"EXTRACTION OF ESSENTIAL OIL USING STEAM DISTILLATION"

A THESIS SUBMITTED IN PARTIAL FULFILLMENT

OF THE REQUIREMENTS FOR THE DEGREE OF

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IN

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UNDER THE GUIDANCE OF

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CERTIFICATE

This is to certify that the thesis entitled, **"Extraction of Essential oil using steam distillation**" submitted by **K. Satish kumar** in partial fulfillments for the requirements for the award of Bachelor of Technology Degree in Chemical Engineering at **National Institute of Technology**, **Rourkela** is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University / Institute for the award of any Degree or Diploma.

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ABSTRACT

A large number of herb materials contain Essential Oils with extensive bioactivities. Acknowledging the importance of plants and its medicinal value, extraction of Essential Oil had been done using Steam Distillation method. In this project Steam Distillation was used to extract oil from different plant materials like eucalyptus leaves, curry leaves, hibiscus leaves, lemon leaves, marigold flowers, rose flowers, orange peels etc. Research has confirmed centuries of practical use of essential oils, and we now know that the 'fragrant pharmacy' contains compounds with an extremely broad range of biochemicaleffects. Essential oils are so termed as they are believed to represent the very essence of odor and flavor. The recovery of Essential Oil (the value added product) from the raw botanical starting material is very important since the quality of the oil is greatly influenced during this step. There are a variety of methods for obtaining volatile oils from plants. Steam distillation method was found to be one of the promising techniques for the extraction of essential oil from plants as reputable distiller will preserve the original qualities of the plant. Thedistillation was conducted in Clevenger apparatus in which boiling, condensing and decantation was done. Analysis of Essential oil was done using Gas Chromatography-Mass Spectrometer apparatus, which givesevaluates Essential Oil qualitatively and quantitatively. Volume of Essential Oil obtained was changing w.r.t temperature and time of heating.

CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1

INTRODUCTION

1. INTRODUCTON:

Essential oils contain highly volatile substances that are isolated by a physical method or process from plants of a single botanical species. The oils normally bear the name of the plant species from which they are derived. Essential oils are so termed as they are believed to represent the very essence of odor and flavor. Essential oil plants and culinary herbs include a broad range of plant species that are used for their aromatic value as flavorings in foods and beverages and as fragrances in pharmaceutical and industrial products. Essential oils derive from aromatic plants of many genera distributed worldwide.

Oils are used in the embalming process, in medicine and in purification rituals. There are also over 200 references to aromatics, incense and ointments in the Old and New Testaments. Research has confirmed centuries of practical use of Essential Oils, and we now know that the 'fragrant pharmacy' contains compounds with an extremely broad range of biochemical effects. There are about three hundred essential oils in general use today by professional practitioners. Continual bombardment of viral, bacterial, parasitic and fungal contamination occurs in our body. Essential oils are a great benefit to help protect our bodies and homes from this onslaught of pathogens. Immune system needs support and these essential oils can give the required endorsement. [1]

Steam distillation is used in the extraction of Essential Oil from the plant material. It is a special type of distillation or a separation process for temperature sensitive materials like oils, resins, hydrocarbons, etc. which are insoluble in water and may decompose at their boiling point. The fundamental nature of steam distillation is that it enables a compound or mixture of compounds to be distilled at a temperature substantially below that of the boiling point(s) of the individual constituent(s). Essential Oil contains components with boiling points up to 200°C or higher temperatures. In the presence of steam or boiling water, however, these substances are volatilized at a temperature close to 100°C, at atmospheric pressure. [2]

Analysis of Essential Oil is done by using Gas Chromatography with Mass Spectrometer. The qualitative and quantitative analysis is done to know the constituents in the oil and the percentage of components present in the oil respectively, by doing so we can know the purity of that particular oil. [3]

CHAPTER 2

LITERATURE REVIEW

It is estimated that there are 250,000 to 500,000 species of plants on Earth. A relatively small percentage (1 to 10%) of these is used as foods by both humans and other animal species. It is possible that even more are used for medicinal purposes (Moerman, D. E. 1996). Moerman (1996) reported that while 625 species of plants have been used by various Native American groups as food, 2,564 have found use as drugs. According to his calculations, this leaves approximately 18,000 species of plants which were used for neither food nor drugs. [4]

Plant oils and extracts have been used for a wide variety of purposes for many thousands of years (Jones 1996). These purposes vary from the use of rosewood and cedar wood in perfumery, to flavoring drinks with lime, fennel or juniper berry oil, and the application of lemongrass oil for the preservation of stored food crops. In particular, the antimicrobial activity of plant oils and extracts has formed the basis of many applications, including raw and processed food preservation, pharmaceuticals, alternative medicine and natural therapies.

Since ancient times, herbs and their essential oils have been known for their varying degrees of antimicrobial activity. More recently, medicinal plant extracts were developed and proposed for use in food as natural antimicrobials.

2.1 DEFINITION:

An essential oil is a concentrated, hydrophobic liquid containing volatile aroma compounds from plants. Essential oils are also known as volatile, ethereal oils or 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 carries a distinctive scent, or essence, of the plant. [5]

Essential oils are frequently referred to as the “life force” of plants. These "essential" oils are extracted from flowers, leaves, stems, roots, seeds, bark, and fruit rinds. The amount of essential oils found in these plants can be anywhere from 0.01 percent to 10 percent of the total. These oils have potent antimicrobial factors, having wide range of therapeutic constituents. These oils are often used for their flavor and their therapeutic or odoriferous properties, in a wide selection of products such as foods, medicine, and cosmetics. Only pure oils contain a full spectrum of compounds that cheap imitations simply cannot duplicate.[5]

Table-2.1

Major Raw Material Used In Extraction of Essential Oils:

Leaves	**Flowers**	**Peel**	**Seeds**	**Wood**
Basil	Chamomile	Bergamot	Almond	Camphor
Bay leaf	Clary Sage	Grape fruit	Anise	Cedar
Cinnamon	Clove	Lemon	Celery	Rosewood
Eucalyptus	Geranium	Lime	Cumin	Sandalwood
Lemon Grass	Hyssop	Orange	Nutmeg Oil	
Melaleuca	Jasmine	Tangerine		
Oregano	Lavender			
Patchouli	Manuka			
Peppermint	Marjoram			
Pine	Orange			
Rosemary	Rose			
Spearmint	Ylang-Ylang			
Tea Tree				
Wintergreen				
Thyme				
Berries	**Bark**	**Resins**	**Rhizome**	**Root**
Allspice	Cassia	Frankincense	Ginger	Valerian
Juniper	Cinnamon	Myrrh		

2.2 CHEMICAL CONSTITUENTS OF ESSENTIAL OILS: AnEssential Oil contains more than 200 chemical components, but some are many times more complex. Essential oils consist of chemical compounds which have hydrogen, carbon and oxygen as their building blocks. [12] 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: This comprises 1–10% of the oil, containing hydrocarbons, fatty acids, sterols, carotenoids, waxes, and flavonoids.

