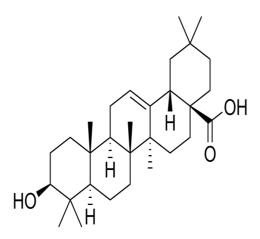
## (2.1.8) Kaempferol



IUPAC name

3,5,7-Trihydroxy-2(4.hydroxyphenyl)-4H.chromen-4-one

# (2.1.9) Oleanolic acid



### (2.2) Clove oil

Also known as clove oil is an essential oil extracted from the clove plant, Syzygium aromaticum. It has the CAS number 8000.34.8.

Clove oil is a natural analgaesic and antiseptic, used primarily in dentistry for its main ingredient eugenol. It can also be purchased in pharmacies over the counter as a home remedy for dental pain relief, mainly toothache. It is also often found in the aromatherapy section of health food stores, and is used in the flavoring of some medicines.

Madagascar and Indonesia are the main producers of clove oil.

Clove oil is used widely in microscopical preparation, as it is miscible with Canada balsam, and has a similar refractive index to glass (1.53).

Oil of clove is also used as an ingredient in cat determent sprays, coupled with garlic oil, sodium lauryl sulfate, and other ingredients.

### (2.2.1) **Types**

There are three types of clove oil.

#### **Bud** oil

Is derived from the flower buds of *S. aromaticum*. It consists of 60–90% eugenol, eugenol acetate, caryophyllene and other minor constituents.

#### Leaf oil

Is derived from the leaves of *S.aromaticum*. It consists of 82–88% eugenol with little or no eugenol acetate, and minor constituents.

#### Stem oil

Is derived from the twigs of *S. aromaticum*. It consists of 90–95% eugenol, with other minor constituents.

### (2.2.2) Efficacy

According to the U.S. Food and Drug Administration (FDA):

"Clove oil and eugenol, one of the chemicals it contains, have long been used topically for toothache, but the FDA has reclassified eugenol, down grading its effectiveness rating. The FDA now believes there isn't enough evidence to rate eugenol as effective for toothache pain".

In a 2006 study conducted by Kuwait University, researchers concluded that a clove preparation was equally effective as a benzocaine gel when administered as a topical anesthetic for intraoral injections.

In Australia, after major flooding throughout Queensland, clove oil mixed with water was used as a spray to kill mold.

### (2.2.3) **Toxicity**

Taking in large amounts of cloves or clove oil may cause nausea, vomiting, abdominal pain, diarrhea, burns in the mouth and throat, sore throat, seizures, difficulty breathing, rapid heartbeat, sleepiness, intestinal bleeding, and liver or kidney failure. More serious effects have been reported in young children, even with small doses.

Severe reactions may occur in people with allergy to cloves (about 1.5% of the population).

## (2.2.4) Regulation

Clove cigarettes, once somewhat popular in the United States, are now prohibited due to health hazards

In Germany, Commission E permits the sale and administration of clove oil as a medicinal herb.

### (2.2.5) Separation techniques

If it were possible to identify or quantitatively determine any element or compound by simple measurement, no matter what its concentration or the complexity of the matrix, separation technique would be of no value to the analytical chemist.

But most techniques fall short of this ideal, because of the interference with the required measurement by other constituents of the sample.

Many techniques for separating and concentrating the species of interest have just been devised.

Technique Phase system Solvent Liquid - liquid extraction Liquid - solid Solid phase extraction Gas-liquid Gas chromatography Gas - solid Liquid chromatograph Liquid-liquid Liquid - solid Thin layer chromatography Liquid – solid Liquid – liquid Ion – exchange and size exclusion Liquid – solid Chromatography Liquid – liquid Supercritical chromatography and Supercritical fluid. Liquid or solid – liquid. electro pharoses. Liquid – solid Capillary electro chromatography

Table (2.2.6) Types of separation techniques:

All separation techniques one or more achieve chemical equilibrium. Consequently, the degree of separation achieved can vary greatly according to experimental conditions. To a large extent; attainment of optimal condition to a large extent, attainment of optimal condition has to be approached empirically rather than by the application of a rigid theory.

