## CHAPTER ONE

## 1. 1 INTRODUCTION

The word perfume derives from the latin “per fumum” meaning through smoke, is fragrant liquid that is sprayed or rubbed on the skin or clothes to give a pleasant smell. Extraction of perfume from various plants resources is of ancient origin. Infact the natives from different tropical regions of the globe have long been extracting oil from numerous oil bearing plants. Human since the ancient time have known how to extract oil from their natural resources. Vegetable oils are naturally occurring esters of higher fatty acids and glycerol. They are widely distributed in nature and were first consumed as food. Later oils were discovered to be used as renewable raw materials for variety of non food production, for instance perfumes, disinfectants, inks to mention but a few.

## 1.2 BACKGROUND OF THE STUDY

Several thousands of plants distributed throughtout the world contain a group of odiferous, fragrance, oily products that are highly volatile organic substances collectively known as essential oils. “Essential” does not mean “most necessary” but rather the concentrated characteristics or quintessence of a natural flavor or fragrance raw material (Coulson et al, 2003). Therefore, perfume may be from essential oils of vegetables or plant origin. It is a complex mixture of aldehydes, ketones, hydrocarbons, alcoholic acid and short chain esters.

The existence of perfume on certain plants has been known for thousands of years. They can be found in leaves, flowers, stems, barks, and roots. Ancient Egytians extracted essential oils from plants tissues by steam distillation (Ogbu,2005). Other methods of isolating essential oils includes solvent extraction, expression, cold plate or enfleurage. Some of these methods have been adopted by essential oil extracting industries.

Information on perfume when the essential oils have been extracted from plants are of different type of oils and these will ultimately influence the smell of perfume over time namely:

1. The base oil (Base notes)- this will produce the scent that stays longest on the skin and for this reason it is usually added to the mixture e.g vanilla, ginnamon, sandalwood, mosses etc.

2. The middle oil (middle notes)- this also influences the smell of the perfume for quite sometime but not as long as the base note does e.g lemon grass, Yalang Ylang etc.
3. Top oil (top notes)- this is added to the mixture after the middle notes and may then be followed by some other substances which will help to

bridge the scents together e.g orchard, rose, bergamount, lemon,orchid etc.

A formulation or recipe that rightly blend with the oil or fragrance is used and the reason perfume differs is due to the formulation or recipe that will give pleasant odour. It is necessary to choose a good recipe of formulation.

Alcohol is added to the fragrance or essential oils as a primary solvent to reduce the strays of the oils.

Fixatives are also used with the other ingredients like water, essential oils and alcohol to lower the rate of evaporation of the fragrance of essential oils. The reason why a perfume losses its fragrance faster than normal is because only a little amount of fixative was used when preparing the perfume.

## 1.3 STATEMENT OF THE PROBLEM

There is a high demand of essential oils for various purposes such as medicinal, perfumery, soap making, insecticides to mention but a few have opened up wide opportunities for global warming.

Imported essential oils are very expensive to meet the demand of our local consumer industries, therefore it becomes necessary to source and extract these oils from local source. In particular perfumes that are usually imported can be produced locally from a vast variety of oil bearing plants yet to be explored. It has also been observed that high concentration of perfumes in the epidermal tissues can cause skin irritation or peeling due to poor formulation. This problem will be explored on this project. Solvent extraction is the safest method for extracting high quality oil because some herbs and spices cannot be extracted from enfleurage method but it has the disadvantage of having residual solvents in the essential oils.

The research is conducted in order to use hydrodistillation as one of the applicable methods to extract essential oils. Hydrodistillation has the advantage of no solvent residues as an alternative to conventional extraction techniques. This research will reveal the difference in yields by the methods.

## 1.4 JUSTIFICATION OF THE STUDY

It is hoped that from this research work, optimum extraction parameters like solvent type, solvent ratio, contact time and particle size and the quality of perfume would be established. The result obtained would add to the data bank that could help potential industrialist who intends to go into perfume production from plants. Consequently, more processing industries would lead to a higher production both for domestic consumption and export. Major consumers of essential oils are the perfumery, cosmetics, food and beverages and pharmaceutical industries.

Most of these industries in Nigeria depend on imported essential oils for their production and this makes their products very expensive. Interestling, some of these source of essential oils are common in Nigeria such as lemon grass, ginger, flowers, eucalyptus to mention but a few.

There is a high potential in this area and it becomes necessary to seek means to explore and exploit area with the aim of providing our local industries with these essential oils. There are great biodiversity in the ecosystem when it is tapped into the economy of Nigeria will be improved upon. It can also create numerous job opportunities for the youth as both industries and farmers will be employers of labour.

## 1.5 OBJECTIVE OF THE RESEARCH

The main objective of this research is to extract essential oil from lemon grass (cymbopogon citrasus) using solvent extraction, enfleurage, and hydrodistillation and formulating the perfume.

## 1.6 SCOPE OF THE RESEARCH

I. Investigate the effect of solvent nature on extraction in terms of yield and formulation of the extracted essential oil. Three methods will be used; solvent extraction, enfleurage and hydrodistillation. Attempt will be made to formulate the essential oil into perfume.

Ii To Formulate perfume using appropriate materials

Iii The composition of perfume and its concentration.

# CHAPTER TWO

## 2.1 THE HISTORY OF PERFUMERY

People's use of scents, aroma and fragrances has been used for many centuries.Since the beginning of recorded history, humans have attempted to mask or enhance their own odor by using perfume, which emulates nature's pleasant smells. Many natural and man-made materials have been used to make perfume to apply to the skin and clothing, to put in cleaners and cosmetics, or to scent the air. Because of differences in body chemistry, temperature, and body odors, no perfume will smell exactly the same on any two people.

Perfume comes from the Latin "per" meaning "through" and "fume," or "smoke." Many ancient perfumes were made by extracting natural oils from plants through pressing and steaming. The oil was then burned to scent the air. Today, most perfume is used to scent bar soaps. Some products are even perfumed with industrial odorants to mask unpleasant smells or to appear "unscented."

While fragrant liquids used for the body are often considered perfume, true perfumes are defined as extracts or essences and contain a percentage of oil distilled in alcohol. (Clark E. et al, 1975). A perfume is composed of three notes. The base note is what a fragrance will smell like after it has dried. The smell that develops after the perfume has mixed with unique body chemistry is referred to as the middle note. And the top note is the first smell experienced in an aroma. Each perfumery has a preferred perfume manufacturing process, but there are some basic steps. The notes unfold over time, with the immediate impression of the top note leading to the deeper middle notes, and the base notes gradually appearing as the final stage. These notes are created carefully with knowledge of the evaporation process of the perfume. The top note consists of small light molecules that evaporate quickly. The middle note forms the heart of main body of a perfume and act to mask the often unpleasant initial impression of base notes.

Traditionally perfumes were made from plant and animal substances and prepared in the form of waters, oils, unguents, powders, and incense. This last method of fragrance gives us our word 'perfume' which means 'to smoke through'. Most modern perfumes are alcohol-based and contain synthetic scents. While the term 'perfume' usually refers to fragrances in general, in the more technical language of the perfumer, a perfume must contain over 15% of fragrance oils in alcohol. The preferred fragrances for perfumes are by no means universal, but differ according to cultural dictates and fashions. In the sixteenth century, for example, pungent animal scents such as musk and civet were very popular. In the nineteenth century, by contrast, such animal scents were generally considered too crude, and light floral fragrances were favored. Perfumes were held in high esteem and widely employed in the ancient world. The wealthy would perfume not only the body, but their furnishings and their favorite horses and dogs. On ancient altars perfumes were offered to the gods, while in the kitchens of antiquity the same scents — Saffron, Cinnamon, Rose, Myrrh — might be used to flavor food and wine.