However the properties of these components can change. For example, the components from the oils extracted from plants can change according to how, when and where these plants are grown and harvested.[6] The constituents can be again subdivided into 2 groups, such as the hydrocarbons which are made up of mostly terpenes and the oxygenated compounds which are mainly alcohols, aldehydes, esters, ketones, phenols and oxides. Some of the common components are listed below along with their properties.

- **Alcohols:**

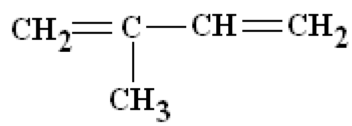
  Alcohols are generally considered safe and have a very low or totally absent toxic reaction in the body or on the skin and so can be used on children. They are extremely useful due to their antiviral, antibacterial and antiseptic properties. Alcohols are present either as a free compound or combined with a terpene or ester and are found in ylang - ylang and lavender as linalool, geraniol in geranium and palmarosa and citronellol found in rose, lemon and eucalyptus. Other alcohols include menthol, nerol and benzyl alcohol.

- **Alderhydes:**

  Alderhydes are found in lemon-scented oils such as Melissa, lemon verbena, citronella etc. and include citral, citronellal and neral. They generally have sedative qualities with specific antiseptic properties. Other known alderhydes include benzaldehyde, cinnamic alderhyde and perillaldehyde. Essential oils containing alderhydes are helpful in treating inflammation, Candida and viral infections.[6]

- **Hydrocarbon:**

Building blocks of Essential Oil are hydrogen and carbon. Basic Hydrocarbon found in plants is isoprene having the following structure.



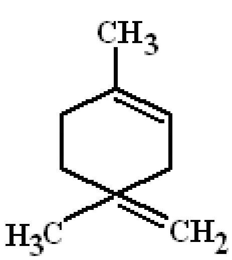
(Isoprene)

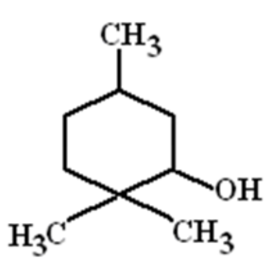
- **Terpenes:**

These components generally have names ending with "**ene**".Some of them are limonene, pinene, piperene, camphene etc. These components act as an antibacterial, antiviral,anti-inflammatory, antiseptic, antiviral and bactericidal. These are further categorized intomonoterpene, sesquiterpene and diterpenes. When two of theisoprene units are joined head to tail, the result is a monoterpene, when three are joined, it's a sesquiterpene and similarly four linked isoprene units are diterpenes. [11]

- **Monoterpene [$C_{10}H_{16}$]:**

Monoterpene 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)

Two isoprene units are present in these branched-chain C_{10} hydrocarbons and are widely distributed in nature with more than 400 naturally occurring monoterpenes. Moreover, besides being linear derivatives (Geraniol, Citronellol), the monoterpenes can be cyclic molecules (Menthol – Monocyclic; Camphor – bicyclic; Pinenes (**α**and **β**) – Pine genera as well. Thujone (a monoterpene) is the toxic agent found in Artemisiaabsinthium (wormwood) from which the liqueur absinthe, is made. Borneol and camphor are two common monoterpenes. Borneol, derived from pine oil is used as a disinfectant and deodorant. Camphor is used as a counterirritant, anesthetic, expectorant, and antipruritic, among many other uses.

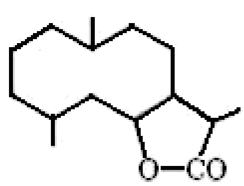
- **Sesquiterpene:**

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.These are having properties like anti-inflammatory, anti-septic, analgesic and anti-allergic.

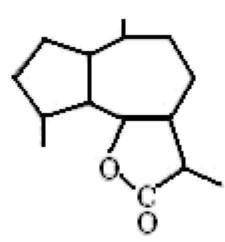
- **Sesquiterpene Lactones:**

These are available as farnesene in chamomile and lavender. They not only have proved to be of interest from chemical and chemotaxonomic point of view, but also possess many antitumor, anti-leukemia, cytotoxic and antimicrobial activities.

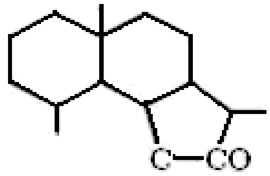
Chemically the compounds can be classified according to their carboxylic skeletons; thus, guaianolides, pseudoguaianolides, eudesmanolides, eremophilanolides, xanthanolides, etc. can be derived from the germacranolides.



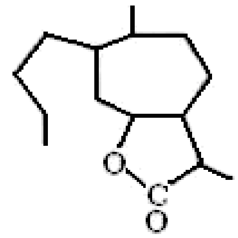
Germacranolides



Guaianlides



Eudesmanolides



Xantnanolides

Structural features of all these compounds are associated with much of the biological activity. For example beta-caryophyllene in basil and black pepper

- **Diterpenes:**

  Isoprene has been an integral part in most of the components as there are four isoprene units in Diterpenes. By Stem Distillation method we cannot detect Diterpenes as this molecule is too heavy to allow for evaporation, so it 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. Derivatives of Diterpenes areplant hormones Gibberellins and phytol occurring as a side chain on chlorophyll. 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). Therapeutically Diterpenes have limited importance and are used in certain sedatives (coughs) as well as in antispasmodics and anxiolytics.

- **Alcohols:**

  Naturally Alcohols exist 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 are not and are suitable to body or skin. Therefore, they are considered safe to use.Some of these properties areanti-septic, anti-viral, bactericidal and germicidal. Some of the examples are linalool found in ylang-ylang and lavender, geraniol in geranium and rose and nerol in neroli.

- **Aldehydes**:

  Aldehyde containing Essential Oils are effective in treating candida and other fungal infections. Some of these properties are anti-fungal, anti-inflammatory, anti-septic, anti-viral, bactericidal, disinfectant, and sedative. Aldehydes are present as citral in lemon, Citronellal in lemongrass, lemon balm and citrus eucalyptus.

- **Acids:**

  Generally Organic acids are found in very small quantities in their free state within Essential Oils. Plant acids act as components or buffer systems to control acidity. These also act anti-inflammatory. Examples are cinnamic and benzoic acid in benzoin, Citric and lactic.

- **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. Examples are linlyl acetate in bergamot and lavender and Geranyl formate in geranium. [15]

- **Ketones:**

  Ketones found in plants 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 (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.