## (2.3) Solvent extraction

## (2.3.1) Principle

Selective transfer of material in microgram to gram quantities between two immiscible liquid phases; separations based on solubility differences; selectivity achieved by PH control and complication.

### (2.3.2) Apparatus and instrumentation

Separating funnel is used for batch extraction and special glass apparatus for continuous extraction; automatic shakes used for discontinuous counter –current distribution.

# (2.3.3) Application

Concentration and determination of metals as trace and minor constituents according to type.

Batch methods are rapid, simple and versatile; application to very coide range of samples and concentrations.

## (2.3.4) Disadvantages

Sometimes requires large quantities of organic solvent; Poor resolution of mixture of organic materials except by counter – current distribution which slow.

Solvent extraction, sometimes call liquid-liquid extraction, it involves the selective transfer of a substance from one liquid phase to another usually; an aqueous solution of the sample is extracted with antimissile organic solvent.

## (2.4) Extraction of covalent, neutral molecules

In the absence of competing reactions in either phase and under controlled condition of a simple molecule can be predicted.

However, the value of the distribution ratio D May be PH dependent or May alert in presence of a complexing agent

It May also be effected ray association of extracting these effects are considered in turn.

### (2.4.1) Methods of extraction

Generally, one has a choice of three methods of extraction: batch extraction, continuous extraction and counter extraction.

The choice is generally determined by the distribution coefficient of the substance extracted and, in the case where separation is desired, by the clasenss of various is distribution coefficients involved.

Because of the speed and simplicity of batch extraction, this methods preferred when applicable.

### I. Batch extraction

When experimental conditions can be adjusted so that the fraction extracted is 0.99 or higher (DR) then a single or batch extraction will transfer the bulk of the desired substance to the organic phase.

Most analytical separator fall into this category.

The usual apparatus for batch extraction is a separation funnel.

Although many special type of funnels have been designed.

Even with Dr Equal to 10, two successive batch extractions will transfer 99% of the material to the organic phase.

If one chooses as criterion of separation that substance A be at least 99% extracted and substance B not more than 1% extracted one must have  $D_A$   $R_V>100$  and  $D_B R_V<0.01$  for a single (batch) extraction.

#### **II.** Continuous extraction

For relativity small  $DR_V$  values even multiple batch extraction.

Continuous extraction using volatile solvent cannot conveniently or economically be used too organic solvent is re apparatus in which the solvent is distilled with the aqueous phase, and returned to the extract collection flask in a continuous fashion.

#### **III.** Counter – current distribution

A Special Multiple – contact extraction is needed to effect the separation of two substances' whose D values are very similar.

In principle, counter – current distribution (CCD) could be carried out a series of spectra ray.

When solutes are allowed to distribute themselves between a pair of immiscible solvent, tremendous variations in the equilibrium concentration ratios are often observed. These differences can serve as the basis for quantities. In the usual case an aqueous solution of the sample is extracted with an organic liquid such as a hydrocarbon; ether an alcohol or the reverse may be case.

A surprising number of inorganic chlorides, nitrates, and their cyamates can be transferred quantitvely to various immiscible organic solvent.

### (2.4.2) Advantages of extraction procedure

Extraction procedure is used extensively by the organic chemist to effect separation; only recently, however, has the technique found widespread use in analysis. Separations by extraction have a number of attractive fearers when compared with the classical preparation method. one of these is its freedom form problems of co precipitation and postrecipition second, extraction procedures are well suited to the separation .of microgram quantizes whereas real difficulty is in attempting to precitat and collect traces of a substance third, a highly favorable equilibrium constant is not a necessary for successful extraction procedure in as much as multiple extraction can be readily performdure inasmuch as multiple extraction of one liquid by author is easily contracted this usually involves continuous evaporation of the extracting liquid followed by its condensation in such a way that it will pass through the solution

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containing the sample extraction processing of ten exhibits a high degree of selectivity. For inorganic actions a number of organic chelating reagents are available that permit extraction of certain but not other .the selectivity of these is often enhanced by control of the PH of the solution being extracted

### (2.4.3) Separation by distillation

Distillation is widely used for separation organic mixtures into their components. where the boiling points of compounds differ by only a few degrees a rather elaborate fractionating column is required to alive a clean separation in this device, continuous equilibration between a vapor and liquid phase is achieved .the liquid and vapor become rid her in the more volatile components with each succeeding point up the column, if column has sufficient length separation of substances of rather similar volatilities is possible.