## 2.2 CONCENTRATION OF PERFUME

Perfume types reflect the concentration of aromatic compounds in a solvent, which in fine fragrance) is typically ethanol or a mix of water and ethanol. Various sources differ considerably in the definitions of perfume types. The intensity and longevity of a perfume is based on the concentration, intensity and longevity of the aromatic compounds (natural essential oils / perfume oils) used: As the percentage of aromatic compounds increases, so does the intensity and longevity of the scent created. Specific terms are used to describe a fragrance's approximate concentration by percent/volume on perfume oil, which are typically vague or imprecise. A list of common terms (Perfume-Classification) is as follows:

- Perfume extract, or simply perfume (Extract): 15-40% aromatic compounds.
- Esprit de Parfum (ESdP): 15-30% aromatic compounds, a seldom used strength concentration in between EdP and perfume.
- Eau de Parfum (EdP), Parfum de Toilette (PdT): 10-20% (typical 15%) aromatic compounds.
- Eau de toilette (EdT): 5-15% (typical 10%) aromatic compounds.
- Eau de Cologne (EdC): Chypre citrus type perfumes with 3-8% (typical 5%) aromatic compounds.
- Perfume mist: 3-8% aromatic compounds (typical non-alcohol solvent).

Splash (EdS) and Aftershave: 1-3% aromatic compounds (Larson and Martin, 1973)

## 2.3 PERFUME NOTES

Perfume is described in a musical metaphor as having three sets of notes, making the harmonious scent accord. The notes unfold over time, with the immediate impression of the top note leading to the keeper middle notes, and the base notes gradually appearing as the final stage. These notes are created carefully with knowledge of the evaporation process of the perfume.

- Top notes: The scents that are perceived immediately on application of a perfume. Top notes consist of small, light molecules that evaporate quickly. They form a person's initial impression of a perfume and thus are very important in the selling of a perfume. Also called the head notes

- Middle notes: The scent of a perfume that emerges just prior to when the top notes dissipate. The middle note compounds form the "heart" or main body of a perfume and act to mask the often unpleasant initial impression of base notes, which become more pleasant with time. They are also called the heart notes.
- Base notes: The scent of a perfume that appears close to the departure of the middle notes. The base and middle notes together are the main theme of a perfume. Base notes bring depth and solidity to a perfume. Compounds of this class of scents are typically rich and "deep" and are usually not perceived until 30 minutes after application.

The scents in the top and middle notes are influenced by the base notes; as well the scents of the base notes will be altered by the type of fragrance materials used as middle notes.(6)

## 2.4 SOURCES OF PERFUMES

### 2.4.1 Aromatics sources

**Plant sources**

Plants have long been used in perfumery as a source of essential oils and aroma compounds. These aromatics are usually secondary metabolites produced by plants as protection against herbivores, infections, as well as to attract pollinators. Plants are by far the largest source of fragrant compounds used in perfumery. The sources of these compounds may be derived from various parts of a plant. A plant can offer more than one source of aromatics, for instance the aerial portions and seeds of coriander have remarkably different odors from each other. Orange leaves, blossoms, and fruit zest are the respective sources of petitgrain neroli, and orange oils.

- Bark: Commonly used barks include cinnamon and cascarilla. The fragrant oil in sassafras root bark is also used either directly or purified for its main constituent, safrole, which is used in the synthesis of other fragrant compounds.
- Flowers and blossoms: Undoubtedly the largest and most common source of perfume aromatics. Includes the flowers of several species of rose and jasmine, as well as osmanthus, plumeria, miosa, tuberose, narcissus, scented geranium, cassie, ambrette as well as the blossoms

of citrus and ylang-ylang trees. Although not traditionally thought of as a flower, the unopened flower buds of the clove are also commonly used. Most orchid flowers are most commercially used to produce essential oils or absolutes, except in the case of Vanilla, an orchid, which must be pollinated first and made into seed pods before use in perfumery.

- Fruits: Fresh fruits such as apples, strawberries, cherries unfortunately do not yield the expected odors when extracted; if such fragrance notes are found in a perfume, they are synthetic. Notable exceptions include litsea cubeba, vanilla, and juniper berry. The most commonly used fruits yield their aromatics from the rind; they include citrus such as oranges, lemons, and limes. Although grapefruit rind is still used for aromatics, more and more commercially used grapefruit aromatics are artificially synthesized since the natural aromatic contains Sulfur and its degradation product is quite unpleasant in smell.
- Leaves and twigs: Commonly used for perfumery are lavender leaf, patchouli, sage, violets rosemary, and citrus leaves. Sometimes leaves are valued for the "green" smell they bring to perfumes, examples of this include hay and tomato leaf.
- Resins: Valued since antiquity, resins have been widely used in incense and perfumery. Highly fragrant and antiseptic resins and resin-containing perfumes have been used by many cultures as medicines

for a large variety of ailments. Commonly used resins in perfumery include labdanum, frankincense, myrrh, Perusbalsam, gum benzoin. Pine and fir resins are a particularly valued source of terpenes used in the organic synthesis of many other synthetic or naturally occurring aromatic compounds. Some of what is called amber and copal in perfumery today is the resinous secretion of fossil conifers.

- Roots, rhizomes and bulbs: Commonly used terrestrial portions in perfumers include iris rhizomes, Vetiver roots, various rhizomes of the ginger family.
- Seeds: Commonly used seeds include Tonka bean, carrot seed, coriander, caraway, cocoa, nutmeg, mace, cardamom, and anise.
- Woods: Highly important in providing the base notes to a perfume, wood oils and distillates are indispensable in perfumery. Commonly used woods include sandalwood, rosewood, Agarwood, birch, cedar, juniper, and pine. These are used in the form of macerations orry-distilled (rectified) forms.

**Animal sources**

- Ambergris: Lumps of oxidized fatty compounds, whose precursors were secreted and excelled by the sperm whale. Ambergris should not be confused with yellow amber, which is used in jewelry. Because the harvesting of ambergris involves no harm to its animal source, it remains one of the few animalic fragrance agents around which little controversy now exists.

- Castoreum: Obtained from the odorous sacs of the North American beaver.
- Civet: Also called Civet Musk, this is obtained from the odorous sacs of the civets, animals in the family Viverridaemongoose. The World Society for the Protection of Animals investigated African civets caught for this purpose.
- Hyraceum: Commonly known as "Africa Stone", is the petrified excrement of the Rock Hyrax.
- Honeycomb: From the honeycomb of the honeybee. Both beeswax and honey can be solvent extracted to produce an absolute. Beeswax is extracted with ethanol and the ethanol evaporated to produce beeswax absolute.
- Deer musk: Originally derived from the musk sacs from the Asian musk deer, it has now been replaced by the use of synthetic musk sometimes known as "white musk".

Other natural sources

- Lichens: Commonly used lichens include oakmoss and treemoss thalli.

- "Seaweed": Distillates are sometimes used as essential oil in perfumes. An example of commonly used seaweed is Fucus vesiculosus, which is commonly referred to as bladder wrack. Natural seaweed fragrances are rarely used due to their higher cost and lower potency than synthetics.

### 2.4.2 Synthetic sources

#### Aroma compound

Many modern perfumes contain synthesized odorants. Synthetics can provide fragrances which are not found in nature. For instance, Calone, a compound of synthetic origin, imparts a fresh ozonous metallic marine scent that is widely used in contemporary perfumes. Synthetic aromatics are often used as an alternate source of compounds that are not easily obtained from natural sources. For example, linalool and coumarin are both naturally occurring compounds that can be inexpensively synthesized from a terapnes  Orchid scents (typically salicylates) are usually not obtained directly from the plant itself but are instead synthetically created to march the fragrant compounds found in various orchids.

One of the most commonly used classes of synthetic aromatic by far are the white musk. These materials are found in all forms of commercial perfumes as neutral background to the middle notes. This musk is added in large quantities to laundry detergents in order to give washed clothes a lasting "clean" scent.

## 2.5 ESSENTIAL OILS

Essential oils are natural fragrances extracted from virtually every parts of a plant. Essential oils are volatile and liquid aroma compounds from natural sources usually plants, they are not oils in a strict sense, but often share with oils poor solubility on water. It contains mainly volatiles as terpenoids, benzenoids, fatty acid derivatives and alcohols. The Federal Drug Agency (FDA) and other authorities recognize essential oils generally as safe. Although essential oils are widely used on cosmetics the uses of essential oils are determined by their chemical, physical and sensory properties, which differ greatly from oil to oil. Each of the individual chemical compounds that can be found on oil contributes to the overall character.