- **Lactones:**

  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 than ketones.

2.3ADVANTAGES OF ESSENTIAL OIL:

- **AROMATHERAPHY:** Aromatherapy is a form of alternative medicine that uses volatile plant materials, known as essential oils, and other aromatic compounds for the purpose of altering a person's mood, cognitive function or health. Science has discovered that our sense of smell plays a significant role in our overall health.[8]

  Since ancient times Essential Oils have been used in medicine because of their medicinal properties, for example some oils have antiseptic properties. In addition, many have an uplifting effect on the mind, though different essential oils have different properties.

  Working of Essential Oil in Aromatherapy: when Essential Oil is inhaled it goes directly from olfactory system to limbic system of the brain. Brain responds to the particular scent affecting our emotions and chemical balance. Essential Oils also absorbed by the skin and carried throughout the body via the circulatory system to reach all internal organs.

  We can be benefited by choosing carefully the desired and suitable oils which can promote overall health. Benefits depend upon the unique nature of each person's response to an aromatic stimulus. [1]

- **Importance of Essential Oil in pharmaceutics:** Essential Oils have versatile applications in pharmaceutics. Some of the applications are listed below.

  Antiseptics: The antiseptic properties of Essential Oil make them active against wide range of bacteria as on antibiotic resistant strains. In addition to this they are also against fungi and yeasts. The most common sources of essential oils used as antiseptics are: Cinnamon, Thyme, Clover, Eucalyptus,Culinsavory,and Lavender. Citral, geraniol, linalool and thymol are much more potent than phenol. [1]

  Expectorants and diuretics: When used externally, essential oils like (L'essence de terebenthine) increase microcirculation and provide a slight local anesthetic action. Till now, essential oils are used in a number of ointments, cream and gels, whereby they are known to be very effective in relieving sprains and other articular pains. Oral administration of essential oils like eucalyptus or pin oils, stimulate ciliated epithelial cells to secrete mucus. On the renal system, these are known to increase vasodilation and in consequence bring about a diuretic effect**.**

Spasmolytic and sedative: Essential oils from the Umbellifereae family, Mentha species and verbena are reputed to decrease or eliminate gastrointestinal spasms. These essential oils increase secretion of gastric juices. In other cases, they are known to be effective against insomnia.

2.4.1 METHODS OF EXTRACTION: The following are the methods of extraction of Essential Oil and their drawbacks.

2.4.1 Solvent-Extraction:

In the Solvent-Extraction method of Essential Oils recovery, an extracting unit is loaded with perforated trays of essential oil plant material and repeatedly washed with the solvent. A hydrocarbon solvent is used for extraction. All the extractable material from the plant is dissolved in the solvent. This includes highly volatile aroma molecules as well as non-aroma waxes and pigments. The extract is distilled to recover the solvent for future use. The waxy mass that remains is known as the concrete. The concentrated concretes are further processed to remove the waxy materials which dilute the pure essential oil. To prepare the absolute from the concrete, the waxy concrete is warmed and stirred with alcohol (ethanol). During the heating and stirring process the concrete breaks up into minute globules. Since the aroma molecules are more soluble in alcohol than the waxes, an efficient separation of the two results. This is not considered the best method for extraction as the solvents can leave a small amount of residue behind which could cause allergies and effect the immune system.

2.4.2 Maceration:

Maceration actually creates more of “infused oil” rather than an Essential Oil. Plant matter is soaked in vegetable oil, heated and strained at which point it can be used for massage. This method is not desirable because it changes the composition of oil.

2.4.3 Cold Pressing:

This method is used to extract the Essential Oils from citrus rinds such as orange, lemon, grapefruit and bergamot. This method involves the simple pressing of the rind at about 120 degrees F to extract the oil. The rinds are separated from the fruit, are ground or chopped and are then pressed. The result is a watery mixture of essential oil and liquidwhich will separate given time. Little alteration from the oil's original state occurs – these citrus oils retain their bright, fresh, uplifting aromas like that of smelling a wonderfully ripe fruit. The drawback of this method is, oils extracted using this method have a relatively short shelf life.

2.4.4 Effleurage:

This is one of the traditional ways of extracting oil from flowers. The process involves layering fat over the flower petals. After the fat has absorbed the essential oils, alcohol is used to separate and extract the oils from the fat. The alcohol is then evaporated and the Essential Oil is collected. [13]

2.4.5 Super Critical CO2 Extraction:

Supercritical CO2 extraction (SCO2) involves carbon dioxide heated to 87 degrees F and pumped through the plant material at around 8,000 psi, under these conditions; the carbon dioxide is likened to a 'dense fog' or vapor. With release of the pressure in either process, the carbon dioxide escapes in its gaseous form, leaving the Essential Oil behind. The usual method of extraction is through steam distillation. After extraction, the properties of a good quality essential oil should be as close as possible to the "essence" of the original plant. The key to a 'good' essential oil is through low pressure and low temperature processing. High temperatures, rapid processing and the use of solvents alter the molecular structure, will destroy the therapeutic value and alter the fragrance. [10]

2.4.6 Turbo Distillation Extraction:

Turbo distillation is suitable for hard-to-extract or coarse plant material, such as bark, roots, and seeds. In this process, the plants soak in water and steam is circulated through this plant and water mixture. Throughout the entire process, the same water is continually recycled through the plant material. This method allows faster extraction of essential oils from hard-to-extract plant materials.

Dr.A.Sahoo et al have studied that Steam Distillation is a special type of distillation or a separation process for temperature sensitive materials like oils, resins, hydrocarbons, etc. which are insoluble in water and may decompose at their boiling point. The temperature of the steam must be high enough to vaporize the oil present, yet not so high that it destroys the plants or burns the essential oils. The experiment has been carried out for the extraction of oil from Eucalyptus which has high essential oil content. Such Eucalyptus essential oil, which have been used as perfume and chemical raw materials for a long time, are now been studied as renewable sources of energy.

G. Anitescu et al have studied that ripe fruits of Coriander sativum L. were extracted by steam distillation and by supercritical fluid extraction (SFE),using CO2 in a two-stage separation system. An inexpensive thermal expansion procedure for supercritical fluid delivery has been developed. The identification of components was performed by gas chromatography and mass spectrometry (GC±MS). The percentage composition of the 40 identified compounds was compared with the composition of commercial coriander oil extracted by hydro distillation.