Fraction distillation has not been widely employed in the separation of inorganic mixtures; applications have largely been confined to the isolation of a relatively volatile component from anon volatile residue. For such a separation a simple batch apparatus can be used; this consists of a distilling flask, as pray trap, and condenser ordinarily the distillate from such a device consists of a mixture of the value components and the so went.

A number of the elements can be converted to volatile compounds by suitable chemical treatment.

Carbon contained in organic carbonates is converted to carbon dioxide upon treatment with acid. The cabbonoli oxide is really distilled from aqueous solution and is collected by passage of the distilled through a solution containing barium hydroxide or by a absorbing the gas eon ascribe.

Nitrogen is readily volatilized from basic solition as ammonia. nitrates as other uitrogen oxides can be reduced to ammonia devarda's alloy.

Sulfur can be distilled from aqueous solution as hydrogen sulfide or sulfur dioxide. These gases are envolved from sulfides or sulfites upon treatment with a non volatile acid.

Hydrogen sulfide can be collected by passage of the vapor through a solution of cadmium ion; sulfur dioxide is oxidized to sulfate in a solution of hydrogen peroxide.

Hydrogen can be distills from aqueous solution in the element form or as the hydrogeohalides. Separation among the halogens is sometimes accomplished by selective oxidation of one if the halides followed by distillation of the elements thus, iodide can be separated from chloride and bromide by treatment with nitrous acid. Bromides can be separated from chlorides by selective oxidation with telluric acid or potassium hydrogen iodide.

Fluoride is readily separated from a variety of elements by distillation of a perchoric acid solution of the fluoride in the presence of silica. Fluosilicic acid IS the volatile product silicon can be volatilized as the tetra fluoride by treatment of silicates with hydrofluoric acid.

Boron is often converted to the volatile methyl borate by heating a sulfuric acid solution of boric acid with wetly alcohol. The boric ester can be collected by passing the distillate into analyze solution.

Arsenic, antimony, and tin can be separated from most other elements as well as from one other by suitable distillation procedures. Arsenic, in the trivalent state, can be quantitatively removed as the chloride from an aqueous solution containing sulfuric and hydrochloric acids.

Distillation at 110  $^{0}$ C is required. With the exception of germanium, no other elements distill under these conditions no other elements distill under these conditions. After removed of the arsenic, antimony tri-chloride can be separated by raising the boiling point of the mixture to 155  $^{0}$ C.

#### (2.5) Extraction chemistry

Extraction in chemistry is a separation process consisting in the separation of a substance from a matrix. It includes Liquid-liquid extraction, and Solid phase extraction.

Extractions often use two immiscible phases to separate a solute from one phase into the other. Typical lab extractions are of organic compounds out of an aqueous phase and into an organic phase. Common extract ants are arranged from ethyl acetate to water )ethyl acetate < acetone < ethanol < methanol < acetone :water (7:3) < ethanol :water (8:2) < methanol :water (8:2) < water) in increasing order of polarity according to the Hildebrand solubility parameter. The extract can be put back to dried form using a centrifugal evaporator or a freeze drier.

#### (2.6) Theory of an extraction between two phases

The distribution of a solute between two phases is an equilibrium condition described by partition theory. This is based on exactly how the analyze move from the water into an organic layer.

The distribution of absolute between two immiscible solvent is governed by the distribution low .if we assume that the solvent species A is allowed to distributed itself between water and an organic phase , the resulting equilibrium may be written

Where the subscript w and o refer to the water and organic phases, respectivites The ratio of Activities two phases will be constant and independent of the total quantity of A that is at any given temperature.

The Part ion Coefficient Takes the Form.

#### (2.7) Extraction of essential oils

Various extraction methods are used in the manufacture and extraction of essential oils, and the method used is normally dependent on what type of botanical material is being used.

