A
Project Report on

EXTRACTION OF ESSENTIAL OIL AND ITS APPLICATIONS

In partial fulfillment of the requirements of
Bachelor of Technology (Chemical Engineering)

Submitted
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CERTIFICATE

This is to certify that that the work in this thesis report entitled "Extraction of Essential Oil and its applications" submitted by Virendra P.S.Rao and Diwaker Pandey in partial fulfillment of the requirements for the degree of Bachelor of Technology in Chemical Engineering Session 2003-2007 in the department of Chemical Engineering, National Institute of Technology Rourkela, Rourkela is an authentic work carried out by them 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.

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B.Tech. Final Yr. Chem. Engg.

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ABSTRACT

Essential oils are highly concentrated substances extracted from flowers, leaves, stems, roots, seeds, barks, resins, or fruit rinds. These oils are often used for their flavor and their therapeutic or odoriferous properties, in a wide selection of products such as foods, medicines, and cosmetics. Extraction of essential oils is one of the most time- and effort-consuming processes. The way in which oils are extracted from plants is important because some processes use solvents that can destroy the therapeutic properties. There are wide number of ways to extract the Essential oil but the quality never remains the same. Here we are using the "Steam Distillation" method for extraction which is the cheapest way for the extraction of Oils from the different parts of the plants. In this process steam is allowed to pass through the extraction chamber which contains plant matter. When steam passes through the herb material under pressure which softens the cells and allows the essential oil to escape in vapor form. The vapour allows to pass through condenser and oil is collected in separating funnel and separated. Composition data for Eucalyptus oil were studied and found the Eucalyptus oil can be used as cosolvent which results in depression of cloud point temperature.

CHAPTER-1

INTRODUCTION

Humankind has used plants for healing for many thousands of years, and it's from this tradition of that the use of aromatic plant compounds is medicine began. Oils were 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; Frankincense, Myrrh, Galbanum, Cinnamon, Cassia, Rosemary, Hyssop and Spikenard are noted for being used for anointing rituals and healing of the sick.

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. With the continual bombardment of viral, bacterial, parasitic and fungal contamination in our world, essential oils are a great benefit to help protect our bodies and homes from this onslaught of pathogens. Immune system systems need support and essential oils can give it.

Because of the enormous amount of raw product used to make wholly natural essential oils, lots of products on the market have been polluted with lower quality, commercial – grade oils or contain other chemical substances to reduce the cost or increase the profit margin – a fact not usually revealed on the label. This is why it is important to study the chemical composition of the volatile fraction once the essential oil is extracted. This fraction is characterized by the complexity in the separation of its components, which belong to various classes of compounds and which are present in a wide range of concentrations. Therefore it is complicated to establish a composition profile of essential oils. The gas chromatographic method (GC) is almost exclusively used for the qualitative analysis of volatiles. The analysis of essential oils was developed in parallel with the technological developments in GC, such as stationary phases, detection devices, etc. However, advances in instrumentation were not the only important factor in the development of analytical methods for essential oils in plants. Sample extraction and concentration were also improved. The most outstanding improvements in the determination of the composition of essential oils came from the introduction of tandem techniques involving prior/further chromatography or spectroscopy. The great amount of information on the application of GC and hyphenated techniques to essential oils has led to much research in this field.

CHAPTER-2

LITERATURE SURVEY

2.1. What are Essential Oils?

Essential oils are concentrated volatile aromatic compounds produced by plants - the easily evaporated essences that give plants their wonderful scents. Each of these complex precious liquids is extracted from a particular species of plant life. Each plant species originates in certain regions of the world, with particular environmental conditions and neighboring fauna and flora.

Essential oils are frequently referred to as the "life force" of plants. Unlike fatty oils, these "essential" oils are volatile, highly concentrated, substances extracted from flowers, leaves, stems, roots, seeds, bark, resin or fruit rinds. The amount of essential oils found in these plants can be anywhere from 0.01 percent to 10 percent of the total. That's why tons of plant material are required for just a few hundred pounds of oil. 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, medicines, and cosmetics. Beware of imitations. Essential oils cannot be substituted with synthetics. Only pure oils contain a full spectrum of compounds that cheap imitations simply cannot duplicate.

What do the Essential Oils do for the Plants?