Roy Teranishi et al have studied that system combines steam distillation and liquid-liquid extraction to recover volatiles from fats and oils. Oil is pumped in at the top of a spinning-band distillation column, in which the oil is heated to100 "C and spread to a thin film. As the oil film drops down to the pot, steam, which is introduced at the bottom, travels upward to strip the volatiles from the oil. The steam distillate is extracted in liquid-liquid extractor incorporated in the system, and the extracted water is recycled as steam. Stripped oil in the pot serves as a liquid seal to force steam up the column. The level of the oil in the pot is maintained automatically by an overflow system. Many liters of oil can be pumped through this system to be stripped of volatiles by steam. The volatiles can be isolated easily from the small amount of solvent recycled in the liquid-liquid extractor.

Referring to the above literature review, it was found that Steam Distillation method is an appropriate and economical method for extraction of Essential Oil.

2.4.7 Extraction of Essential Oils Using Steam distillation Method:

Steam distillation is a special type of distillation or a separation process for temperature sensitive materials like oils, resins, hydrocarbons, etc. which are insoluble in water and may decompose at their boiling point. The fundamental nature of steam distillation is that it enables a compound or mixture of compounds to be distilled at a temperature substantially below that of the boiling point(s) of the individual constituent(s). Essential oils contain substances with boiling points up to 200°C or higher temperatures. In the presence of steam or boiling water, however, these substances are volatilized at a temperature close to 100°C, at atmospheric pressure.

Fresh, or sometimes dried, botanical material is placed in the plant chamber of the still and the steam is allows to pass through the herb material under pressure which softens the cells and allows the Essential Oil to escape in vapor form. The temperature of the steam must be high enough to vaporize the oil present, yet not so high that it destroys the plants or burns the EssentialOils. Besides the steam tiny droplets of Essential Oil evaporates and travel through a tube into the still's condensation chamber. Here Essential Oil vapors condense with the steam. The essential oil forms a film on the surface of the water. To separate the Essential Oil from the water, the film is then decanted or skimmed off the top. The remaining water, a byproduct of distillation, is called floral water, distillate, or hydrosol. It retains many of the therapeutic properties of the plant, making it valuable in skin care for facial mists and toners (A solution containing chemicals that can change the color of a photographic print). In certain situations, floral water may be preferable to be pure essential oil, such as when treating a sensitive individual or a child, or when a more diluted treatment is required. Rose hydrosol, for example, is commonly used for its mild antiseptic and soothing properties, as well as its pleasing floral aroma.

A number of factors determine the final quality of a steam distilled essential oil. Apart from the plant material, most important are time, temperature and pressure, and the quality of the distillation equipment. Essential oils are very complex products. Each is made up of many, sometimes hundreds, of distinct molecules which come together to form the oil's aroma and therapeutic properties. Some of these molecules are fairly delicate structures which can be altered or destroyed by adverse environmental conditions. So, much like a fine meal is more flavorful when made with patience, most oils benefit from a long, slow 'cooking' process. [9] It is possible that longer distillation times may give more complete oil. It is also possible however, that longer distillation time may lead to the accumulation of more artifacts than normal. This may have a curious effect of appearing to improving the odor, as sometimes when materials that have a larger number of components are sniffed, the perception is often of slightly increased sophistication, added fullness and character, and possibly, and extra pleasantness.

Advantages of using Steam Distillation:

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 not altered. As steam reduces the boiling point of a particular component of the oil, it never decomposes in this method. This method apart from being economical, it is also relatively faster than other methods.

CHAPTER 3

EXPERIMENTAL WORK

3.1 Experimental Setup:

The schematic diagram of experimental setup is shown below. The experiment was conducted in a Clevenger's Apparatus. Apparatus consist of one round bottom flask of 1000ml which is connected with another two way round flask which holds raw material. The top flask is connected with condenser through the connecter. The separating funnel is used for the separation of essential oil and water.

3.2 Experimental Procedure:

Fresh leaves (rosemary) or bark (cinnamon) are cut into pieces less than 2 X 2 cm within half a day after collection and 150-200 g boiled with 500 ml of distilled water in a Clevenger apparatus until oil distillation ceased after 5-6 h. The volume of essential oils was determined from a calibrated trap. The essential oils in the distillate were dried over anhydrous Na_2SO_4 and kept in the freezer.

Figure-3.1 Essential Oil Steam Distiller

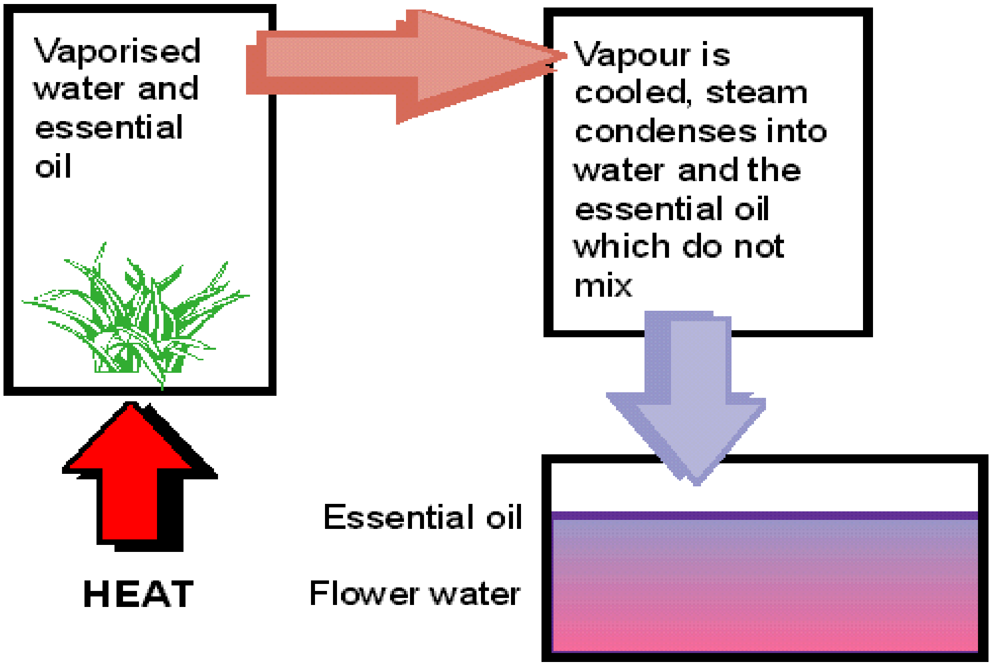


Figure-3.2Flow sheet of Steam Distillation

3.3Analysis of Essential Oils:

Analysis of Essential Oil was done using Gas Chromatography with Mass spectrometer to know the composition of oil and to the quantity of each composition.

