THE EXTRACTION OF ESSENTIAL OIL FROM RED PEPPER (Capsicum annum); FORMULATION AND PRODUCTION OF PERFUME FROM THE EXTRACT

Tolulope, F.A. Olubanwo, Moses O. Ekeoma, Blessing Onuora, Stephena E. Tolubanwo.

CHAPTER 1

1. 1 INTRODUCTION

The word perfume is used today to describe scented mixtures and is derived from the Latin word, "per fumus," 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. In fact the natives from different tropical regions of the globe have long been extracting oil from numerous oil bearing plants. Human beings 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

Egyptians were responsible for the origin of perfume. They utilized scents in everything from religious ceremonies to burial preparations and even daily wear. The rich elites of Egyptian society, male and female alike would adorn themselves with aromas like lily to denote their status. The Persians took over the use of perfume as a sign of political status, but it wasn't until the Greeks and Romans became acquainted

with it that it began to be viewed as a form of art and produced in large quantity and in consistent quality. Archaeologists recently uncovered a perfume factory from 2,000 BC, located in Cyprus, which seemed to have specialized in the production of scents like coriander, laurel, myrtle, lavender, and rosemary. Perfume slowly spread throughout the globe, and for a while, scents were reserved mainly for use in religious ceremonies. However, in 1190, perfume began to be produced commercially in Paris, and from there, it blossomed into a massive industry once more (https://www.perfume.com/article-history-of-perfume). Ancient texts and archaeological excavations show the use of perfumes in some of the earliest human civilizations. Modern perfumery began in the late 19th century with the commercial synthesis of aroma compounds such as vanillin or coumarin that allowed for the composition of perfumes with smells previously unattainable solely from natural aromatics alone (Levey, 1973).

Several thousands of plants distributed throughout the world contain a group of odiferous, fragrance, oily products that are highly volatile organic substances collectively known as essential oils. The term essential used here does not mean indispensable as with the term essential amino acids or essential fatty acids which are so called since they are nutritionally required by a living organism (Reeds, 2000). 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 Egyptians extracted essential oils from plants tissues by steam distillation (Eke et al 2005). Other methods of isolating essential oils include solvent extraction, expression, cold

plate or enfleurage. Some of these methods have been adopted by essential oil extracting industries.

Information on perfume indicates the type of essential oils that have been extracted from plants and is of different quality in terms of fragrance, for example jasmine perfume is from jasmine plants, and lemon perfume is from lemon grass and so on.

A formulation or recipe for perfume that is rightly blended with the essential oil and other additives is used to differentiate one perfume from the other.

Alcohol is added to the fragrance or essential oils as a primary solvent to reduce the strays of the oils and as the vehicle through which the perfume can come out as smoke. Essential oil can be used directly as perfume by robbing the oil on the skin, cloth or any other materials. It is usually stored in a bottle different from spraying bottle where it can be applied without been sprayed because of its viscosity. Alcohol is therefore the perfect liquid through which essential oil can be sprayed as perfume.

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 fragrance of perfume will last longer on the user when the right fixatives such as Polysorbate is used in its formulation.

1.3 Statement of the Problem

Recently, there has been a high demand of essential oils for various purposes such as medicinal, perfumery, soap making, insecticides to mention but a few products been produced from essential oil which has opened up wide opportunities even for small scale businesses.

Imported synthetic essential oils are very expensive to import whit high cost of importation to meet the demand of our local consumption more especially the small and medium enterprises. Therefore it becomes necessary to source and extract these oils from local sources. In particular, good perfumes that are usually

imported can be produced locally from a vast variety of oil bearing plants yet to be explored. Solvent extraction is the safest method for extracting high yield oil because some herbs and spices cannot be extracted by other methods, but it has the disadvantage of having residual solvents in the essential oils.

The research is conducted in order to use hydro-distillation as one of the applicable methods to extract essential oils. Hydro-distillation has the advantage of no solvent residues as an alternative to conventional extraction techniques.

1.4 Justification of the Study

It is hoped that from this research work, optimum extraction parameters like solvent type, percent extraction, 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. Interestingly, some of these source of essential oils are common in Nigeria such as lemon grass, ginger, flowers, eucalyptus and in addition red pepper (*Capsicum annum*), to mention but a few.

There is a high potential in this area and it becomes necessary to seek means to explore 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 red pepper (*Capsicum annum*) using three extracting methods.

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; Soxhlet solvent extraction by hot method, Solvent extraction by cold method and Hydro-distillation.

ii. To formulate perfume using appropriate materials.

iii. The composition and production of perfume and its concentration.

CHAPTER 2
2.0 LITERATURE REVIEW

2.1 The History of Perfumery

The word perfume is used today to describe scented mixtures and is derived from the Latin word, "perfumare", meaning ‘to smoke through’. The word Perfumery refers to the art of making perfumes. Perfume was discovered by ancient Mesopotamia and Egypt. It was however further refined by the Romans, the Persians (present Iraq) and the Arabs. Although perfume and perfumery also existed in East Asia, much of its fragrances are incense based. The basic ingredients and methods of making perfumes are described by Pliny the Elder, 1855 in his Naturalis Historia. 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 odour 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. Due to differences in body chemistry, temperature, and body odours, no perfume will smell exactly the same on any two people.

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

odourants 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, 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 that 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 favoured. 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 flavour 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 (Glossary, 2012). 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:

1. Perfume extract, or simply perfume (Extract): 15-40% aromatic compounds.
2. Esprit de Parfum (EsdP): 15-30% aromatic compounds, a seldom used strength concentration in between EdP and perfume.
3. Eau de Parfum (EdP), Parfum de Toilette (PdT): 10-20% (typical 15%) aromatic compounds.
4. Eau de toilette (EdT): 5-15% (typical 10%) aromatic compounds.