## 2.4 SOURCES OF ESSENTIAL OIL

Essential oils are desired from various types and parts of plant.

Some of them include:

### 2.4.1 LEMONGRASS

Family: Poaceae (Gramineae), Cymbopogon species

The genus has about 55 species, most of which are native to South Asia, Southeast Asia and Australia. Two major types have considerable relevance for commercial use: East Indian lemongrass (*Cymbopogon flexuosus* ) is native to India, Sri Lanka, Burma and Thailand, whereas West Indian lemongrass (*Cymbopogon citratus*) is assumed to originate in Malaysia. The plants grow in dense clumps up to 2 meters in diameter and have leaves up to 1 meter long. Further *Cymbopogon martini (Roxb.) J.F. Watson var. martini*, which is native to India and cultivated in Java is worth mentioning as it also grows in Bhutan and is extracted for palmarosa oil. Another species with commercial relevance is citronella grass (*Cymbopogon winterianus Jowitt*) which also stems from India, but is today grown throughout the tropics.

The reported life zone for lemongrass is 18 to 29 degrees centigrade with an annual precipitation of 0.7 to 4.1 meters with a soil pH of 5.0 to 5.8

(East Indian) or 4.3 to 8.4 (West Indian). The plants need a warm, humid climate in full sun. They grow well in sandy soils with adequate drainage. Since the plants rarely flower or set seed, propagation is by root or plant division. The plants are harvested mechanically or by hand about four times each year with the productive populations lasting between four and eight years Extensive breeding programs have developed many varieties of lemongrass.

The quality of lemongrass oil is generally determined by the content of citral, the aldehyde responsible for the lemon odor. Some other constituents of the essential oils are -terpineol, myrcene, citronellol, methyl heptenone, dipentene, geraniol, limonene, nerol, and farnesol .West Indian oil differs from East Indian oil in that it is less soluble in 70 percent alcohol and has a slightly lower citral content.

Lemongrass is used in herbal teas and other nonalcoholic beverages in baked goods, and in confections. Oil from lemongrass is widely used as a fragrance in perfumes and cosmetics, such as soaps and creams. Citral, extracted from the oil, is used in flavoring soft drinks in scenting soaps and detergents, as a fragrance in perfumes and cosmetics, and as a mask for disagreeable odors in several industrial products. Citral is also used in the synthesis of ionones used in perfumes and cosmetics.

As a medicinal plant, lemongrass has been considered a carminative and insect repellent. West Indian lemongrass is reported to have antimicrobial activity .Oil of West Indian lemongrass acts as a central nervous system depressant .Oil of East Indian lemongrass has antifungal activity .The volatile oils may also have some pesticide and mutagenic activities .*Cymbopogon nardus* is a source of citronella oil. *Cymbopogon martinii* is reportedly toxic to fungi.(Moore, Michael 2006).

### 2.4.2 **Grass Oils**

Several important essential oils are derived from grasses and used in the perfume industry. The genus Cymbopogon (formerly Andropogon) is especially rich in perfume species.

### 2.4.3 **Jasmine**

A highly esteemed perfume, jasmine is cultivated in southern France and surrounding areas. The main source is <u>Jasminum officinarum</u> var. grandiflorum, which is usually grafted on a less desirable variety. The flowers are picked as soon as they are open and the oil is extracted by effleurage.

### 2.4.4 Violet

One of the most popular perfumes is made from violets. Blue and pur0le double varieties of Viola odorata, native to Europe, are grown mainly in the vicinity of Nice. Solvents or maceration with hot fats extracts the oil. It occurs in such minute amounts that 15 tons of flowers are required to obtain only one pound of oil. Genuine violet perfume is rare and expensive, and it has been almost entirely replaced by synthetic products derived from ionone.

### 2.4.5 Lavender

Lavender perfumes are very old and were used by the Romans in their baths. It is still one of the most important scents. It is a low shrub with terminal spikes of very fragrant bluish flowers. . The oil is important in the manufacture of Eau de Cologne and other perfumes and is also used in soaps, cosmetics and medicine as a mild stimulant. Lavender water, a mixture of the oil in water and alcohol, is popular in England (Yardley brand).

### 2.4.6 Otto of Roses

This is valuable oil that is also called **Attar of Roses**. It has been one of the most favorite perfumes either in combination with other oils or alone. Bulgaria supplied most the commercial supply in the 20th

Century. The damask rose, Rosa damascena, was the main source. Flowers are picked in the early morning just as they are opening and are distilled immediately. The oil is colorless at first but gradually turns to a yellowish or greenish color.

### 2.4.7 **Rosemary**

Rosemary, (Rosmarinus officinalis), is a native of the Mediterranean region. It has long been a favored sweet-scented plant and has been important in the folklore of many countries. It is one of the least expensive and most refreshing odors. The plant is a small evergreen shrub that is cultivated in Europe and the United States. The oil is extracted by distillation of the leaves and fresh flowering tops or by solvent extraction. The leaves are valuable as a spice.

## 2.5 **CHEMICAL CONSTITUENTS OF ESSENTIAL OILS**

Pure essential oils are mixtures of more than 200 components, normally mixtures of terpenes or phenylpropanic derivatives, in which the chemical and structural differences between compounds are minimal. They can be essentially classified into two groups:

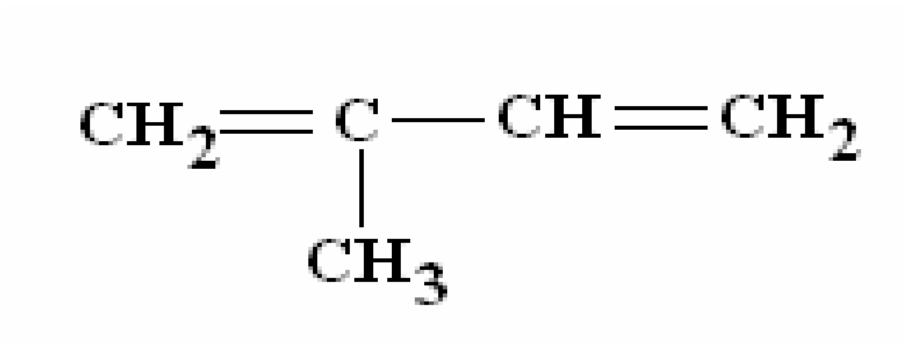
**Volatile fraction:** Essential oil constituting of 90–95% of the oil in weight, containing the monoterpene and sesquiterpene hydrocarbons, as well as their oxygenated derivatives along with aliphatic aldehydes, alcohols, and esters.

**Nonvolatile residue:** that comprises 1–10% of the oil, containing hydrocarbons, fatty acids, sterols, carotenoids, waxes, and flavonoids. (10)

## 2.6 CHEMICAL STRUCTURE OF ESSENTIAL OIL

### 2.6.1. Hydrocarbon:

Essential Oils consist of Chemical Compounds that have hydrogen and carbon as their building blocks. Basic Hydrocarbon found in plant are isoprene having the following structure.



(Isoprene)

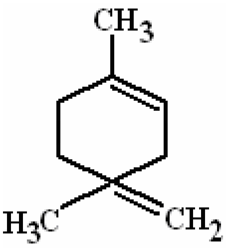
### 2.6.2. Terpenes:

Generally have names ending in " **ene**." For examples: Limonene, Pinene, Piperene, Camphene, etc. Terpenes are anti-inflammatory, antiseptic, antiviral, and bactericidal. Terpenes can be further categorized in monoterpenes, sesquiterpenes and diterpenes. Referring back to isoprene units under the Hydrocarbon heading, when two of these isoprene units join head to tail, the result is a monoterpene, when three join, it's a sesquiterpene and four linked isoprene units are diterpenes.