3.3.1 Gas Chromatography-Mass Spectrometer:

Gas Chromatography-Mass Spectrometry (GC-MS) is a method that combines the features of gas-liquid chromatographyand mass spectrometry to identify different substances within a test sample.Applications of GC-MS include drug detection, fire investigation, environmental analysis, explosives investigation, and identification of unknown samples. Additionally, it can identify trace elements in materials that were previously thought to have disintegrated beyond identification. [7]

Instrumentation:

The GC-MS is composed of two major building blocks: the gas chromatograph and the mass spectrometer. The gas chromatograph utilizes a capillary column which depends on the column's dimensions (length, diameter, film thickness) as well as the phase properties (e.g. 5% phenyl polysiloxane). The difference in the chemical properties between different molecules in a mixture will separate the molecules as the sample travels the length of the column. The molecules take different amounts of time (called the retention time) to come out of (elute from) the gas chromatograph, and this allows the mass spectrometer downstream to capture, ionize, accelerate, deflect, and detect the ionized molecules separately. The mass spectrometer does this by breaking each molecule into ionized fragments and detecting these fragments using their mass to charge ratio.[14] These two components, used together, allow a much finer degree of substance identification than either unit used separately. It is not possible to make an accurate identification of a particular molecule by gas chromatography or mass spectrometry alone. The mass spectrometry process normally requires a very pure sample while gas chromatography using a traditional detector (e.g. Flame Ionization Detector) detects multiple molecules that happen to take the same amount of time to travel through the column (*i.e.* have the same retention time) which results in two or more molecules to co-elute. Sometimes two different molecules can also have a similar pattern of ionized fragments in a mass spectrometer (mass spectrum). Combining the two processes makes it extremely unlikely that two different molecules will behave in the same way in both a gas chromatograph and a mass spectrometer. Therefore when an identifying mass spectrum appears at a characteristic retention time in a GC-MS analysis, it typically lends to increased certainty that the analyte of interest is in the sample. [7]

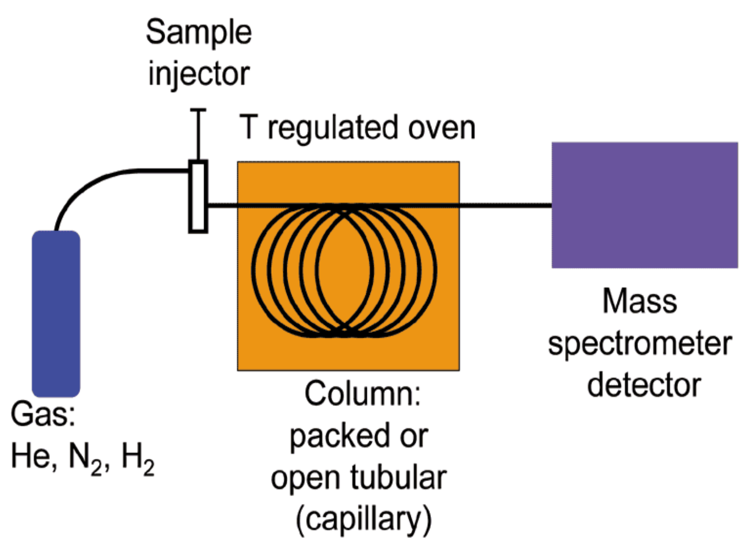


Fig 3.3 Gas Chromatography-Mass spectrometer schematic diagram

3.4 Experimental Observation:

Experiment had been conducted using different plant materials at different temperatures and time of heating, keeping pressure constant (atmospheric pressure).

3.1 Table showing different volumes of Essential Oil extracted from different plant materials and their time of heating.

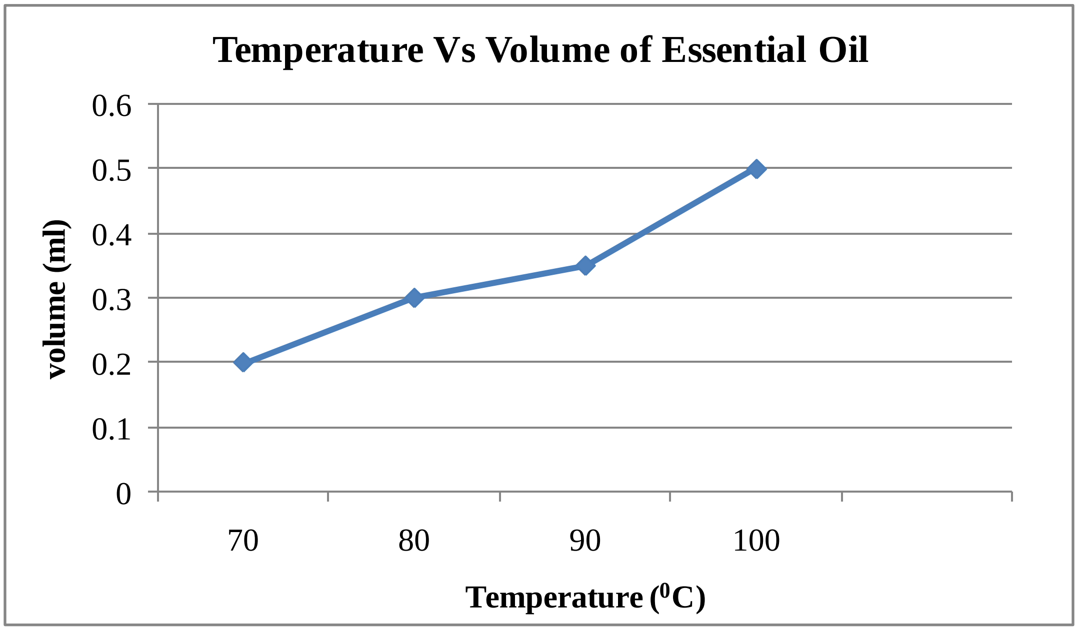
Sl. No.	Plant material	Weight (gm.)	Volume of distilled water (ml)	Time of heating (min)	Temperature (^{0}C)	Volume of Essential Oil (ml)	Volume of hydrosol (ml)
1	**Orange peels**	300	750	80	100	0.5	>10
2		300	750	80	70	0.2	<10
3		300	750	80	80	0.3	~10
4		300	750	120	90	0.4	>10
5		300	750	90	90	0.35	>10
6	**Eucalyptus leaves**	130	750	93	100	0.5	>10
7		130	750	100	80	0	>10
8		130	750	80	100	0.2	>10
9		130	750	120	100	0.3	>10
10	**Lemon leaves**	150	800	84	105	0.6	>10
11		154	800	90	100	0.45	>10
12		142	800	180	100	0.65	>10
13		141	800	120	100	0.5	>10
14	**Marigold flowers**	150	750	120	100	0	>10
15		151	750	180	100	0	>10
16	**Rose petals**	141	750	180	100	0	>10
17	**Hibiscus leaves**	198	750	180	100	0	>10
18	**Curry leaves**	88	750	180	100	0	>10