Essential oils are extracted from oil 'sacs' in flowers, leaves, stems, roots, seeds, wood and bark. They differ significantly from the well-known vegetable, nut and seed oils which are made up of various fatty acids (essential oils are not). Essential oils are used by the plants in somewhat the same way they are by humans - they fight infection, contain hormone-like compounds, initiate cellular regeneration, and work as chemical defense against fungal, viral, and animal foes. Despite their foliar origins however, essential oils have a similar structure to some compounds found in blood and tissues, allowing them to be compatible with our own physiology.

How to Use Essential Oils?

The most effective way to use most essential oils is by external application or inhalation, though some can be very beneficial when taken internally. The use of essential oils include body oils, compresses, cosmetic lotions, baths, hair rinses, inhalation by steam, perfumes and room sprays. Essential oils are *very* potent - some will cause skin irritation or have other harmful effects if not used properly. Unless specifically noted, it is best to dilute all essential oils in a carrier of *base* oil like Almond, Jojoba or Apricot Kernel before applying to the skin - appropriate dilution is usually only 1 - 10% essential oil in carrier. For inhalation, a diffuser or oil lamp is effective for releasing essential oils into your environment - a very pleasant way of creating a particular atmosphere.

Table-1.1

Major Raw Material Used In Extraction Of Essential Oils:

Essential Oils are derived from various parts of Plants:

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

2.2. What is Aromatherapy???

"The treatment of anxiety or minor medical conditions by rubbing pleasant smelling natural oils into the skin or breathing in their smell."

It is the use of aromatic essential oils to benefit the body – in emotional and physical health and beauty. Science has discovered that our sense of smell plays a significant role in our overall health.

Many common essential oils have medicinal properties that have been applied in medicine since ancient times and are still widely used today. For example, many essential oils have antiseptic properties, though some are stronger than the other. In addition, many have an uplifting effect on the mind, though different essential oils have different properties.

The Beginnings of Modern Aromatherapy

The first modern-day distillation of essential oil was performed by the Persian philosopher Avicenna (980-1037 A.D.) who extracted the essence of rose petals through the 'enfleurage' process. His discovery and subsequent use of a wonderful perfume substance eventually lead him to write a book on the healing properties of essential oil of Rose.

Early in the 20th century a French Chemist, Rene-Maurice Gattefosse, began studying what he called " Aromatherapy ." After several burning his arm in a laboratory accident, he thrust the arm into the nearest liquid, which happened to be tub of Lavender Oil. Surprised by the quick healing that followed, Dr. Gattefosse spent the remainder of his life researching the value of Essential Oils. His success made aromatherapy popular, and it became well-known in Europe.

How Essential Oil Works in Aromatherapy?

An Essential Oil is inhaled and directly by the olfactory system to the limbic System of the Brain. In true, the 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.

By carefully choosing one or more oils, you can experience benefical effects promoting overall health - and even specific targets. Benefits depend upon the unique nature of each person's response to an aromatic stimulus.

2.3. Pharmacological Properties of Essential Oils

☆ **Antiseptics:**

Essential oils have antiseptic properties and are active against a wide range of bacteria as well as on antibio-resistant strains. Moreover, they are also known to be active against fungi and yeasts (Candida).The most common sources of essential oils used as antiseptics are: Cinnamon, Thyme; Clover; Eucalyptus; Culin savory; Lavender. Citral, geraniol, linalool and thymol are much more potent than phenol**.**

☆ **Expectorants and diuretics:**

When used externally, essential oils like (L'essence de terebenthine) increase microcirculation and provide a slight local anaesthetic 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.

☆ **Others**:

Cholagogue; anti-inflammatory; cicatrizing

2.4. Chemical Constituents of Essential Oils

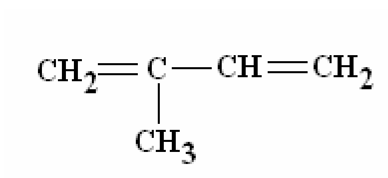
Pure essential oils are mixtures of more than 200 components, normally mixtures of terpenes or phenylpropanic derivatives, in which the chemical and structural differences between compounds are minimal. They can be essentially classified into two groups:

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

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

2.4.1. Hydrocarbon:

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



(Isoprene)

2.4.2. Terpenes:

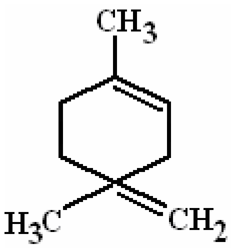
Generally have names ending in " **ene**."