His type of plant material that determines which method will be used to obtain the essential oil. But there are exceptions. For instance.  $CO_2$  is a great way to extract most oils, but the cost involved in following this method, would place it out of the financial reach of most people. Although the extraction of essential oils may sounds only to be of technical interest, it is one of the key points which determine the quality of the oil that is used, since a wrong or wrongly executed extraction, can damage the oil, and alter the chemical signature of the essential oil.

### (2.7.1) Maceration extraction method

With the maceration extraction method, the flowers are soaked in hot oil to have their cell membranes ruptured and the hot oil then absorbs the essence. The oil is then cleared of the botanical and decanted.

His is very much the same technique used in solvent extractions, where solvents are used instead of the hot oil as used in maceration.

Although stem distillation is must well know techniques for extracting essential oil from plants, there are several other method that are used to removed and can cent rate the aromatic constituents form materials.

## (2.7.2) Distillation extraction

Essential oils are produced in the cell of aromatic plants and one held in specialized of ants. They are released from the plants and collected most of tent through steam distillation.

## (2.7.3) Concretes and absolute: solvent extraction

Concepts and absolutes are highly concentrated aromatic methods extracted from plants; the multistep process includes first extracting the aromatic oil form plant material with such as hexane. After hexane is renewal what is left behind is a waxy substance called concrete. This semi Solid to solid highly fragment material contains a levy amount of pigments and wakes due to their waxy fixture, concretes are perfect for malign soiled perfumes. They have somewhat delicate, yet lang lasting aroma and are soluble in both carries oil and alcohol, though often its necessary to filter any insoluble waxes and solid material that remains from most of plant axes and nom. Aromatic material with ethyl alcohol. After the ethyl alcohol is removed, The mining substance is called an absolute. an absolute is most concentrated form of natural fragrance, with an arana close to A plant from which it come and highly regarded in natural perfumery. Abselutestius contain some wakes and pigment ad any with other constitutes from the plant, but are mostly copnesd of the concerned aromatic oil.

In addition, they usually contain a small percentage of alcohol reaming from second there process (typically up to 2 or 3 facet) A absolute differ Essential oil in that essential oil do not cant waxes, are muchboghter in color or number color, and have alighted Aram. Essential oil is typically used in silin care and for therafeutic purpose, absolute and concepts are for natural few funey.

### (2.7.4) CO<sub>2</sub> extracts: solvent extraction

 $CO_2$  extract display some of choractistic of belth essential oils and absolutes. Lille essential oils they contain many beneficial therapeutics. But an lilies absolutes, they are not scent extracted us in Carbon dioxide ( $CO_2$ ) gas under pressure at ambient temferdue. Under normal at mospeic conditions  $CO_2$  is gas, but when highly compersed it become suffer criticed.

Neither a gas now aligid. Super critical  $CO_2$  is an excellent arganic scleat that cambe used to extracted aromatic oils from plants. The beauity ( $CO_2$ ) extraction is that once the oil is extracted from the plant metrical, The  $CO_2$  returns to is gas out state by lowering its pressure, allowing the gas to griclly and comfblely dissipate.

Defending on the pressure pressed "select" an "fota" extract will result. Select extracts are created at lower pressure, and are move similar to essential oils constituents malle up the used natanity of the extracts are exited using high pressure and contain mane constituents of the plant , can be thicller an waxier, and more closely resemble the constituents of the whole plant rather than gust the essential oil fraction of the plant.

Because y punity  $CO_2$  extracts and they display some very favorable characteristic not found in essential oils,  $CO_2$  extracts are primarily used by the

food, body come, and ferbal industlines, yet  $CO_2$  extracts are also excellent for ceramatherofy and natural perfemerly. This extraction techniques is are atively new and exfensive technology that is more efficient in some ways than steam distillation become the process has the ability to coffere abroad sfectrum y the plant comfortable, giving a froyrance more true to the anginal plant matwied without use of chemical selents. Other sensilits are the extraction process halfpens at lover temperatures than steam distillation and that the carbon dioxide is nonteic, and is easily frame the extracted oil at the end of the process.