CHAPTER 4

RESULTS AND DISCUSSION

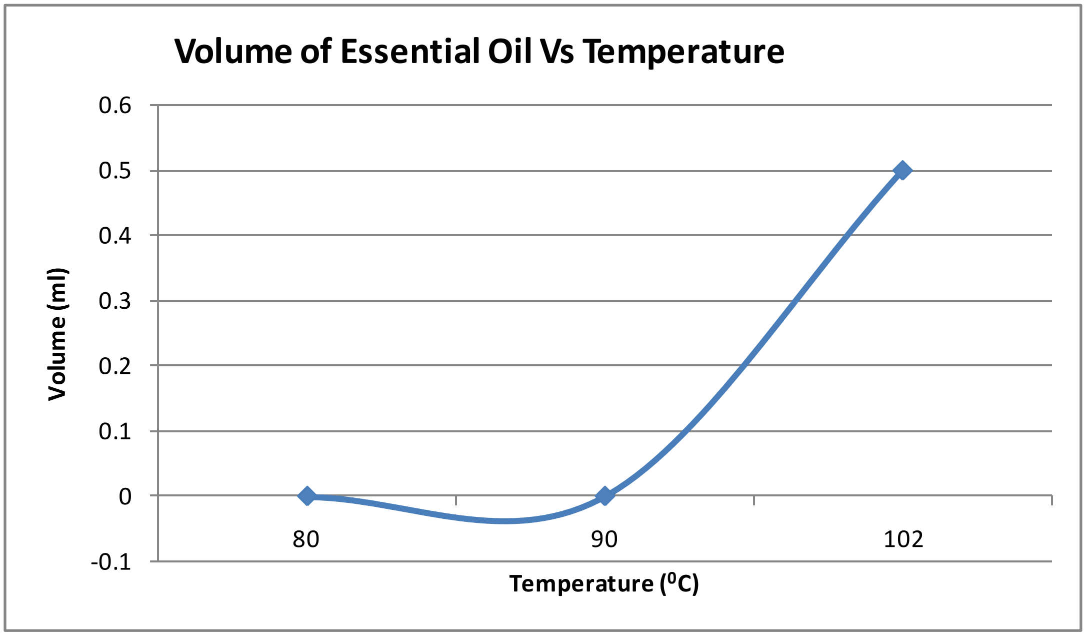
4.1 Inferences:

Volume of Essential Oil obtained from a particular plant material was different for different temperatures and at a particular temperature for different time of heating. Volume of Essential Oil obtained is less in comparison to hydrosol of the same plant material. Its variation along with temperature and time of heating is shown in graphs drawn below.



4.1 Graph showing variation of Volume of Essential Oil against Temperature, obtained from Orange peels.

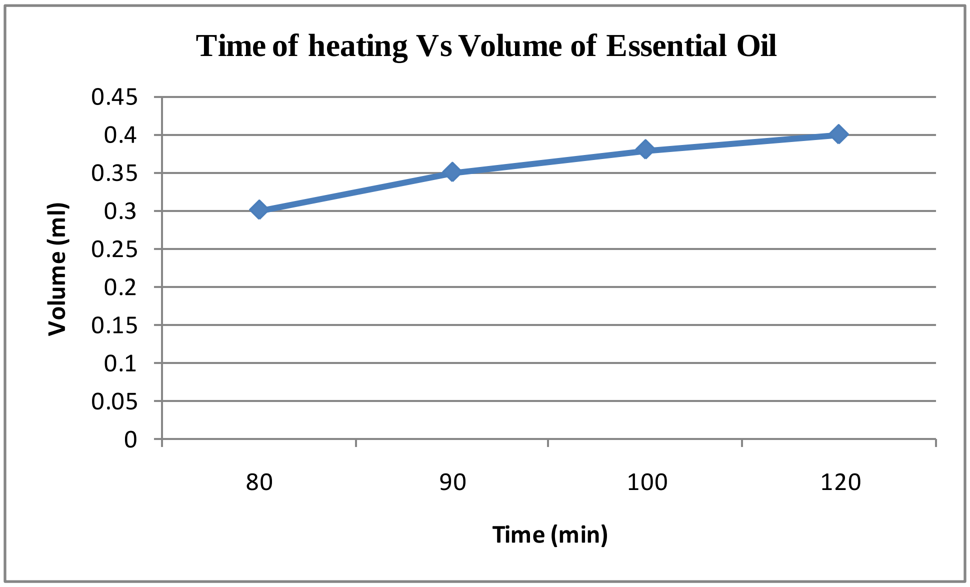
From the graph we can observe that volume of Essential Oil obtained from orange peels is almost increasing linearly with Temperature, if we ignore the slight decline in volume at 90^{0}C. The decline may be due to leakage of vapor during Steam Distillation or because of improper decantation.



4.2 Graph showing variation of Volume of Essential Oil against Temperature, obtained from Eucalyptus leaves.

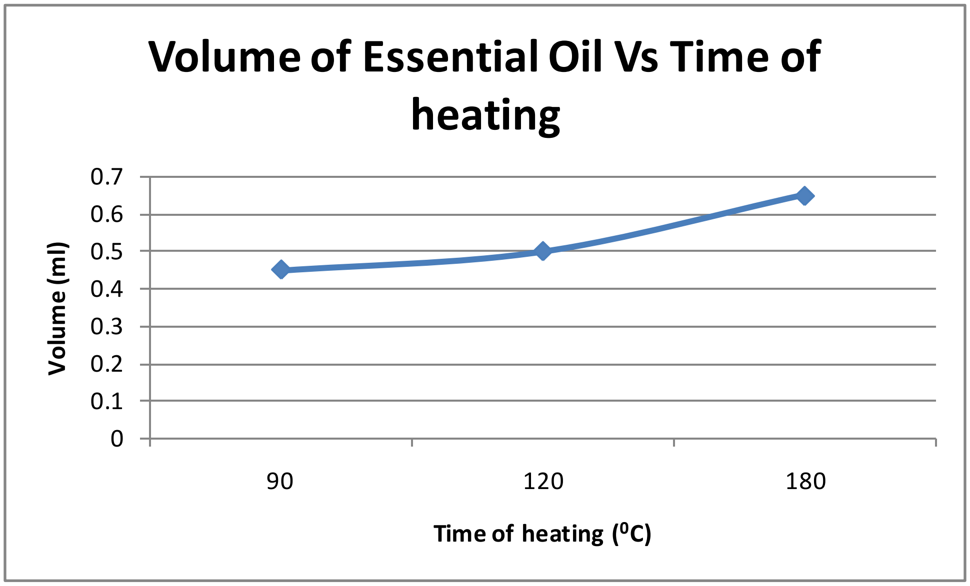
From the above graph we can observe that volume of Essential Oil obtained is negligible before the components of the oil reach to their boiling point. After reaching to their boiling points Essential Oil is obtained and further we can speculate that if increase more temperature proliferation in volume can be achieved.