5. Eau de Cologne (EdC): Chypre citrus type perfumes with 3-8% (Typical 5%) aromatic compounds.
6. Perfume mist: 3-8% aromatic compounds (typical non-alcohol solvent).
7. Splash (EdS) and Aftershave: 1-3% aromatic compounds (Larson et al, 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.

1. Top notes: Also called the head 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. Examples of top notes include mint, lavender and coriander.
2. Middle notes: Also referred to as heart notes. The scent of a perfume that emerges just prior to the dissipation of the top note. 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. Examples of middle notes include seawater, sandalwood and jasmine.
3. 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. Examples of base notes include tobacco, amber and musk.

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. Manufacturers of perfumes usually publish perfume notes and typically they present it as fragrance pyramid, with the components listed in imaginative and abstract terms.

2.4 Sources of Perfumes

2.4.1 Aromatics sources

1. **Plant sources**

Plants have long been used in perfumery as a source of essential oils and aromatic 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 odours 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.

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, Perus balsam, 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, and 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.

2. **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 animatic fragrance agents around which little controversy now exists.

Castoreum: Obtained from the odourous 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 Viverridae mongoose. 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. 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".

3. **Other natural sources**

Lichens: Commonly used lichens include oak moss and tree moss 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

Aromatic compound

Many modern perfumes contain synthesized odourants. 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 terpenes 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 is 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. They are fragrant materials that have been extracted from a source material directly through distillation or expression and obtained in the form of an oily liquid. Oils

extracted through expression are sometimes called expression oils. 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 essential oil contributes to the overall character.

2.5.1 Sources of Essential Oil

An essential oil is a concentrated hydrophobic liquid containing volatile aroma compounds from plants. Essential oils 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. Oil is "essential" in the sense that it contains the "essence of" the plant's fragrance, the characteristic fragrance of the plant from which it is derived. The term essential used here does not mean indispensable as with the terms essential amino acid or essential fatty acid which are so called since they are nutritionally required by a given living organism (Reeds, 2000).

Dalila (2016) defined essential oils as oil which is concentrated complex mixtures of many natural components that contain volatile aromatic compounds extracts from fragrant plants. It can be extracted from the seeds, flowers, leaves, wood bark, stems, fruits, rhizomes and roots, from a wide variety of plant shrub and tree species.

Essential oils are an important source of raw material to the pharmaceutical, cosmetic and food industries.

Essential oils are generally extracted by distillation, often by using steam. Other processes include expression, solvent extraction, absolute oil extraction, resin tapping, and cold pressing. They are used in perfumes, cosmetics, soaps and other products, for flavouring food and drink, and for adding scents to incense and household cleaning products.

Essential oils can be obtained from any of the following sources.

2.5.2 Plant Source:

2.5.3 Lemon Grass

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. 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 odour. Some other constituents of the essential oils are terpineol, myrcene, citronella, 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 slightly lower citral content.

Lemongrass is used in herbal teas and other non-alcoholic 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 odours 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 martini* is reportedly toxic to fungi (Moore, 2006).

2.5.4 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.

2.5.5 Jasmine

A highly esteemed perfume, jasmine is cultivated in Southern France and surrounding areas. The main source is *Jasminum officinarum* 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.

2.5.6 Violet

One of the most popular perfumes is made from violets. Blue and purple double varieties of Viola odourata, 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.

2.5.7 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 terminals 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).

2.5.8 Otto of Roses

This is valuable oil that is also called **Attar of Roses**. It has been one of the most favourite perfumes either in combination with other oils or alone. Bulgaria supplied most of 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 colourless at first but gradually turns to a yellowish or greenish color.

2.5.9 Rosemary

Rosemary, (*Rosmarinus officinalis*), is a native of the Mediterranean region. It has long been a favoured sweet-scented plant and has been important in the folklore of many countries. It is one of the least expensive and most refreshing odours. 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.

2.6.0 Red Pepper (Capsicum annum):

Red Pepper (*Capsicum annuum* L.) is one of the most significant vegetable plants in Western Balkans. Production of spice paprika in the region of Pannonian plate has a long tradition. One of the very important products of pepper processing is ground paprika, a spice which is widely used throughout the World. The main quality parameter for ground paprika products are the colour and pungency (Carvajal et al., 1998; Govindarijan, 1986; Govindarijan et al., 1987), while aroma recently started to attract the attention of scientists (Mateo et al., 1997; Eichner, 1973; Kocsis et al., 2002; 2003).

In the process of food production red pepper is mainly used as a colourant. Unique red colour of red pepper is due to presence of capsantin and capsorubin, two carotenoids characteristic for the capsicum genus. Besides changing the colour of the food, red pepper also affects the flavour.

2.6.1 Chemical Constituents of Essential Oils.

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

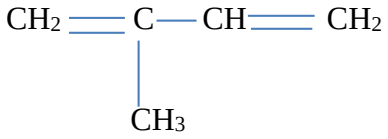
Volatile fraction: Essential oil constituting of 90–95% of the oil in weight, containing the monoterpene and sesquiterpene hydrocarbons, as well as their oxygenated derivatives along with aliphatic aldehydes, alcohols, and esters.

Non-volatile residue: that comprises 1-10% of the oil, containing hydrocarbon, fatty acids, carotenoids, sterols, waxes and flavonoids.

2.6.2 Chemical Structure of Essential Oil

2.6.3. Hydrocarbon:

Essential Oils consist of chemical compounds that have hydrogen and carbon as their building blocks. Basic hydrocarbon found in plant is isoprene having the following structure.