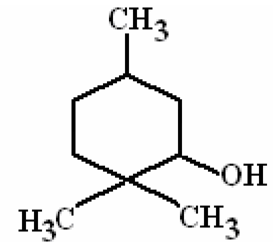
**i. Monoterpenes [$C_{10}H_{16}$ ]**

Properties: Analgesic, Bactericidal, Expectorant, and Stimulant.

Monoterpenes are naturally occurring compounds, the majority being unsaturated hydrocarbons ($C_{10}$). But some of their oxygenated derivatives such as alcohols, Ketones, and carboxylic acids known as monoterpenoids.




(Limonene)




(Menthol)

The branched-chain $C_{10}$ hydrocarbons comprises of two isoprene units and is widely distributed in nature with more than 400 naturally occurring monoterpenes identified.

**ii. Sesquiterpenes**

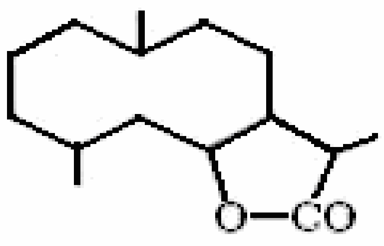
Properties**:** anti-inflammatory, anti-septic, analgesic, anti-allergic.

Sesquiterpenes are biogenetically derived from farensyl pyrophosphate and in structure may be linear, monocyclic or bicyclic. They constitute a very large group of secondary metabolites, some having been shown to be stress compounds formed as a result of disease or injury.

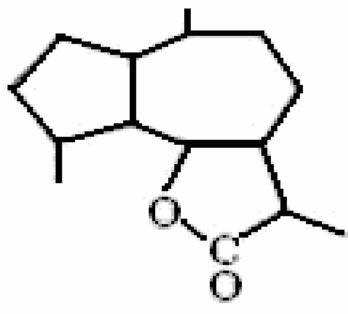
### iii. Sesquiterpene Lactones:

Over 500 compounds of this group are known; they are particularly characteristics of the Composite but do occur sporadically in other families. Not only have they proved to be of interest from chemical and chemotaxonomic viewpoints, but also possess many antitumor, anti-leukemia, cytotoxic and antimicrobial activities. They can be responsible for skin allergies in humans and they can also act as insect feeding deterrents.

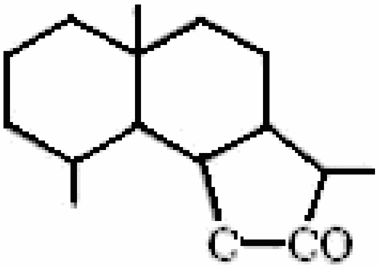
Chemically the compounds can be classified according to their carboxylic skeletons; thus, from the germacranolides can be derived the guaianolides, pseudoguaianolides, eudesmanolides, eremophilanolides, xanthanolides, etc.



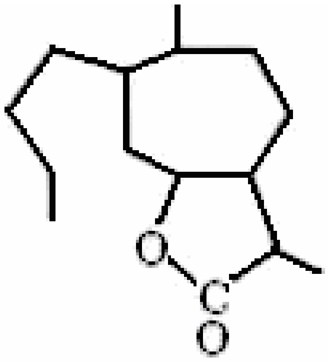

Germacranolides




Guaianlides




Eudesmanolides




Xantnanolides

A structural feature of all these compounds, which appears to be associated with much of the biological activity, is the α, β -unsaturated- γ-lactones.

**iv. Diterpenes**

Properties: anti-fungal, expectorant, hormonal balancers, hypotensive

Diterpenes are made of up four isoprene units. This molecule is too heavy to allow for evaporation with steam in the distillation process, so is rarely found in distilled essential oils. Diterpenes occur in all plant families and consist of compounds having a $C_{20}$ skeleton. There are about 2500 known diterpenes that belong to 20 major structural types. Plant hormones Gibberellins and phytol occurring as a side chain on chlorophyll are diterpenic derivatives. The biosynthesis occurs in plastids and interestingly mixtures of monoterpenes and diterpenes are the major constituents of plant resins. In a similar manner to monoterpenes, diterpenes arise from metabolism of geranyl geranyl pyrophosphate (GGPP).

### 2.6.3. Alcohols

Properties: anti-septic, anti-viral, bactericidal and germicidal.

Alcohols are the compounds which contains Hydroxyl compounds. Alcohols exist naturally, either as a free compound, or combined with a terpenes or ester. When terpenes are attached to an oxygen atom, and hydrogen atom, the result is an alcohol. When the terpene is monoterpene, the resulting alcohol is called a monoterpenol. Alcohols have a very low or totally absent toxic reaction in the body or on the skin. Therefore, they are considered safe to use.

### 2.6.4. Aldehydes:

Properties: anti-fungal, anti-inflammatory, anti-septic, anti-viral, bactericidal, disinfectant, sedative.

Medicinally, essential oils containing aldehydes are effective in treating Candida and other fungal infections.

Example: Citral in lemon, Lemongrass and lemon balm and Citronellal in lemongrass, lemon balm and citrus eucalyptus.

### 2.6.5. Acids

Properties: anti-inflammatory.

Organic acids in their free state are generally found in very small quantities within Essential oils. Plant acids act as components or buffer systems to control acidity.

### 2.6.6. Esters

Esters are formed through the reaction of alcohols with acids. Essential oils containing esters are used for their soothing, balancing effects. Because of the presence of alcohol, they are effective antimicrobial agents. Medicinally, esters are characterized as antifungal and sedative, with a balancing action on the nervous system. They generally are free from precautions with the exception of methyl salicylate found in birch and wintergreen which is toxic within the system.

### 2.6.7. Ketones:

Properties: anti-catarrhal, cell proliferant, expectorant, vulnery.

Ketones often are found in plants that are used for upper respiratory complaints. They assist the flow of mucus and ease congestion. Essential oils containing ketones are beneficial for promoting wound healing and encouraging the formation of scar tissue. Ketones are usually (not always) very toxic**.** The most toxic ketone is Thujone found in mugwort, sage, tansy, thuja and wormwood oils. Other toxic ketones found in essential oils are pulegone in pennyroyal, and pinocamphone in hyssops. Some non-toxic ketones are jasmone in jasmine oil, fenchone in fennel oil, carvone in spearmint and dill oil and menthone in peppermint oil.

### 2.6.8. Lactones

Properties: anti-inflammatory, antiphlogistic, expectorant, febrifuge.

Lactones are known to be particularly effective for their anti-inflammatory action, possibly by their role in the reduction of prostaglandin synthesis and expectorant actions. Lactones have an even stronger expectorant action then ketones. (11)

## PARAMETERS EFFECTING YIELD & QUALITY OF ESSENTIAL OILS

- Mode of Distillation;

  Technique for the distillation should be chosen on basis of oil boiling point and nature of herb as the heat content and temperature of steam can alter the distillation characteristics.

- Proper design of equipment's;

  Improper designing of tank, condenser or separators can lead to loss of oils and high capital investments.

- Material of Construction of equipment's;

  Essential oils which are corrosive in nature should be preferably distilled in stills made of resistant materials like aluminum, copper or stainless steel.

- Condition of Raw material;

  Condition of raw material is important because some materials like roots and seeds will not yield essential oil easily if distilled in their natural state. These materials have to be crushed, powdered or soaked in water to expose their oil cells.

- Filling of raw material / steam distribution;

  Improper loading of the herb may result in steam channeling causing incomplete distillation.

- Operating parameters like steam injection rate inlet pressure/ condensate temperature;

  Proper control of injection rates and pressure in boiler operated units is necessary, to optimize the temperature of extraction for maximal yield

  Temperature of condensate should not be high as can result in oil loss due to evaporation.

- Time given for distillation

Different constituents of the essential oil get distilled in the order of their boiling points. Thus the highest boiling fractions will be last to come over when, generally, very little oil is distilling. If the distillation is terminated too soon, the high boiling constituents will be lost.

- Pre condition of tank / equipments

  Tanks should be well steamed for multiple crop distillation

  Tank / equipments should not be rusted for quality oil.