Now variation of Volume of Essential Oil obtained with Time of heating for different plant materials is shown below.



4.3 Graph showing variation of Volume of Essential Oil with Time of heating, obtained from Orange peels.

From the graph we can observe that volume of Essential Oil obtained from orange peels is almost increasing linearly with time of heating at a particular temperature (90^0C). But the increment is not steep in this case.



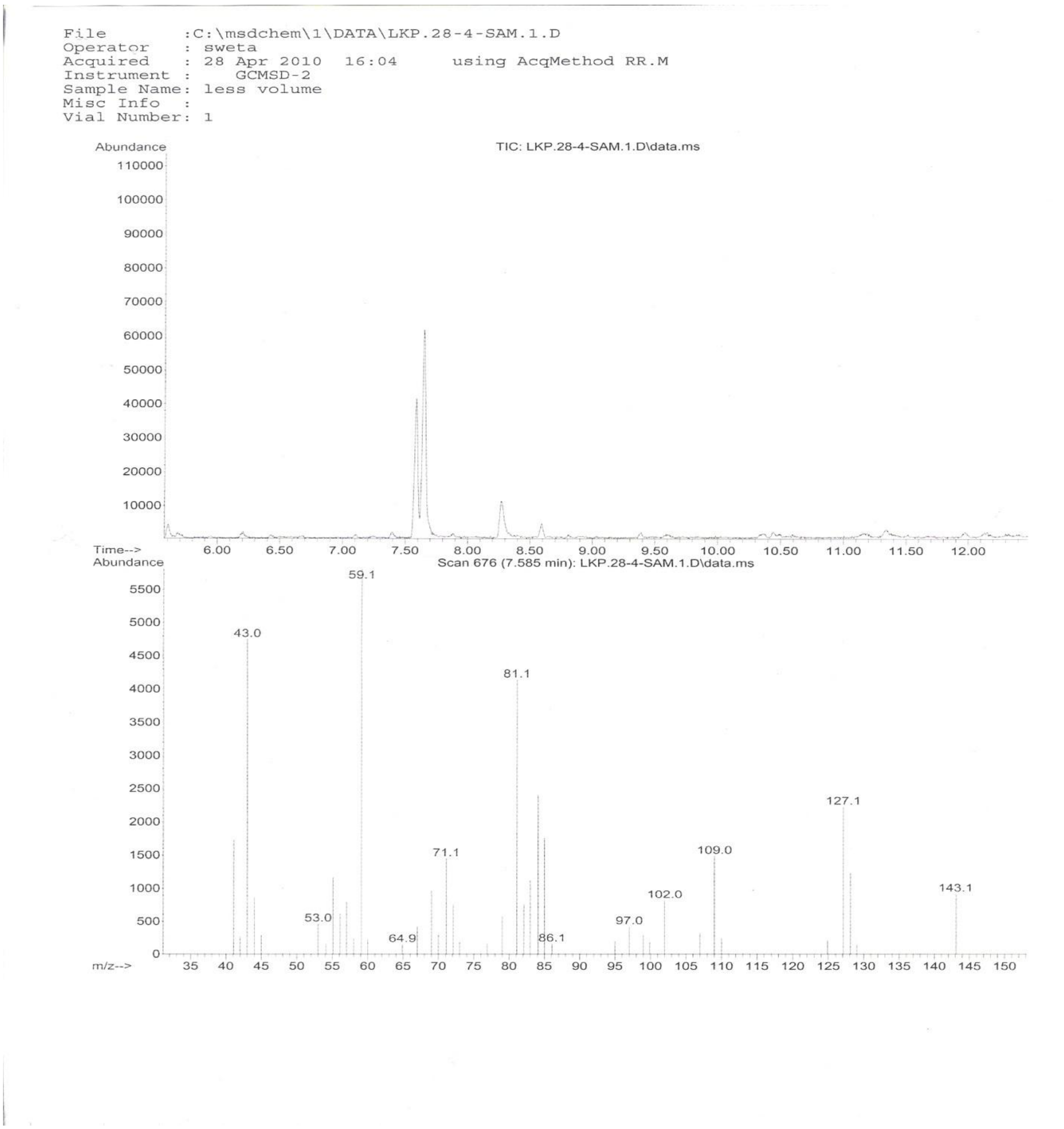
4.4 Graph showing variation of Volume of Essential Oil against Time of heating, obtained from Lemon leaves.

From the graph we can observe that volume of Essential Oil obtained from Lemon leaves is almost increasing linearly with time of heating at a particular temperature (100^0C). But the increment is not steep in this case.

In case of other plant materials volume of Essential Oil was not obtained, may be due to the temperature supplied was not sufficient to vaporize the components of oils to their boiling points, as show in the table 3.4.1.

4.2 Graphs showing analysis of Essential Oil:

Analysis of Essential Oil obtained after Steam Distillation was done using Gas chromatography-Mass Spectrometer method. The analysis shows the composition of the oil and also the percentage of each component. This is shown in graph produced by Gas chromatography-Mass Spectrometer apparatus. The graph generated by Gas chromatography shows the composition of the oil and the other graph shown by Mass Spectrometer gives the percentage of each component. Some of the graphs obtained after analysis are shown below.



4.5 Graphs showing the quantitative and qualitative analysis of Essential Oil extracted from Lemon leaves.

From the above graph we find the composition of the Lemon Essential Oil as α-thujene,α-Pinene, Camphene, β-Pinene, Sabinene, Myrcene, Limonene, ρ-cymene, α-terpinolene.

Sesquiterpenes: α-Terpinene, ϒ-Terpinene.

Monoterpenol: Citronellol, Capraldehyde.

Aldehydes: Caprinaldehyde, Citral, Benzaldehyde, 2-hexene, Decanol, Borneol, Linalool.

Ester: Citranelyl acetate.

Of all the components the major component is Limonene, which is having highest abundance w.r.t the mass/charge and retention time as shown by the highest peaks in the graph.