(Isoprene)

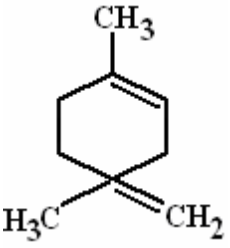
2.6.4. Terpenes:

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.

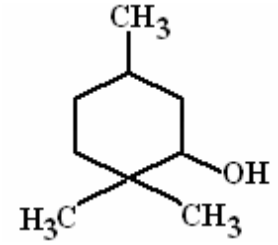
i. Monoterpenes ($C_{10}H_{10}$)

Properties: Analgesic, Bactericidal, Expectorant and Stimulant.

Monoterpenes are naturally occurring compounds, the majority being unsaturated hydrocarbons (C_{10}). But some of their derivatives such as alcohols, ketones and carboxylic acids known as mono-terpenoids.



(Limonene)



(Menthol)

Hydrocarbons comprises of two isoprene units.

The branched-chain C_{10} and is widely distributed in nature with more than 400 naturally occurring monoterpenes identified.

ii. Sesquiterpenes

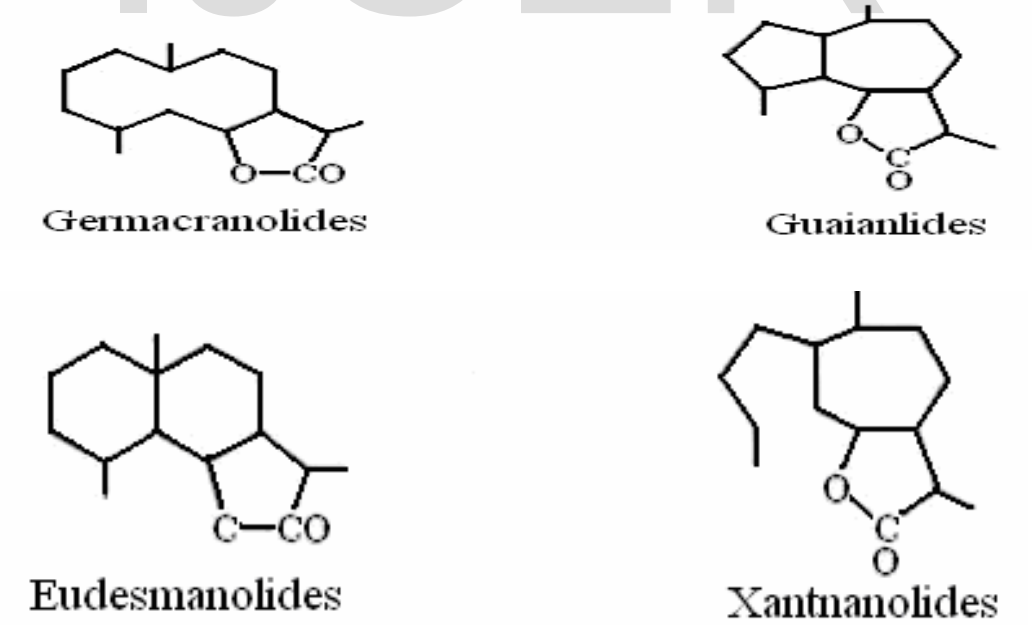
Properties: Anti-inflammatory, anti-septic, analgesic, anti-allergic. Sesquiterpenes are

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



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 C20 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).

2.6.5. 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.

2.6.6. 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.

2.6.7. 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.

2.6.8. 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.

2.6.9. Ketones and Lactones

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.

Lactone properties include anti-inflammatory, anti-phlogistic, 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.

2.7.0. Conditions that can affect the yield and quality of Essential Oils.

Currently, the conventional methods for extraction of essential oil that are widely used by the industry are steam distillation, hydro-distillation and solvent extraction. These methods have some weaknesses which are highly affecting the yield and quality of the essential oil. For steam distillation and hydro-distillation, the high temperature used can cause chemical modification of the essential oil component especially to the heat sensitive volatile compound (Castro, 1999). Furthermore, the use of solvent extraction is not an effective way due to the present of solvent residue in the product and also resulting in the loss of the highly volatile component of the essential oil (Rezzoug, 2005). This includes:

1. 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.
2. Improper design of equipment: Improper designing of tank, condenser or separators can lead to loss of oils and high capital investments.
3. Material of Construction of equipment: Essential oils which are corrosive in nature should be preferably distilled in stills made of resistant materials like aluminum, copper or stainless steel.
4. 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.

5. Filling of raw material / steam distribution: Improper loading of the herb may result in steam channeling causing incomplete distillation.
6. 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 this 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/equipment: Tanks should be well steamed for multiple crops and distillation tank/equipment should not be rusted for quality oil.

2.7.1 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 (Sundeep, 2007).

2.7.2 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

Container Material	**Remarks**
G.I.(White sheet)	For All oils
Aluminium	For All/ High Value Oils
Iron/Mild Steels	For some oils. Not recommended for long storage
Stainless steel	For All high values oils
GI/MS with glass/stainless steel lining	For high value oils
Plastic HDPE	For all oils for less time only
Glass Bottles	For All oils lesser quantity only

2.8 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 process denature the aromatic compounds, either changing their odour character or rendering them odourless.

2.8.1 Extraction of Fragrance

It is known that before perfumes can be composed, the odourants used in various perfume compositions must first be obtained. Synthetic odourants are produced through organic synthesis and purified. Odourants 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 (Camps, 2000). All these techniques will, to a certain extent, distort the odour 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 from 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 odourants 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" (Sandeep, 2008).

Supercritical fluid extraction: A relatively new technique for extracting fragrant compounds from a raw material, which often employs Supercritical CO_2. Due to the low heat of process and the relatively non-reactive solvent use in the extraction, the fragrant compounds derived often closely resemble the original odour 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 can also be extracted into the ethanol.