## 2.7 TREATMENT / PURIFICATION OF ESSENTIAL OILS

Essential oil as obtained from the oil separator is in crude form. It may have suspended impurities and appreciable moisture content. It might even contain some objectionable constituents which degrade its flavour/fragrance quality.

The presence of moisture and impurities adversely affects the keeping quality of oil and accelerates the polymerization and other undesirable reactions.

**Remedies**

Filtration of oil through Marking is a simple method for removal of impurities. For removal of the moisture and free the oil of suspended impurities, addition of a drying agent like Anhydrous Sodium Sulphate to the oil, standing the oil overnight will get the oil clear of moisture. On industrial scale use of high speed centrifuge to clarify the essential oils can be also used.

Essential oils can also be rectified or re-distilled to remove objectionable constituents, dark colour or polymerized oil. (12)

## 2.8 STORAGE OF ESSENTIAL OILS

- Oils should be stored in shaded areas away from direct sunlight
- Should always be filled up to brim level
- Containers / bottles should be well cleaned / steamed

## 2.9 PERFUME EXTRACTION METHOD

Fragrance extraction refers to the extraction of aromatic compounds from raw materials, using methods such as distillation, solvent extraction, expression [or enfleurage. The results of the extracts are either essential oils, absolutes, concretes, or butters, depending on the amount of waxes in the extracted product.

To a certain extent, all of these techniques tend to distort the odour of the aromatic compounds obtained from the raw materials. Heat, chemical solvents, or exposure to oxygen in the extraction pr process denature the aromatic compounds, either changing their odour character or rendering them odourless.

### 2.9.1 Extraction of Fragrance

Before perfumes can be composed, the odorants used in various perfume compositions must first be obtained. Synthetic odorants are produced through organic synthesis and purified. Odorants from natural sources require the use of various methods to extract the aromatics from the raw materials. The results of the extraction are essential oils, absolutes, concretes, or butters, depending on the amount of waxes in the extracted produced.

All these techniques will, to a certain extent, distort the odor of the aromatic compounds obtained from the raw materials.

**Maceration/Solvent extraction**: This is most used and economically important technique for extracting aromatics in the modern perfume industry. Raw materials are submerged in a solvent that can dissolve the desired aromatic compounds. Maceration lasts anywhere from hours to months. Fragrant compounds form woody and fibrous plant materials are often obtained in this manner as are all aromatics from animal sources. The technique can also be used to extract odorants that are too volatile for distillation or easily denatured by heat. Commonly used solvents for maceration/solvent extraction include hexane, and dimethyl ether. The product of this process is called a "concrete."

Figure 2.5 Solvent extracts separation

**Supercritical fluid extraction:** A relatively new technique for extracting fragrant compounds from a raw material, which often employs Supercritical CO2. Due to the low heat of process and the relatively nonreactive solvent use in the extraction, the fragrant compounds derived often closely resemble the original odor of the raw material.

**Ethanol extraction**: A type of solvent extraction used to extract fragrant compounds directly from dry raw materials, as well as the impure oily compounds materials resulting from solvent extraction or effleurage. Ethanol extraction is not used to extract fragrance from fresh plant materials since these contain large quantities of water, which will also be extracted into the ethanol.

### 2.9.2 Distillation

Distillation is the process in which a liquid or vapour mixture of two or more substance is separated into its component fractions of desired purity, by the application and removal of heat. In simplier term, implies vaporizing or liberating the oils from the trichomes / plant cell membranes of the herb in presence of high temperature and moisture and then cooling the vapour mixture to separate out the oil from water.

Distillation is a common technique for obtaining aromatic compounds from plants, such as orange blossoms and roses. The raw material is heated and the fragrant compounds are re-collected through condensation of the distilled vapor. Distilled products, whether through steam or dry distillation are known either as essential oils or ottos.

Today, most common essential oils, such as lavender, peppermint, and eucalyptus, are distilled. Raw plant material, consisting of the flowers, leaves, wood, bark, roots, seeds, or peel, are put into an alembic (distillation apparatus) over water.

**Steam distillation**: Steam from boiling water is passed through the raw material for 60-105 minutes, which drives out most of their volatile fragrant compounds. The condensate from distillation, which contains both water and the aromatics, is settled in a Florentine flask. This allows for the easy separation of the fragrant oils from the water as the oil will float to the top of the distillate where it is removed, leaving behind the watery distillate. The water collected from the condensate, which retains some of the fragrant compounds and oils from the raw material, is called hydrosol and is sometimes sold for consumer and commercial use. This method is most commonly used for fresh plant materials such as flowers, leaves, and stems.

**Advantages:**

The advantage of steam distillation is that it is a relatively cheap process to operate at a basic level, and the properties of oils produced by this method are well known. Newer methodology, such as sub critical water extraction, may well eventually replace steam distillation, but so far even contenders such as carbon dioxide extraction - although establishing a firm market niche - have not really threatened to take over as the major preparative technique.

**Dry/destructive distillation**: Also known as rectification, the raw materials are directly heated in a still without a carrier solvent such as water. Fragrant compounds that are released from the raw material by the high heat often undergo anhydrous pyrolysis, which results in the formation of different fragrant compounds, and thus different fragrant notes. This method is used to obtain fragrant compounds from fossil amber and fragrant woods (such as birch tar) where an intentional "burned" or "toasted" odour is desired.

**Fractionation distillation**: Through the use of a fractionation column, different fractions distilled from a material can be selectively excluded to manipulate the scent of the final product. Although the product is more expensive, this is sometimes performed to remove unpleasant or undesirable scents of a material and affords the perfumer more control over their composition process.

**Hydro distillation**

Hydro / water distillation is one of the simplest, oldest and primitive process known to man for obtaining essential oils from plants.

Mostly used by small scale producers of essential oils in water / hydro distillation the plant material is almost entirely covered with water as suspension in the still which is placed on a furnace. Water is made to boil and essential oil is carried over to the condenser along with the steam. It is useful for distillation of powders of spices and comminuted herbs etc. The Deg Bhabka method of India using copper stills is an example of this technique. Some process becomes obsolete to carry out extraction process like Hydro Distillation which often used in primitive countries. The risk is that the still can run dry, or be overheated, burning the aromatics and resulting in an Essential Oil with a burnt smell. Hydro distillation seems to work best for powders (i.e., spice powders, ground wood, etc.) and very tough materials like roots, wood, or nuts. (13)

**DISADVANTAGES OF THE HYDRO DISTILLATION**

- The process is slow and the distillation time is much longer thereby consuming more firewood / fuel making process uneconomical.
- Variable rate of distillation due to difficult control of heat.
- Extraction of the herb is not always complete
- As the plant material near the bottom walls of the still comes in direct contact with the fire from furnace there is a likelihood of its getting charred and thus imparting an objectionable odor to the essential oil
- Prolong action of hot water can cause hydrolysis of some constituents of the essential oils such as esters etc which reacts with the water at high temperatures to form acids & alcohols
- Not suitable for large capacity / commercial scale distillations
- Not suitable for high boiling hardy roots / woody plant materials

### 2.9.4 Enfleurage

This is the absorption of aroma materials into solid fat or wax and extracting the odorous oil with ethyl alcohol. Extraction by enfleurage was commonly used when distillation was not possible because some fragrant compounds denature through high heat. This technique is not commonly used in the present day industry due to its prohibitive cost and the existence of more efficient and effective extraction methods.

Enfleurage is a two-step process during which the odour of aromatic materials is absorbed into wax or fat, and then extracted with alcohol.

## 2.10 FORMULATION OF PERFUMES

Perfume oils usually contain tens to hundreds of ingredients and these are typically organized in a perfume for the specific role they play.

These ingredients can be roughly grouped into four groups:

- Primary scents (Heart):- can consist of one or a few main ingredients for a certain concept such as “rose”. Alternatively, multiple ingredients can be used together to create an “abstract” primary scent that does not bear a resemblance to a natural ingredient. For instance, jasmine and rose scents are commonly blended for abstract floral fragrances.