### (2.7.5) Organic extract

Organic extracts are made using modernized technology similar to the ancient method, en fleuraye gentel extraction of oil from precious flour by soiling them first in veltable fat them using alcohol ad asdvent to seprate the oils from the fat. Extraction process use only centiliced organic solvents such as fixed oils and alcohol to coa the aromatic essence out of the plant anoterial.

The resulting bio available essence, extracted with art added that, aplave the indicate aroma of the original plant material we are one of sale few in north America who carry the original extract made from: rose gas mine, cornation, argene blossom, and tubeose. These oils are best used for body care ant aramathy due to the alieness of the essences.

However, natural performs who wish to create organic perfunes will also love these oils. This new technology for the first time allow gasmine, car nation, and tuberose – in the form of organic extracts – to be used few true arama therafy purpose, whereas previasly they had only been available as absolute and there for had not been recommended for application.

### (2.8) Resin and other types of oils

We carry a few other types of natural aromatic oils that have been extracted ant resinalr retinoid – these aromatic essence are collected from the out of the fares when the tress are fapped (rubber tress an tress).

Another types of aromatic oil we offer comes fram the traditional of structive oils illation method that give rise to fossilize oil. In destructive distillation, the stanting such as Benzoinresin is super – heated and collect until an oil con from the selit stalivy methinal.

### (2.8.1) 10% dilution

We also offer 10% dilutions which are pue essential oil for absolute dilution in fractionated coconut oil. Our 10% dilutions are in expensive same of the more range and precioy oil that we often. They contain 10% of essential oil or absolute in a 90% fractionate coconut oil base.

Fractionated coconut oil has an extrely long shelf is clear and ode less, holds the frarane of aromatic oils well, and is beneficial for the skin.

# (3.0) Chapter Three

# (3.1) Material and Method

# (3.1.1) Material

## Clove

Clove is purchased from Omdurman market.

# (3.1.2) Chemicals

Alcoholic potassium hydroxide (0.5M)

Hydrochloric acid solution (0.5M)

Oil sample

Phenolphthalein indicator

Potassium hydroxide solution (0.5M)

Mixture of alcoholic and chloroform (3:2)

Potassium iodide 10% and potassium thiosulfate solution (0.05N).

# (3.1.3) Glassware

Conical flask, volumetric flask, burette, pipette, droppers, round bottom flask.

# (3.1.4) Instruments

Sensitive balance, IR

# (3.2) Methods

# (3.2.1) Extraction of oil from clove

100g of clove sample were weighed and placed into 1000 ml round bottom flask. The apparatus was setup for distillation with water using aclaisen connecting tube heating mantle was checked out from the service center to accommodate the large round bottom flask. The distillate was collected in a250 ml flask. The apparatus was allowed to cool before disassembling. The heating mantle was sufficiently cooled before returned it to the service center.

Result was recorded in table (4.1) in chapter four.

### (3.2.2) Determination of Saponification value of clove oil

2.0 g of oil were weighed into conical flask and 25ml of alcoholic potassium hydroxide 0.5M were added from burette, refluxed on water bath for about 45 minutes. The conical flask contents were cooled and titrated against hydrochloric acid solution 0.5M until the end point using phenolphthalein indicator ( $V_1$ ).

The titration was repeated without the sample (blank titration) ( $V_2$ ). The result in table (4.2)

### (3.2.3) Determination of Acid value in clove oil

10.0g of oil were weighed into a conical flask 20 ml of ethanol ether were added, shake and titrated against standard potassium hydroxide solution  $(V_1)$  using ph.ph indicator Titration was repeated without the sample  $(V_2)$ . The result until the color change from colorless to pink in table (4.3)

## (3.2.4) Determination of iodine value in clove oil

2.0g of cloves oil was weighed in a conical flask 10.0 ml of chloroform and 25 ml of wijis solution were added to conical flask. The conical flask was left in the dark for 30 minutes. 10ml of potassium iodide solution 10% were added followed by 100ml of distilled water and the liberated iodine was titrated against 0.1N sodium thiosulphate solution using starch as indicator ( $V_1$ ). The titration was repeated without the sample blank titration ( $V_2$ ). The result was in table (4.4) in chapter four.