2.8.2 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 simple terms distillation of aromatic herbs 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 (Sandeep, 2007).

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. The 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 (Sandeep, 2007).

Disadvantages of the Hydro Distillation

The process is slow and the distillation time is much longer thereby consuming more firewood / fuel making process uneconomical and 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 and alcohols which are not suitable for large capacity/commercial scale distillations and also not suitable for high boiling hardy roots/woody plant materials.

2.9 **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 they can be used as a major component of the primary scent. Common blending ingredients include linalool and hydroxyl citronellal.

Fixatives: Fixatives are used with the other ingredients in order to lower the rate of evaporation of the fragrance or essential oils. The reason why a perfume may lose its fragrance faster than normal is because only a little amount of fixative was used when preparing the perfume. 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.

2.10 The importance of Perfume

There are many reasons why everyone should be advised to use perfume on daily basis. The use of perfumes promotes good healthy living and undoubtedly makes one smell good.

As I said earlier that aldehyde and alcohol, some of the essential substances found in perfume have anti-fungal, anti-inflammatory, anti-septic, anti-viral, bactericidal, disinfectant, and sedative properties. Medicinally, essential oils contain aldehydes which 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.

Christian Nordqvist, 2016 stated in his publication “Body odour (or B.O., bromhidrosis, osmidrosis or ozochrotia)” is a perceived unpleasant smell our bodies can give off when bacteria that live on the skin break down sweat into acids; some say it is the smell of bacteria growing on the body, but it really is the result of bacteria breaking down protein into certain acids.

Body odour usually becomes evident if measures are not taken when a human reaches puberty; 14-16 years of age in females and 15-17 years of age in males. People who are obese, those who regularly eat spicy foods, as well as individuals

with certain medical conditions, such as diabetes, are more susceptible to having body odour.

People who sweat too much, those with hyperhidrosis may also be susceptible to body odour. However, often the salt level of their sweat is too high for the bacteria to break down, it depends where the excess sweating is occurring and which type of sweat glands are involved.

Sweat itself is virtually odourless to humans; it is the rapid multiplication of bacteria in the presence of sweat and what they do (break sweat down into acids) that eventually causes the unpleasant smell. The smell is perceived as unpleasant, many believe, because most of us have been brought up to dislike it. Body odour is most likely to occur in our feet, groin, armpits, genitals, pubic hair and other hair, belly button, anus, behind the ears, and to some (lesser) extent on the rest of our skin.

Body odour can have a nice and specific smell to the individual, and can be used especially by dogs and other animals to identify people. Each person's unique body odour can be influenced by diet, gender, health, and medication.

CHAPTER 3

3.0 MATERIAL AND METHODOLOGY

3.1 Sample Preparation

Fresh Red Pepper Capsicum anuum which is called 'tatase' in Nigeria language and was identified by Dr. Garuba Musun of Plant Science and Biotechnology, College of Natural Science, Michael Okpara University of Agriculture, Umudike (MOUAU). The sample was collected from Ubani Main market, Umuahia in Abia State. However, the original source of farm was from Jos, Plateau State, Nigeria. Part of the sample was treated fresh while the other sample was allowed to dry for about three days under the sun.

Plate 3.1 Red Pepper, Capsicum anuum (Tatase).

3.2 Apparatus and Reagents

A retort stand

500ml, 250ml and 100ml Beakers

500ml Separating funnel

Electronic weighing scale

Water bath

Aluminum foil

Electric Grinding Machine

Distilled water

Soxhlet Extractor

Ethyl Alcohol

n-Hexane

Polysorbate

3.3 Extraction Methods

Three different methods were employed to extract the essential oil from the red pepper sample. These methods include

1. Soxhlet Extraction using normal hexane
2. Solvent Extraction by cold method
3. Hydro-distillation method.

3.3.1 Soxhlet Extraction using n-hexane:

10g of dried pepper (Capsicum annum) was crushed and placed in a Soxhlet extraction apparatus with 150 ml hexane.

Precaution: At least 2/3 of the volume of solvent should be used of the capacity of the round bottom flask, this is to prevent the solvent from drying up before extraction is achieved.

The solution was gently refluxed for 8 hours and allowed to cool to room temperature. The process was repeated three times and the average was taken.

Plate 3.2 Soxhlet Extraction Apparatus.

The extracted solution was mixed 100ml of ethyl alcohol for further separation of solvents. The mixture was then taken to a separating funnel of 250ml capacity. The content was allowed to stay for 48 hours for liquid-liquid separation (partition) to take place. The liquid from separating funnel containing the mixture of ethyl alcohol and essential oil was then taken to a water bath where the temperature was adjusted to 78^{o}C. The remaining oily liquid in the flask was the essential oil was weighed and the amount of extraction determined