- Modifiers: These ingredients alter the primary scent to give the perfume a certain desired character for instance fruit esters may be included in a floral the cherry scent in cherry cola can be considered a modifier.
- Blenders: A large group of ingredients that smooth out the transitions of a perfume between different ”layers” or bases. These themselves can be used as a major component of the primary scent. Common blending ingredients include linalool and hydroxycitronellal.
- Fixatives: they are used to support the primary scent by bolstering it. Many resins, wood scents, and bases are used as fixatives.

  The top, middle, and base notes of a fragrance may have separate primary scents and supporting ingredients. The perfume’s fragrance oils are then blended with ethyl alcohol and water aged in tanks for several weeks and filtered through processing equipment to respectively allow the perfume ingredients in the mixture to stabilize and to remove any sediment and particles before the solution can be filled into the perfume bottles.

# CHAPTER THREE

# MATERIAL AND METHODS

## 3.1 SAMPLE PREPARATION

Fresh Lemongrass sample was collected from the garden in Caritas University Enugu in Enugu State. The sample was allowed to dry for about three days in the laboratory. The leaves were later cut into slices to reveal the tighter inner stem until when ready for use.

Figure 3.1 lemon grass

## 3.2 APPARATUS AND REAGENTS

- A retort stand
- 500ml Separation funnel
- 250ml and 100ml Beakers
- Electronics weighting balance (V 100)
- Water bath (DC 1000)
- Mortar and pestle

- 500ml Round bottom flask
- Knife
- Aluminum foil
- Electric heater
- Distilled water
- N-hexane
- Ethanol
- Olive oil

## 3.3 PROCEDURE FOR SOLVENT EXTRACTION METHOD

130g of the dry sample of lemongrass were weighed from the sliced lemongrass sample and placed in a 500ml clean flat bottom flask. 600ml of N-hexane solvent were poured into the 500ml flask and stopped. The flask and content were allowed to stand for 24hrs; this was done to extract all the oil content in the lemongrass and for complete extraction. After which the extract was decanted into another 500ml beaker. 200ml of Ethanol were added to extract the essential oil since essential oil is soluble in Ethanol. The mixture was then transferred to 500ml separating funnel and separated by a process called liquid/liquid separation process. The content of the separating funnel was and allowed to come to equilibrium, which separated into two layers (depending on their different density). The lower Ethanol extract and the upper Hexane layer were collected into two separate 250ml beaker and were placed in a water bath at 78$^{o}$C. This was done to remove the Ethanol leaving only the natural essential oil. The yield of oil was determined by weighing the extract on an electronic weighing balance. The difference between the final weight of the beaker with extract and the initial weight of the empty beaker gave the weight of essential oil.

Figure 3.3. solvent extraction separation

### 3.4 PROCEDURE FOR ENFLEURAGE METHOD

130g of the dry sample of lemongrass were weighed out and pounded with mortar and pestle (to reveal the tighter inner stem). The pounded sample was then placed in a 500ml beaker. About 70ml of light-flavored olive oil were warmed and mixed with the mashed lemongrass (to allow for efficient absorption of the essential oil). The beaker was covered with aluminum foil and shaken until the lemongrass was distributed throughout the oil. It was then allowed to stand for 24hours at room temperature for proper absorption. 140ml Ethanol were added to absorb the essential oil leaving behind the light-flavoured olive oil and the lemongrass residue. The Ethanol extract was decanted and placed on a water bath at $78^oC$ to vaporize the Ethanol leaving behind the essential oil. The yield of oil was determined by weighing on an electronic weighing balance. The difference between the final weight of the beaker and the initial weight gave the yield of essential oil.

### 3.5 HYDRODISTILLATION METHOD

130g of fresh lemongrass sample were placed into a 500ml round bottom flask containing 250ml of distilled water. The flask was fitted with a rubber stopper connected to a condenser and heated. Water at $0^0C$

flowed counter currently through the condenser to condense the ensuring steam. When the water reached 100$^{o}$C it started boiling ripping off the essential oil from the lemongrass. When the lemongrass got heated up, the essential oil that was extracted from the leaf mixed with the water vapour. Both passed through the condenser and the vapour was condensed into liquid. With the use of ice block, cooling was made possible and volatilization of the essential oil was avoided.

The condensate was directly collected using a 500ml beaker and then poured into a separating funnel. This formed two layers of oil and water. The tap of the separating funnel was opened to let out the water while the oil was immediately collected into a 100ml stoppered. The bottle was closed tightly to prevent vaporization of the essential oil. The oil was collected and the volume of oil obtained was weighed.

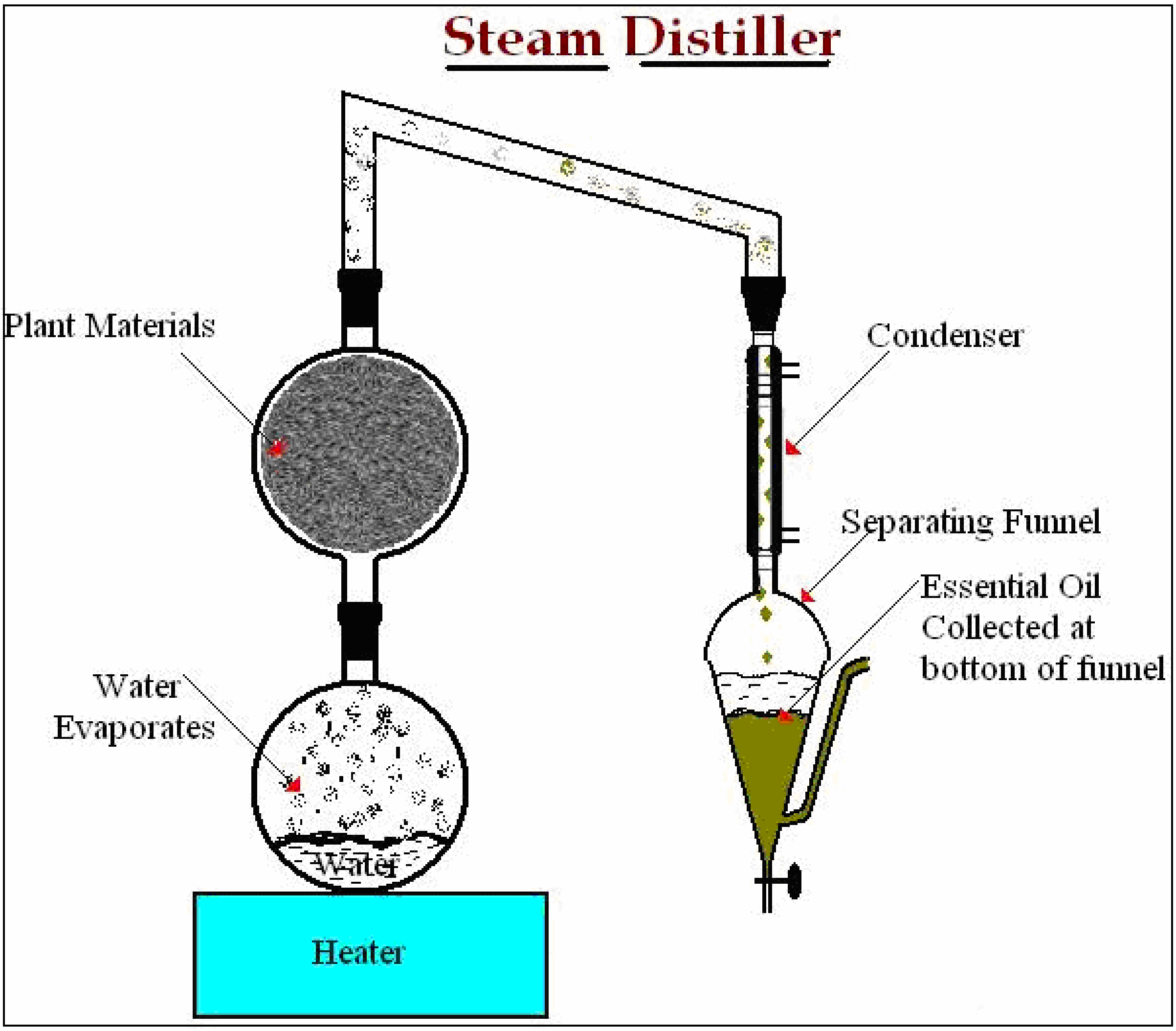



Figure 3.4: Extraction of essential oil using water distillation

## FORMULATION OF PERFUME WITH LEMON GRASS ESSENTIAL OIL PRODUCED

### Apparatus and reagents

- Pipette
- Funnel
- 50ml and 120ml beakers

- Perfume bottle
- Fixatives (Surprise and Dream)
- Methanol
- Distilled water
- Lemongrass essential oil

PROCEDURE

10ml of lemongrass essential oil extract were measured and placed in a 120ml beaker containing 5ml of Methanol. 5ml of the Fixatives were added to the mixture (to improve the longevity of the perfume). The solution were shaken and poured into a 50ml bottle.