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

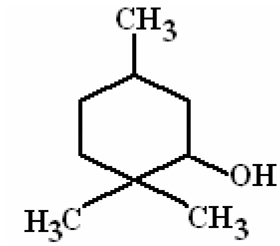
2.4.2.a. Monoterpenes [$C_{10}H_{16}$]

Properties: Analgesic, Bactericidal, Expectorant, and Stimulant.

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



(Limonene)



(Menthol)

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

Example:

- Camphene and pinene in cypress oil.
- Camphene, pinene and thujhene in black pepper.

2.4.2. b. Sesquiterpenes

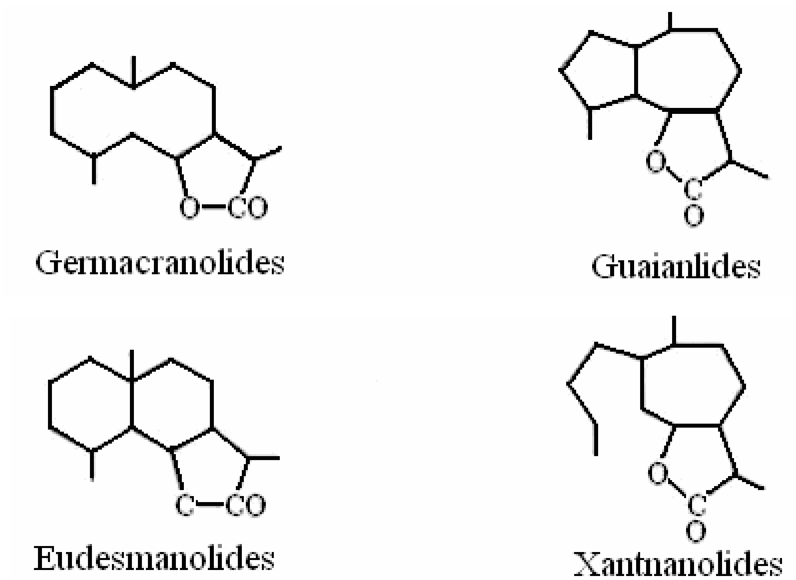
Properties: anti-inflammatory, anti-septic, analgesic, anti-allergic.

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

Sesquiterpene Lactones:

Over 500 compounds of this group are known; they are particularly characteristics of the Compositae 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.

Example:

- Farnesene in chamomile and lavender.
- Beta-caryophyllene in basil and black pepper.

2.4.2.c. 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).

Diterpenes have limited therapeutical importance and are used in certain sedatives (coughs) as well as in antispasmodics and antoxiolytics.

Example:

- Sclareol in clary sage is an example of a diterpene alcohol.

2.4.3. Alcohols

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

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

Example:

- linalool found in ylang-ylang and lavender.
- Geraniol in geranium and rose.
- Nerol in neroli.

2.4.4. Aldehydes:

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

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

Example:

- Citral in lemon.

- Lemongrass and lemon balm.
- Citronellal in lemongrass, lemon balm and citrus eucalyptus.

2.4.5. Acids

Properties: anti-inflammatory.

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

Example:

- Cinnamic and benzoic acid in benzoin.
- Citric and lactic.

2.4.6. Esters

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

Example:

- linlyl acetate in bergamot and lavender.
- Geranyl formate in geranium.

2.4.7. Ketones:

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

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

Example:

- fenchone in fennel, carvone in spearmint and dill
- Menthone in peppermint.

2.4.8. Lactones

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

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

2.5. Methods of Extracting Essential Oils

Early efforts at extraction used alcohol and a fermentation process. New methods of essential oils extraction are entering the mainstream of aromatherapy, offering new choices in oils never before available. With the new labels of CO_2 and Super Critical CO_2, along with the traditional ‘steam’ and ‘hydro’ distillations, 'absolutes', and 'cold pressing', a little education for the aromatherapy enthusiast can go a long way in essential oil selection. Is one process better than another? Does one produce nicer smelling oil, or one with greater aroma therapeutic value? It turns out that essential oil production, like winemaking, is an art form as well as a science. The way in which oils are extracted from plants is important because some processes use solvents that can destroy the therapeutic properties. Some plants, and particularly flowers, do not lend themselves to steam distilling. They are too delicate, or their fragrance and therapeutic essences cannot be completely released by water alone. These oils will be produced as 'absolutes' – and while not technically considered essential oils they can still be of therapeutic value. Jasmine oil and Rose oil in particular are delicate flowers whose oils are often found in 'absolute' form.