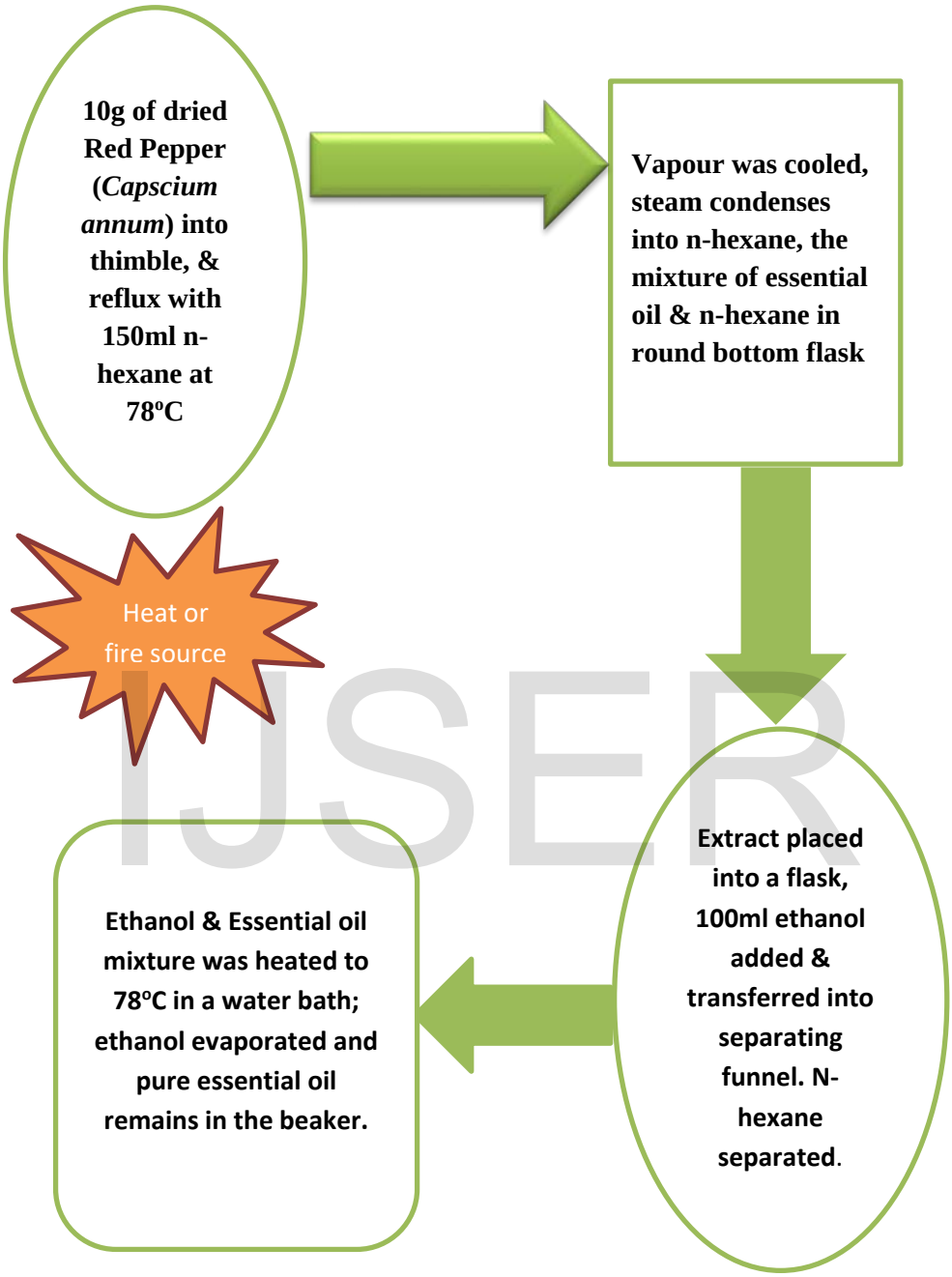


Figure 3.1 Flow Diagram for Soxhlet Solvent Extraction of Essential oil by hot method

Plate 3.3 Separating funnel apparatus.

3.3.2 Solvent Extraction by cold method.

Dried pepper was blended in an electric grinding machine so as to increase the surface area of the sample. 10g of the pepper was placed in a 500ml beaker. 150ml of n-hexane solvent was added and covered by aluminum foil. The flask and content was allowed to stand for 8hrs; this was done to extract all the oil content in the red pepper and for complete extraction. After which the extract was decanted into another 500ml beaker. 100ml of Ethanol was 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 (because of their different density). The lower Ethanol extract and the upper n-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. The process was repeated three times.

3.3.3 Hydro-distillation Method

10g of dried sample of red pepper was placed into a thimble and placed in the extractor unit of Soxhlet extractor and the 250ml round bottom flask with distilled water. The temperature was adjusted to 100^{O}C. When the water reached 100^{O}C it started boiling ripping off the essential oil from the red pepper. When the red pepper got heated up, the essential oil that was extracted from the sample mixed with the water vapour. Both passed through the condenser and the vapour was condensed into liquid. The condensate was collected in the round bottom flask containing water and oil. This formed two layers of oil and water and it was transferred to a separating funnel. The tap of the separating funnel was opened to let out the water while the oil was immediately collected into a 100ml beaker. The beaker was closed tightly with aluminum foil to prevent vaporization of the essential oil. The oil was collected and the volume of oil obtained was weighed.

3.4 Perfume Production.

Formulation of perfume from the essential oil produced from red pepper (*Capsicum annum*).

Apparatus/Reagents:

1. Beakers, 50ml
2. Pipette.

3. Measuring cylinder.
4. Ethanol.
5. Polysorbate 20.
6. Water.
7. Essential oil.
8. Bottle.

Procedure:

For every perfume produce, the formulation of the ingredients must make up to 100% of the content in order to give actual value of its quality.

For the purpose of this project, a 20 ml perfume was produced according the formulation.

Table 3.1 Basic perfume formulation

Ingredients	%	ml
Ethanol	68	13.6
Essential Oil	20	4.0
Water	10	2.0
Polysorbate 20	2	0.4
Total	100	20

1. 13.6ml of ethanol was measured into a 50ml beaker.
2. 2.0ml of water was measured and added to the ethanol in the beakers.
3. This was followed by the addition of 0.4ml of Polysorbates 20 into the mixture.
4. The mixture was thoroughly mixed together for homogeneity.
5. Finally the 4.0ml essential or fragrance oil was added to the mixture and stirred.
6. The resultant perfume of 20 ml was put inside a bottle and covered.