# CHAPTER FOUR

## RESULTS AND DISCUSSION

### 4.1 SOLVENT EXTRACTION METHOD

Result obtained by solvent extraction is shown in Table 4.1 below

| Weight of oil (g) | Time (mins) |
|---|---|
| 0.2 | 240 |
| 0.3 | 480 |
| 0.7 | 720 |
| 0.72 | 960 |
| 0.78 | 1200 |

The amount of essential oils obtained by solvent extraction method was 2.7g of essential oil per 130g of dry lemongrass sample. This gave 2.08% yield of essential oil per 130g of dry lemongrass. The temperature used was $78^0$C i.e. the boiling point of ethanol. The volume of essential oil was measured at every 4hr interval to determine the oil yield at varying time. As the time increases the Ethanol solvent reduces thereby leaving the essential oil in the mixture.

## 4.2 ENFLEURAGE EXTRACTION METHOD

Table 4.2 weight of oil with respect to time

| Weight (g) | Time (mins) |
|---|---|
| 0.31 | 240 |
| 0.41 | 480 |
| 0.55 | 720 |
| 0.58 | 960 |
| 0.70 | 1200 |

The essential oil produced by enfleurage method is 2.55g weight of essential oil per 130g of dry lemongrass sample thereby producing 1.96% oil yield at $78^0$C.

## 4.3 HYDRO DISTILLATION METHOD

Table 4.3 weight of oil yield with respect to time

| Weight (g) | Time (mins) |
|---|---|
| 0.10 | 240 |
| 0.14 | 480 |
| 0.26 | 720 |
| 0.35 | 960 |
| 0.38 | 1200 |

The result of hydro distillation process was 1.23g per 130g of lemongrass sample giving 0.95% yield of oil.

**Physical and Chemical Properties of lemongrass oil**

The essential oil produced was pale yellow, with an aromatic camphoraceous odour, pungent and cooling taste. Because of its high volatility, it was stored in an air-tight container protected from light in cool place. The essential oil is insoluble in water, miscible in alcohol and in oil.

**Table 4.4 Result of Essential oil Extraction**

| Method of extraction | % yield |
|---|---|
| Solvent extraction | 2.08 |
| Enfleurage | 1.96 |
| Hydro distillation | 0.95 |

From the experiment carried out it was observed that the best method used in extraction is solvent extraction method because it yielded more oil than any other method. This conforms to works done by other researchers.

Enfleurage method, yielded less oil when compared to the solvent extraction this could be because most volatile content gets lost during the pounding process while hydro distillation gave low yield. This could be because the extraction of the essential oil was not always complete due to variable rate of distillation cause by heat.

## GENERAL OBSERVATION ON THE PREFUME PRODUCED

- The quantity of essential oil has the highest yield in solvent extraction
- The mixture is pale yellow.
- Has a clear lemongrass fragrance.
- It is volatile and has a cooling effect in the skin.

# CHAPTER FIVE

# CONCLUSION AND RECOMMENDATION

## 5.1 CONCLUSION

Solvent extraction, enfleurage and hydrodistillation methods are effective and efficient means of extracting essential oils. Solvent extraction is the most common and most economically technique for extracting oil in modern perfume industry because of its simplicity. Extraction by enfleurage was commonly used when distillation was not possible because some fragrant compounds denature through high heat. This technique is not commonly used in modern industries because of its prohitive cost. The essential oil extracted by hydrodistillation has strong odor characteristics of the raw material from which they were produced. When compared with other methods of extraction. It is cheaper because the cost involved is that of energy used in heating water to generate steam. Water itself is the commonest material easily available from nature supply or other alternative sources.

There is high demand for essential oils for various purposes such as medicinal, perfumery, soap making, insectides to mention but a few. Imported essential oils are very expensive to meet the demand of our local consumer industries, therefore it becomes necessary to source and synthesis these oils from local sources, in particular lemon grass.

With essential oils made from lemon grass, perfume can be produced locally using different methods of extraction, thereby creating employment.

## 5.2 RECOMMENDATIONS

There is urgent need for perfume production and from local raw materials in order to supplement the existing ones. I recommend more research to be carried out on extraction of essential oil and its formulation from vast variety of oil bearing plants in our ecosystem.

Further work should be carried out  to analyse the lemongrass essential oil as this could not be done due to time constraint.

Characterization of lemongrass essential oil components should be made in order to determine which is responsible for the characteristics of lemon grass odor.

Furthermore, large scale extraction of oil from lemongrass through enzymatic process should be explored, feasibility studies on the economic viability of the process should be conducted.

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# APPENDIX A

## CALCULATION OF PERCENTAGE YIELD OF ESSENTIAL OIL

FOR SOLVENT EXTRACTION METHOD

MATERIAL BALANCE

Weight of sliced lemongrass leave= 130g

Quantity of hexane used= 600ml

Quantity of Ethanol used= 200ml

Weight of beaker= 105.26g

Weight ethanol and essential oil= 202.7g

The weight of oil obtained= 2.7g

%yield = ME/ML x 100

Where ME = Mass of essential oil

ML = Mass of lemongrass sample

ME = 2.7g

ML = 130g

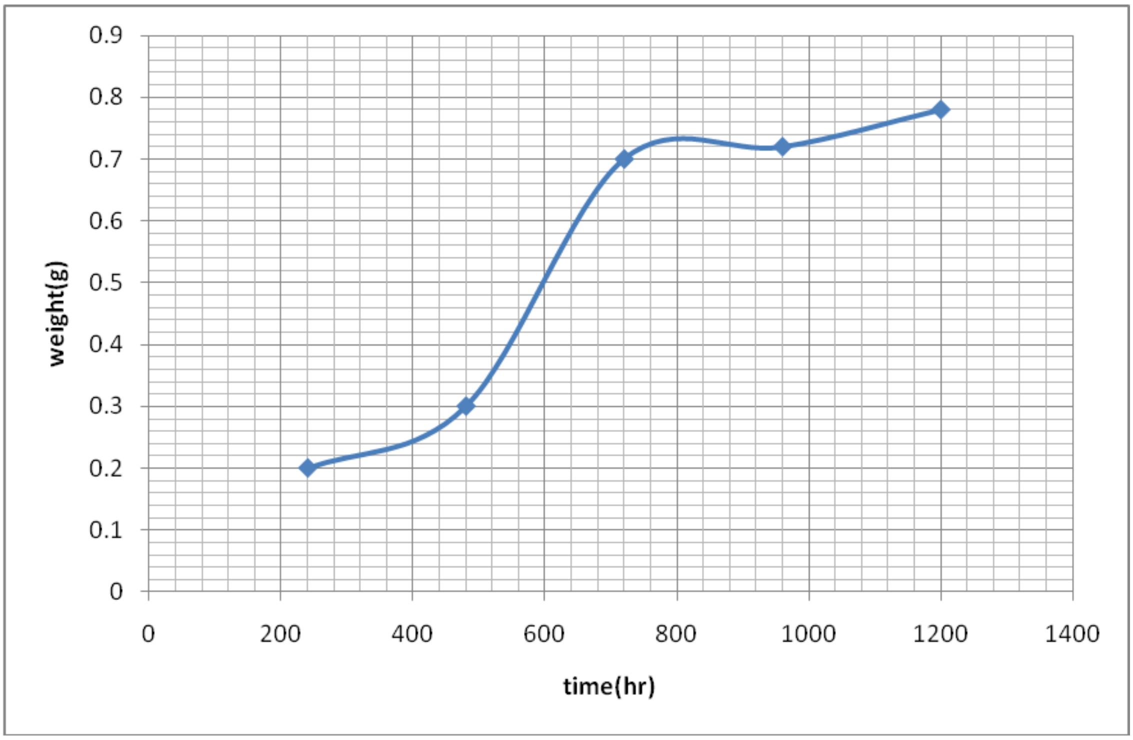
By substituting values

%yield = 2.7/130 x 100 =2.08%

Therefore % yield= 2.08%

The graph below shows the plot of the weight of essential oil with respect to time for solvent extraction method