The value of the newer processing methods depends greatly on the experience of the distiller, as well as the intended application of the final product. Each method is important, and has its place in the making of aromatherapy-grade essential oils. Some of the few methods are available for extractions of essential oils are given below:

2.5.1. Maceration: Maceration actually creates more of an"infused oil" rather than an "essential oil". The plant matter is soaked in vegetable oil, heated and strained at which point it can be used for massage.

2.5.2. Cold Pressing: Cold pressing 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 liquid which will separate given time. Little, if any, 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. It is important to note that oils extracted using this method have a relatively short shelf life, so make or purchase only what you will be using within the next six months.

2.5.3. Solvent Extraction: A hydrocarbon solvent is added to the plant material to help dissolve the essential oil. When the solution is filtered and concentrated by distillation, a substance containing resin (resinoid), or a combination of wax and essential oil (known as concrete) remains. From the concentrate, pure alcohol is used to extract the oil. When the alcohol evaporates, the oil is left behind. 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.

2.5.4. Enfleurage: An intensive and traditional way 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 collected.

2.5.5. Hydro distillation: 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.

2.5.6. CO_2 & Super Critical CO_2 Extraction: The most modern technologies, Carbon Dioxide and Supercritical Carbon Dioxide extraction involve the use of carbon dioxide as the 'solvent' which carries the essential oil away from the raw plant material. The lower pressure CO_2 extraction involves chilling carbon dioxide to between 35 and 55 degrees F, and pumping it through the plant material at about 1000 psi. The carbon dioxide in this condition is condensed to a liquid. Supercritical CO_2 extraction (SCO_2) 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.

2.5.7. 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.

2.5.8. Steam Distillation: Most commonly, the essence is extracted from the plant using an technique called distillation. One type of distillation places the plants or flowers on a screen. Steam is passed through the area and becomes "charged" with the essence. The steam then passes through an area where it cools and condenses. This mixture of water and essential oil is separated and bottled. Since plants contain such a small amount of this precious oil, several hundred pounds may need to produce a single ounce.

2.6.Extraction of Essential Oils Using Steam distillation Method

Essential oils can be extracted using a variety of methods, although some are not commonly used today. Nowadays, a reputable distiller will try to preserve the original qualities of the plant, but the final therapeutic result is often not formed until after the extraction process. During extraction, the qualities of the oil change to give it more value - for example, chamazulene (characteristic of the pure blue colour of German Chamomile) is formed during the steam distillation process. Currently, the most popular method for extraction is steam distillation.

Many old-time distillers favor this method for most oils, and say that none of the newer methods produces better quality oils. 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 essential oils. As they are released, the tiny droplets of essential oil evaporate and, together with the steam molecules, travel through a tube into the still's condensation chamber. As the steam cools, it condenses into water. 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. 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 it's mild antiseptic and soothing properties, as well as it's pleasing floral aroma.

Essential oil isolated by steam distillation are different in composition to those naturally occurring in the oil bearing glands of plants, since the steam distillation conditions cause chemical reactions to occur which result in the formation of certain artificial chemicals, called artifacts. Some of these are considered beneficial e.g. the formation of chamazulene during the steam distillation of Chamomile oil; whilst others may not be e.g. the hydrolysis of linalyl acetate during the distillation of clary sage. Few, if any, essential oils are unscathed by the thermal conditions of steam distillation, but some distillation techniques can, in certain instances, be a measure less damaging than others (e.g. hydro diffusion – a sort of inverted steam distillation where steam is introduced at the top of the vegetable material-packed container, and oil and condensate issue from the bottom – can produce oils with higher ester contents i.e. less thermally induced hydrolysis).

A number of factors determine the final quality of a steam distilled essential oil. Aside from the plant material itself, 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. 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 odour, 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.

2.6.2. 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.

2.6.3. Uses of Essential Oils:

Essential oils are products of the secondary metabolism of plants, and generally are fragrant volatile materials consisting of complex mixtures of mono- and sesqui-terpene hydrocarbons, and oxygenated materials biogenically derived from them hence essential oils are used in flavourings, perfumes, in Aromatherapy, as insect & animal repellents, in pharmaceutical preparations, as anti-microbial agents and in many other ways.

2.6.4. Methods for Using Essential Oils:

Essential Oils are very powerful components of plants - they have the capability of being harmful if improperly used. Essential Oils can be very helpful for some cases, supportive in others, and have little to no effect in others. There are three traditional uses of Essential Oils in Aromatherapy.