3.5 p^H Determination

The p^H for the essential oil as well as the perfume produced from the extraction was determined. The p^H was first calibrated by inserting the electrode of the p^H meter into a buffer solution of p^H 4.5. It was rinsed with distilled water, wiped with soft tissue and thereafter inserted into another buffer solution of p^H 7.1.

The p^H meter was inserted into the extracted oil and the p^H taken.

The process was repeated for the perfume produced from the extracted oil.

Plate 3.4 Apparatus showing p^H meter calibration

3.6 Specific Gravity Determination

The specific gravity for both the extracted oil and the formulated was determined. The specific gravity of water is 1. This means 1gm of water weighed will be equivalent to 1ml of water if measured in volume.

1ml of the essential oil was weighed and the weight was taken.

The same procedure was used for the perfume, as 10ml of perfume was weighed on

an analytical scale and the weight was taken.

Plate 3.5 the determination of specific gravity by weighing.

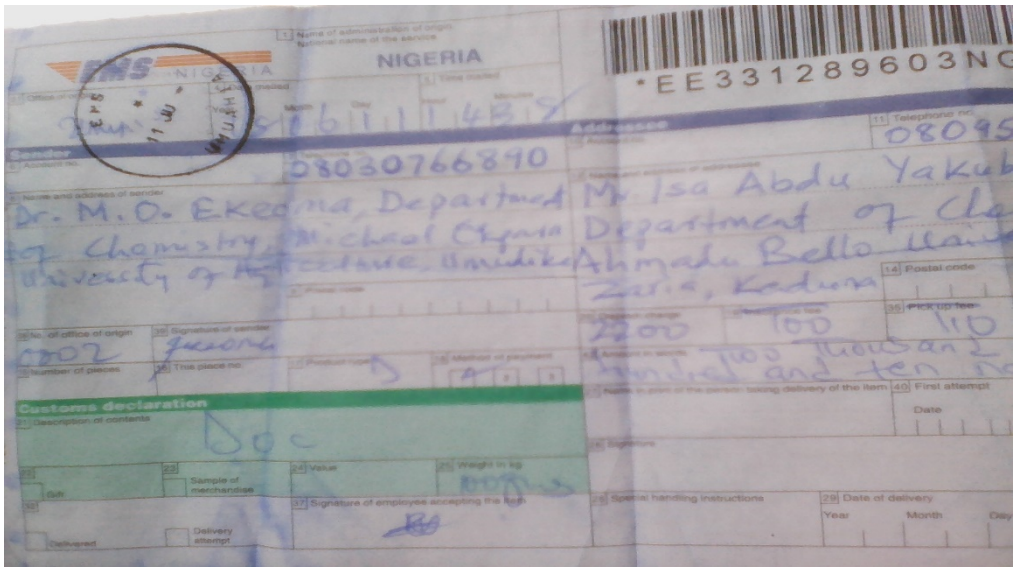


Plate 3.6 GC-MS Analysis: Receipt of courier services to Ahmadu Bello University (ABU), Zaria.

CHAPTER 4

4.0 Result and Discussion

The result obtained by Soxhlet-solvent extraction method is shown below.

4.1 Soxhlet Extraction using n-hexane Method.

Table 4.1 Weight of extract using hot method

Weight of samples (g)	1st (g)	2nd (g)	3rd (g)	Total (g)
Initial Weight of sample	10.00	10.00	10.00	30.00
Final Weight of sample	9.77	9.65	9.71	29.13
Weight of extract	0.23	0.35	0.29	0.87

The amount of essential oil obtained from 30g of pepper sample by 450ml of n-hexane, using Soxhlet extraction apparatus was 0.87g in 8 hours at 70^{O}C. This is equivalent to 2.9% yield of essential oil. The mixture of n-Hexane and ethanol was later separated by heating at 78^{O}C on a water bath for 3 hours 30 mins.

4.2 Solvent Extraction by cold method.

Table 4.2 Weight of extract using cold method

Weight of sample (g)	1st (g)	2nd (g)	3rd (g)	Total (g)
Initial Weight of sample	10	10	10	30
Final Weight of sample	9.86	9.87	9.83	29.56
Weight of extract	0.14	0.13	0.17	0.44

The amount of essential oil extracted was 0.44g from 30g of pepper sample by adding a total of 450ml of n-hexane in 8 hours. This gave 1.47% yield of essential oil.

4.3 Hydro-distillation Method

The result obtained by Soxhlet-solvent extraction is shown below.

Table 4.3 Weight of extract hydro-distillation method

Weight of samples	1st (g)	2nd (g)	3rd (g)	Total (g)
Initial Weight of sample	10.00	10.00	10.00	30.00
Final Weight of sample	9.86	9.81	9.84	29.51
Weight of extract	0.14	0.19	0.16	0.49

The amount of essential oil obtained by hydro-distillation method using Soxhlet extraction apparatus was 0.49g from 30g of sample for 8 hours at 100^{O}C. This gives 1.6% yield of essential oil. The mixture of oil and water was separated by decantation at room temperature. The oil with lower density was on top of the mixture while water was below.

4.4 Physio-chemical properties of essential oil extract of *Capsicum annum* (red pepper).

Physical Properties

The following was the properties of the essential oil extracted from *Capsicum annum* red pepper oil.

Colour: Red to amber moon colour

p^{H} : 5.9 @ 25^{O}C

Specific Gravity (SG): 0.860

Solubility: It is insoluble in water, soluble and miscible with oil and ethanol.

Odour: It has classic and pungent odour characteristic of aromatic compound.