**Graph of the weight (g) of essential oil to the time (mins)**



# APPENDIX B

MATERIAL BALANCE FOR ENFLEURAGE DISTILLATION METHOD

Weight of sliced lemongrass leave= 130g

Quantity of Olive oil used= 600ml

Quantity of Ethanol used= 140ml

Weight of beaker= 97.86g

Weight ethanol and essential oil= 100.41g

The total weight= 2.55g

%yield = ME/ML x 100

Where ME = Mass of essential oil

ML = Mass of lemongrass sample

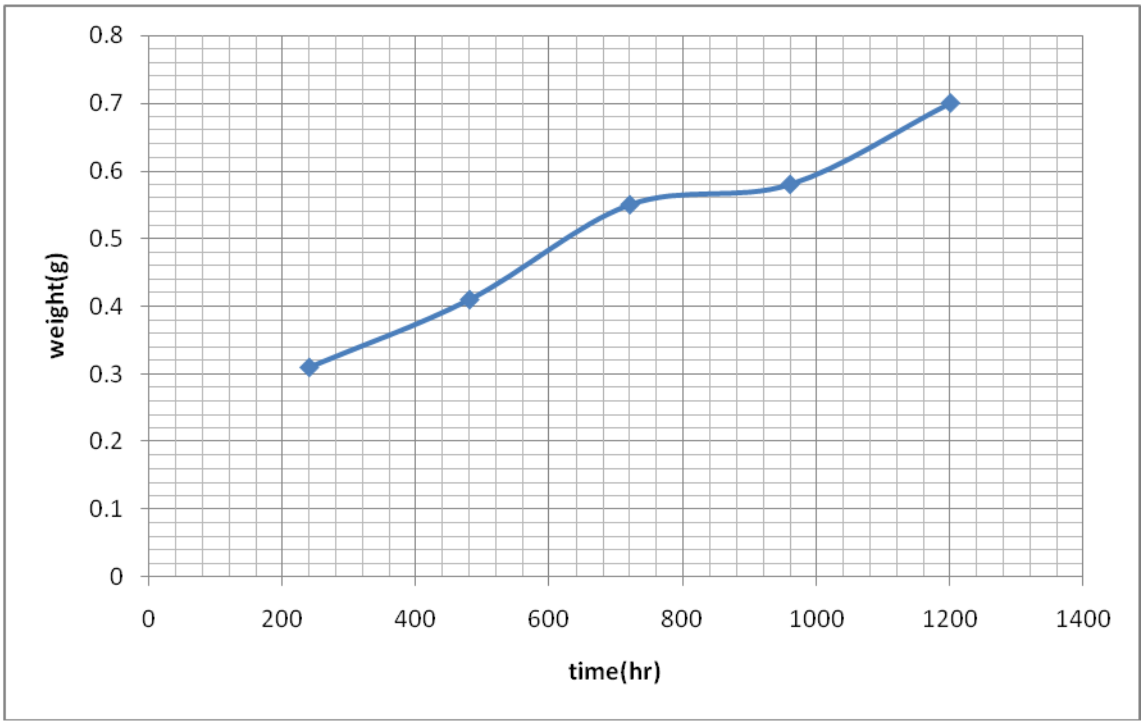
ME = 2.55g

ML = 130g

By substituting values

%yield = 2.55/130 x 100 = 1.96%

Therefore % yield= 1.96%




Graph of the weight (g) of essential oil to the time (mins) for enfluerage extraction method

# APPENDIX C

MATERIAL BALANCE FOR HYDRO DISTILLATION

Weight of sliced lemongrass leave= 130g

Quantity of Water used= 250g

Weight of beaker= 500g

The weight of oil obtained= 1.23 g

%yield = ME/ML x 100

Where ME = Mass of essential oil

ML = Mass of lemongrass sample

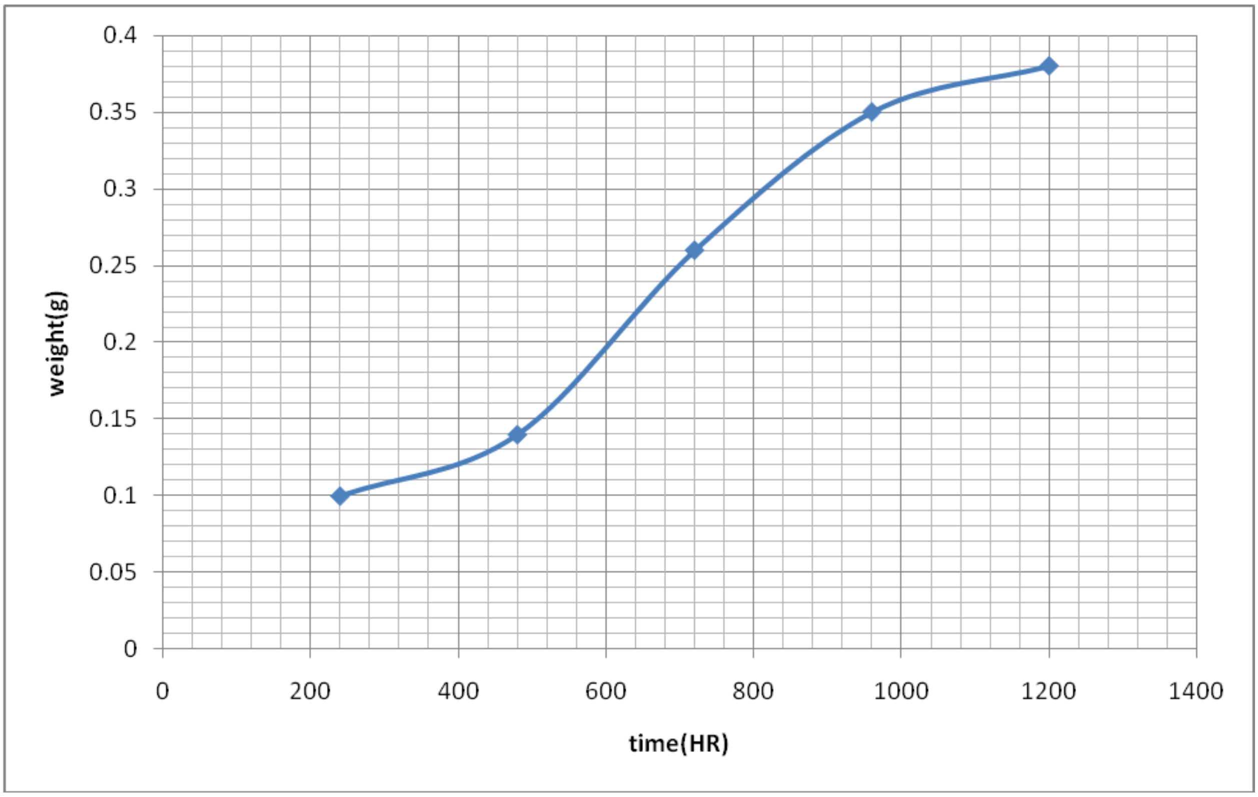
ME = 1.23g

ML = 130g

By substituting values

%yield = 1.23/130 x 100 = 0.95%

**Graph showing the weight (g) of essential oil to the time (mins) for Hydro distillation method**


weight(g)
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
0
200
400
600
800
1000
1200
1400
time(HR)