Properties of Limonene:

- Molecular formula : $C_{10}H_{16}$
- Molar mass : 136.24 g/mol
- Density : 0.8411 g/cm³
- Melting point : 74.35 °C
- Boiling point : 176 °C

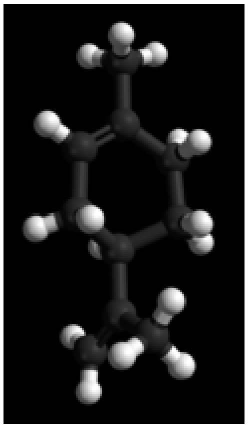
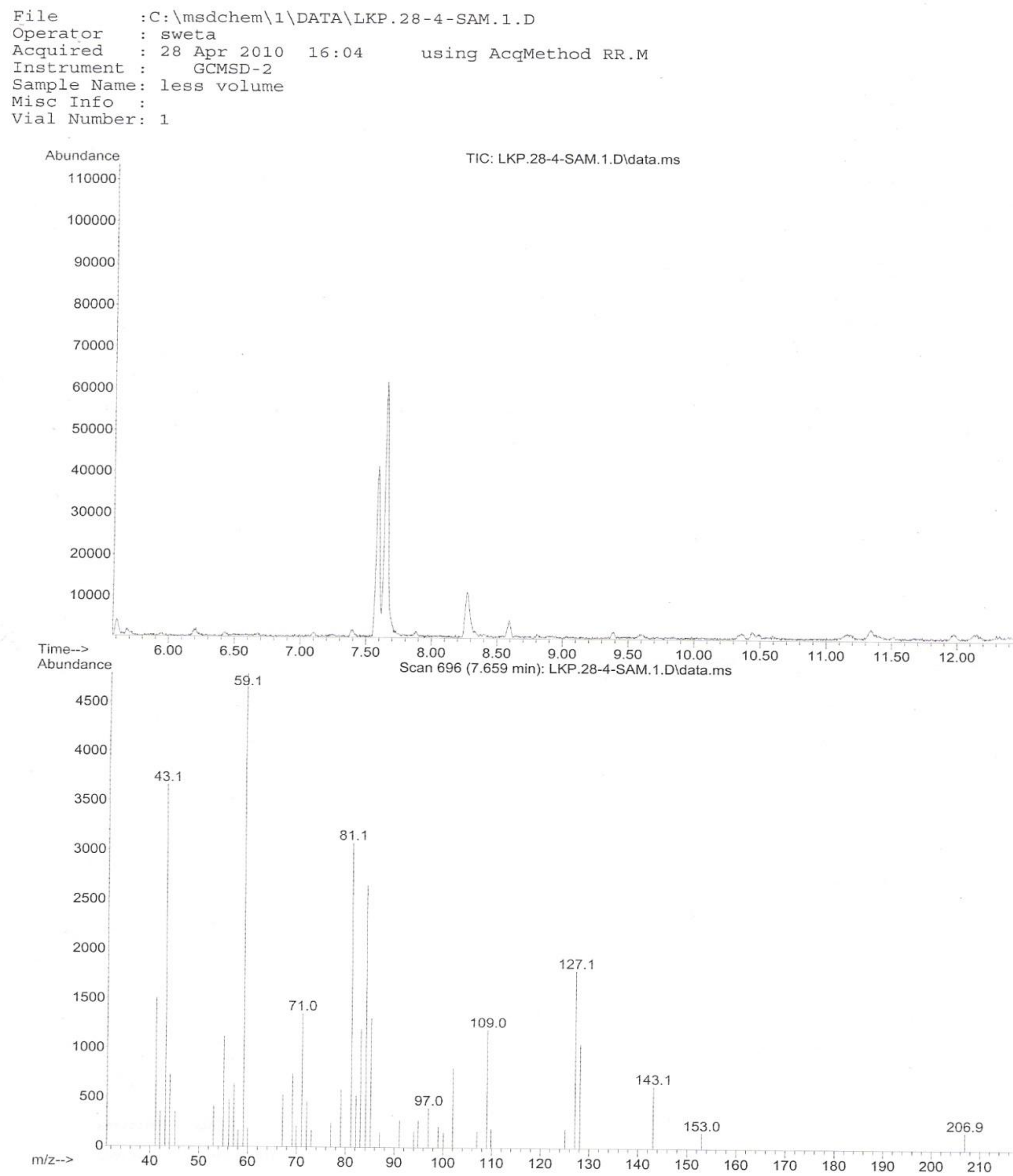


Fig 4.1 structure of Limonene



4.6 Graphs showing the quantitative and qualitative analysis of hydrosol extracted from hibiscus leaves.

The chemical composition of the essential oil of Hibiscus was examined by GC-MS. Fifty-eight components were characterized from this oil with (E)-phytol , (Z)-phytol, n-nonanal, benzene acetaldehyde, (E)-2-hexenal and 5-methylfurfural as the major constituents.

The oil was phytotoxic to lettuce and bent grass and had antifungal activity against Colletotrichum fragariae, Colletotrichum gloeosporioides, and Colletotrichum accutatum but exhibited little or no algicidal activity.

Of all the components the major component is (E)-phytol, which is having highest abundance w.r.t the mass/charge and retention time, as shown by the highest peaks in the graph.

Properties of E-phytol:

- IUPAC name : (E,7R,11R)-3,7,11,15-tetramethylhexadec-2-en-1-ol
- Molecular formula : $C_{20}H_4$
- Molar mass : 296.53 g/mol.
- Density : 0.85 g/cm³
- Boiling point : 202-204^{o}C

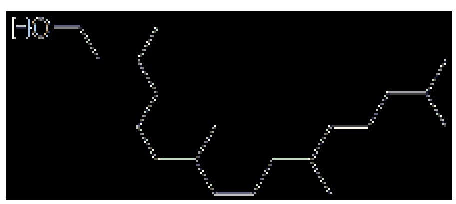


Fig 4.2 structure of E-phytol

CHAPTER 5

CONCLUSIONS

Finally we can conclude the following deductions from the above experimental and analysis part.

- Steam distillation method was found to be one of the promising techniques for the extraction of Essential Oil from plants as this process will preserve the original qualities of the plant.
- Steam distillation is a special type of distillation or a separation process for temperature sensitive materials like oils, resins, hydrocarbons, etc. which are insoluble in water and may decompose at their boiling point (which can be prevented using Steam Distillation method).
- Volume of Essential Oil increases with increase in temperature by keeping time of heating as constant.
- Volume of Essential Oil increases with increases in time of heating keeping the temperature constant.
- On decreasing the pressure we can extract Essential Oils of different plant materials at relatively less temperature and within less time of heating.
- Analysis using Gas Chromatography-Mass Spectrometer was found to be the best method to identify even the minor components of particular oil along with major components.
- Extraction of Essential Oils using Steam Distillation can be used on industrial scale to make various finished products which includes body oils, cosmetic lotions, baths, hair rinses, soaps, perfumes and room sprays.

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