Chemical Properties:

GC-MS analysis was paid for and the sample sent to Ahmadu Bello University

Zaria for analysis but we were later told that the machine was faulty. We lost both the money and the sample**.**

Table 4.4 Results from methods of essential oil extraction

Method of Extraction	% yield
Solvent Hot Method	2.9
Solvent Cold Method	1.44
Hydro-distillation method	1.6%

From the experiment carried out it was observed that the best method used in extraction was Soxhlet solvent extraction hot method because it yielded more oil than any other method. The cold method yielded the lowest.

4.5 Physio-chemical properties of Perfume.

Physical Properties

The perfume produced from the extracted essential oil has the following properties:

Colour: Bright/light red colour

pH: 7.9 @ 25^0C

Specific Gravity (SG): 0.72

Odour: Classic smell of musk fragrance.

Appearance: Clear transparent light red colour liquid.

It is volatile and has cooling effect with long lasting fragrance.

Solubility: It is soluble in ethanol and sparingly soluble in water.

Table 4.5 Summary of extractive methods

Solvent Hot Method (10g)			Solvent cold Method (10g)			Hydro distillation (10g)	
weight	%		weight	%		weight	%
0.23	2.3		0.14	1.4		0.14	1.4
0.29	2.9		0.13	1.3		0.19	1.9
0.35	3.5		0.17	1.7		0.16	1.6

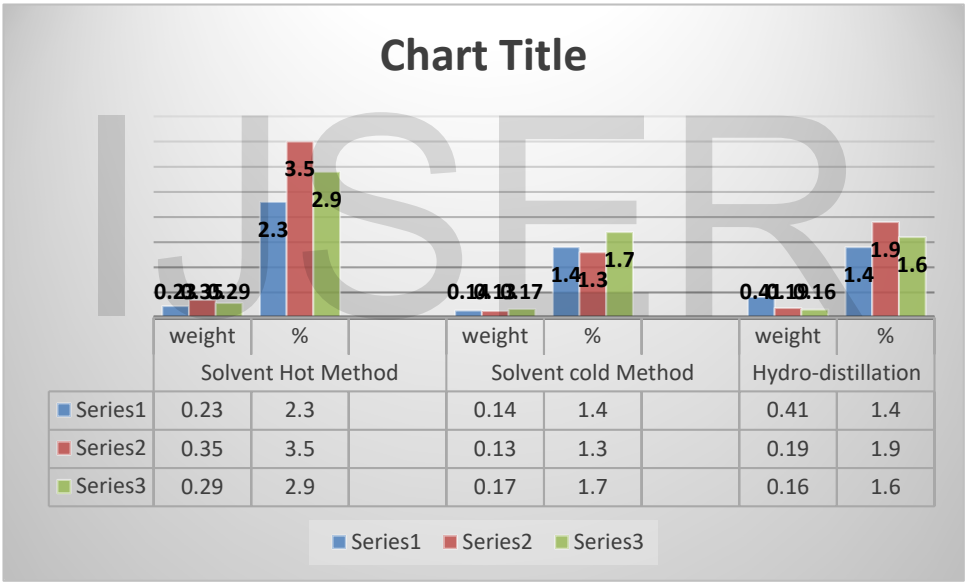


Figure 4.1 bar chat of weight to % extraction of extractive methods.

CHAPTER 5.

5.0 CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Solvent extraction by hot method, cold method as well as hydro-distillation methods are effective and efficient means of extracting essential oils. The Soxhlet solvent extraction is the most effective of all methods for extracting oil in modern perfume industry because of its high yield (2.9% yield) simplicity and continuous production process. The essential oil extracted by hydro-distillation has strong odour characteristics of the raw material from which they were produced. It is devoid of any interference of hexane odour, 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.

5.2 RECOMMENDATIONS

From this work, it is observed that Capsicum annum is an efficient source of essential oil for perfume's production. Hence, we recommend that the Government and Universities should encourage young graduates to invest in large scale production and processing of Capsicum Annum for the purpose of commercial production of perfume to keep the youth off the street.

REFERNCES

1. Aneta Wesołowska, Dorota Jadczak, Monika Grzeszczuk (2011): Chemical Composition of The Pepper Fruit Extracts of Hot Cultivars *Capsicum annuum* L. *Acta Sci. Pol., Hortorum Cultus 10(1) 2011, 171-184,* West Pomeranian University of Technology, Szczecin.
2. Antmon, D.G., Barton, A.F.M. and Clarke, D.A., (1986). *Essential Oils Introduction and Evolution.* Ibadan: AF publishers.
3. Arthur, I.V. (1975) *Elemental Practice Organic Small Scale Production Part 1*.London, Longman publishers.
4. Camps, Arcadi Boix (2000). "Perfumery Techniques in Evolution". *Allured Pub Corp*. ISBN 0-931710-72-3
5. Carvajal, M., Gimenez, J.L., Riquelme, F., Alcaraz, C.F. (1998). Antioxidant content and colour level in different varieties of red pepper (Capsicum annuum L.) affected by plant-leaf Ti4+ spray and processing. *Acta Alimentaria*, *27*, 365-375.
6. Castro, M. D, Carmona, M. M, and Perez, V. (1999): "Towards more rational techniques for the isolation of valuable essential oils from plants," *Trend in Analytical Chemistry*, vol. 8, no. 11, pp. 708-716, 1999.
7. Clark E. and Grande I., (1975): Study of Odour Variation with Structural Change in Cosmetic Perfume. Florida: CRC Press. Coulson.
8. Christian Nordqvist, 2016; Body Odor: Causes, Prevention, Treatments, Medical News Today Newsletter, Feb. 15, 2016, Health Media UK Ltd.
9. Cordell GA, Araujo OE. (1993): Capsaicin: Identification, Nomenclature, and Pharmacotherapy; US National Library of Medicine, National Institute of Health.
10. Dalila Nasshorudin, Muhammad Syarhabil Ahmad, Awang Soh Mamat, (2016):