# (4.0) Chapter four

## (4.1) Result and calculation

### Table (4.1) percentage of cloves oil

NO		First extraction	Second extraction
1	Wt of clove	100	100
2	Wt of oil	7.37g	6.9g
3	Percentage of extracted oil	7.37%	6.9%
	Mean Percentage value	7.37 + 6.9 / 2 = 7.1	35%

The percentage of cloves oil extracted was 7.135% by weight obtained result for properties carried out in the cloves oil.

## (4.1.1) for saponification value

## Table (4.2) saponification value

V <sub>1</sub>	<b>V</b> <sub>2</sub>
16ml	2.5ml

## Calculation

The volume of acid that reacted with the sample  $(V_3) = V_2 - V_1$ 

V<sub>3</sub>=16.0-2.5=13.5ml

Number of moles of HCL=MV\*10<sup>.3</sup>

 $0.5*13.5*10^{.3} = 6.75*10^{.3}$ 

Number of mole of KOH = $6.75 \times 10^3$ 

Weight of KOH (For 2g of oil) =

Weight of KOH (For 2mg oil) =

0.378\*1000=378 mg/g

Weight of KOH (in 1g of oil) =

378/2=189 mg/g

 $SV = (V_1 - V_2) * 28.5/w$ 

<sup>=</sup>(16-2.5)\*28.05/2 mg/g

=189.33 mg/g

## Table (4.3) for acid value

V <sub>1</sub>	V <sub>2</sub>
10ml	3.7ml

### Calculation

Number of mole of HCL =  $M.V*10^3$ 

=0.1\*6.3\*1<sup>.3</sup>

 $=6.3*10^4$  mole

Number of moles of KOH = $6.3 \times 10^4$  mole

Weight of KOH (For 5g of oil) =

Number of moles \*M.wt

6.3\*10<sup>4</sup>\*56.1= 0.0353 g

Weight of KOH (For 5mg of oil) =

0.0353\*1000=35.3 mg

Weight of KOH (For 1mg oil) =

7.06 mg/g

Or from the relation:

 $AV = (V_2 - V_1) * 5.61/w$ =6.3\*5.61/5

=7.06 mg/g

# Table (4.4) for iodine value

V <sub>1</sub>	V <sub>2</sub>
25ml	6ml

# Calculation

 $= (V_2 - V_1) * M * 12.69 / W$ 

= (25-6)\*0.1\*12.69/2

=12.055 g/100g

# IR

C=C Aliphatic	absorbance	$(1753.42) \mathrm{Cm}^{-1}$
C-0	absorbance	(1271.23) Cm <sup>-1</sup>
C=C aromatic	absorbance	(1600-1364.85) Cm <sup>-1</sup>
O-H	absorbance	(3506.85) Cm <sup>-1</sup>
C-C	absorbance	(1030.14) Cm <sup>-1</sup>
C-H Aliphatic	absorbance	(2931.51) Cm <sup>-1</sup>
=C-H aromatic	absorbance	(3068.49) Cm <sup>-1</sup>

# (5.0) Chapter five

## (5.1) Discussion

The results obtained for the percentage of cloves oil is 7.135% based on the type of clove which is used and the method of extraction.

The saponification value of cloves oil which found to be comparable to that of sesame oil, sun flower oil, olive oil and corn oil which found to be 189.33% 190.5% 190 and 1s90 respectively.

The higher iodine value the more c=c bonds are present in the oil.

The result obtained for the iodine value of clove oil 12.055 shows increase in the average degree of unsaturation of the oils as such the amount of iodine sesame oil 111.5, olive oil 84.5, and corn oil 115.5.

Acid value was used to quantify the amount of the acid present.

The acid value of clove oil was found to be 7.06 mg where as that of sesame oil was 2 mg.

# (5.2) Conclusion

The acid values of cloves oil was three times higher comparable to sesame oil.

Where as its iodine value and saponification value were compare much closed with sesame oil, sun flower oil and corn oil.

# (5.3) Recommendations

- 1. Expansion of studies on demodulation clove oil.
- 2. Expand the use of clove oil in medical fields.
- 3. Make people aware of dangers of the use by large quantities.
- 4. Make people aware of the extent of toxicity.
- 5. Distillation method is used to extract because it gives good results.

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