Novel Closed System Extraction of Essential Oil: A Green Approach, American Journal of Biochemistry 2016, 6(6): 145-148, DOI: 10.5923/j.ajb.20160606.02

11. Dorland E., and Rogers M., (1977): *The Fragrance and Flavour Industry.* Volume 5.London: Longman publishers.
12. Debboun, Mustapha; Frances, Stephen P.; Strickman, Daniel, eds. (2014). Insect Repellents Handbook (2nd Ed.). CRC Press. p. 362. ISBN 1466553553).
13. Eke, E., Ogbu J., and Okoro, K. (2005): *Isolation of Essential oils from Plants Volume* 7. India: Elsevier Publishers.
14. Eichner, K. (1973). Indikatoren für beginnende qualitative Veränderungen von Lebensmitteln. *Deutsche Lebensmittel-rundschau, 69,* 39-47.
15. Free Mantle M.H, Onyinbo, L. and Tidy S.G. (1992) *Essential science Chemistry,* Ibadan: University press.
16. "Glossary (C)". The Fragrance Foundation. Archived from the original on 31 July 2010. Retrieved 7 November 2012.
17. Govindarajan V.S. (1986). Capsicum-production, technology, chemistry, and quality. Part III. Chemistry of the color, aroma, and pun-gency stimuli. *Critical Reviews in Food Science and Nutrition*, *24,* 245-355.
18. Govindarajan, V. S., Rajalakshmi, D., Chand, N. (1987). Capsium - production, technology, chemistry, and quality. Part V. Impact on physiology, pharmacology, nutrition, and me-tabolism; structure, pungency, pain, and de-sensitization sequences. *Critical Reviews in Food Science and Nutrition, 25,* 185-282.
19. Hornero-Méndez, Dámaso, Mínguez Mosquera, María Isabel, (2008): Isolation and Identification of the Carotenoid Capsolutein from Capsicum annuum as Cucurbitaxanthin A, Pub: American Chemical Society.
20. https://www.perfume.com/article-history-of-perfume.

21. J.M. and Richardson J., (2003) *Particle Technology and Separation Process*. Volume 6.India: Elsevier Publishers.

22. John Bostock, M.D, Riley, Esq., B.A, (1855): The Natural History. Pliny the Elder. London. Taylor and Francis, Red Lion Court, Fleet Street. 1855.

23. Kocsis, N., Amtmann, M., Mednyánszky, Zs., Korány, K. (2002). GC-MS Investigation of the aroma compounds of Hungarian red paprika (*Capsicum annuum*) Cultivars. *Journal of Food Composition and Analysis*, *15*, 195-203.

24. Kocsis, N., Márkus, F., Mednyánszky, Zs., Amtmann, M., Korány, K. (2003). Recognition experiments of the vintage year 1997 hot and red paprika (*Capsucum annuum*) varietes grown in Kalocsa. *Acta Alimentaria*, *32*, 63-75.

25. Leung A. Y. and Foster S. (1996): Encyclopedia of Common Natural Ingredients Used in Food, Drugs and Cosmetics. Second edition, 649 pages. John Wiley & Sons, Inc., New York, Chichester, Brisbane, Toronto, Singapore 1996.

26. Levey, Martin (1973). *Early Arabic Pharmacology: An Introduction Based on Ancient and Medieval Sources*. Brill Archive. p. 9. ISBN 90-04-03796-9.

27. Mateo, J., Aguirrezábal, M., Domíngez, C., Zumalacárregui, J.M. (1997). Volatile com-pounds in Spanish paprika. *Journal of Food Composition and Analysis*, *10*, 225-232.

28. Moore, M.J and Michael, O (2006). *Fragrances of the World 2006*. London: Crescent House Publishing.

29. N. Groom., 1997 New Perfume Handbook. Springer Science & Business Media, 1997 ISBN 9780751404036, pp. 184-186).

30. Ralkin Robert, R. Jellinek, and J. Stephen (1994): Leaching Method, London; Longman publishers

31. Reeds, *P. J. (2000).* "Dispensable and indispensable amino *acids* for humans". *The*

Journal of nutrition. ***130*** *(7): 1835S–40S. PMID 10867060.*

32. Rezzoug, S. A, Boutekedjiret, C, Allaf, K. (2005): "Optimization of operating conditions of rosemary essential oil extraction by fast controlled pressure drop process using response surface methodology," *Journal of Food Engineering*, vol. 1, pp. 9-17, 2005.
33. Sudeep Tandon, (2008): Hydro-distillation and steam distillation and steam distillation from aromatic plants, Chemical Engineering Division, CIMAP, Lucknow, Indian.
34. Sundeep Tandon (2008): Distillation Technology for Essential Oils; Extraction Technologies for Medicinal and Aromatic Plant; International Centre for Science and High Technology, Trieste 2008.
35. Thorpe's Dictionary *of* Applied Chemistry. *8 (4th Ed.).* Longmans *Green. 1947.*
36. Žarko S. Kevrešan, Nataša Č. Hrabovski, Ksenija N. Kuhajda, Neda Mimica-Dukić, Marijana B. Sakač: (2009): Essential Oil Composition Of Fresh And Dried Pepper Fruits (Capsicum annuum L), Food and Feed Research, Journal of Institute of Food Technology, Novi Sad.
37. Rumsfield J.A, West D.P, (1991): Topical Capsaicin in Dermatologic and Peripheral Pain Disorders; US National Library of Medicine, National Institute of Health.
38. Yahara S., Ura T, Sakamoto C., Nohara T., (1994): Steroidal Glycosides From Capsicum Annuum; US National Library of Medicine, National Institute of Health.