2.6.4.a. Inhalation: Inhalation is often effective for mood-altering effects of essential oils; Rosemary for mental 'stimulation', Lavender for relaxation, etc. This is the direct effect of essential oil components on the limbic system. One may certainly blend essential oils in a diffuser or burner, adding a couple drops of each oil desired. Often a nice result can be had from mixing a brighter or sweeter oil (Rosemary, Basil, Orange) with one more earthy and grounding (Patchouli, Frankincense, Cedar).

2.6.4.b Topical Application: Perhaps even more common than inhalation, topical application is the preferred method of use for many essential oils. However, most essential oils require significant dilution as they can cause skin irritation. Lavender Oil and Chamomile Oil are two essential oils that can be applied 'neat' or without dilution; others, such as Cinnamon Oil and Oregano Oil should not be applied topically in most cases - they may be applied once highly diluted to the bottoms of the feet. A very small amount should be tested first. The interesting thing about topical application is that essential oils tend to pass through the skin fairly readily, as they are lipotropic (fat soluble) and their molecular structure is fairly small. Essential oils can pass into the bloodstream and surrounding tissues.

2.6.4.c Ingestion: Finally, some essential oils are ingested, usually either in water or in capsules. As this technique is rare, and not really considered effective in most cases.

2.7 Application of oil extracted from Eucalyptus

Eucalyptus oil as cosolvent

The recognition of petroleum shortages and limited reserves has prompted the need for renewable energy sources as fuels. Thus short term approaches involving the mixing of petrol with ethanol have become increasingly attractive.

However, one well-documented problem associated with the use of ethanol blends is their affinity of water. Ethanol is completely miscible with all petrol formulations, but the mixtures are subjected to phase separation in the presence of small amount of water. This phenomenon is unacceptable in climates in which temperature drop below freezing point. Anhydrous ethanol may be employed to minimize the problem but its cost of production has hindered its use in many countries. The application of aqueous ethanol as a fuel extender remains an attractive proposition in countries where temperatures below 0ºC are rare. Since it is both difficult and expensive to prevent water entry or to install water absorbing devices, a better approach would be to introduce a low cost stabilizer or cosolvent.

The addition of eucalyptus oil as a cosolvent resulted in a significant depression of the cloud point temperature. Eucalyptus oil and its main component, 1,8-Cineole have been investigated as potential cosolvents. This study formed one aspect of utilizing solar energy stored in plants as liquid fuel components. The use of cosolvents to increase the mutual miscibility of ethanol-petrol-water blends is the most immediately feasible modification for controlling the phenomena of phase separation at low degree of temperature. The phase stability of water-ethanol-1,8-Cineol ternary systems, which are potentially valuable liquid fuels from renewable sources.

Materials required: nhydrous ethanol, gent grade anhydrous ethanol of boiling point 78.1 ºC and density 0.785 g/cc. 1,8-Cineole was obtained by fractionation of leaf oils of Eucalyptus species. Water: the water double distilled from $KMnO_4$.

The Co-existence Curve and tie line measurements

The binodal curves were determined at a constant temperature by isothermal titration. This method consists of titrating mixtures of known compositions of two components with a third component to the cloud point, yielding a point in the ternary diagram that lays on the liquid-liquid equilibrium curve

In most of the mixtures titrated, the end points determined with the use of the cloud point apparatus were sharp .The addition of a small drop of titrant resulted in a strong increase in turbidity of the mixture. All compositions were made up by weight and were accurate reproducible to better than 0.3 ºC.

After the above mixtures were titrated to the cloud point, they were suspended in a constant temperature bath at the desired temperature and their refractive indices were measured to the nearest 0.0001 unit at a given temperature. By plotting weight percentage of each component against refractive index in the equilibrium mixture the refractive index- composition curve was constructed.

The tie-lines can be determined by the following procedure. A number of mixtures having compositions within the two phase region were prepared. They were shaken thoroughly in temperature – controlled cells for 10 hours to allow separation into two phases. Once equilibrium had been achieved, samples of both phases were withdrawn and their refractive indices were measured. By employing the refractive index – composition calibration curve the composition of both phases could be determined. This method of obtaining the tie lines was essentially described by Wasburn. Each tie-line was checked from the from the graph to ensure that it passed through the composition of the overall mixture.

Solubility of 1,8-Cineole in water:

An open-hlask method was employed for the solubility determination. The solubility was measured over the temperature range of 4 - 50 ºC and the amount of 1,8-Cineole in water at equilibrium was determined with a gas chromatograph, while density was also determined.

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.

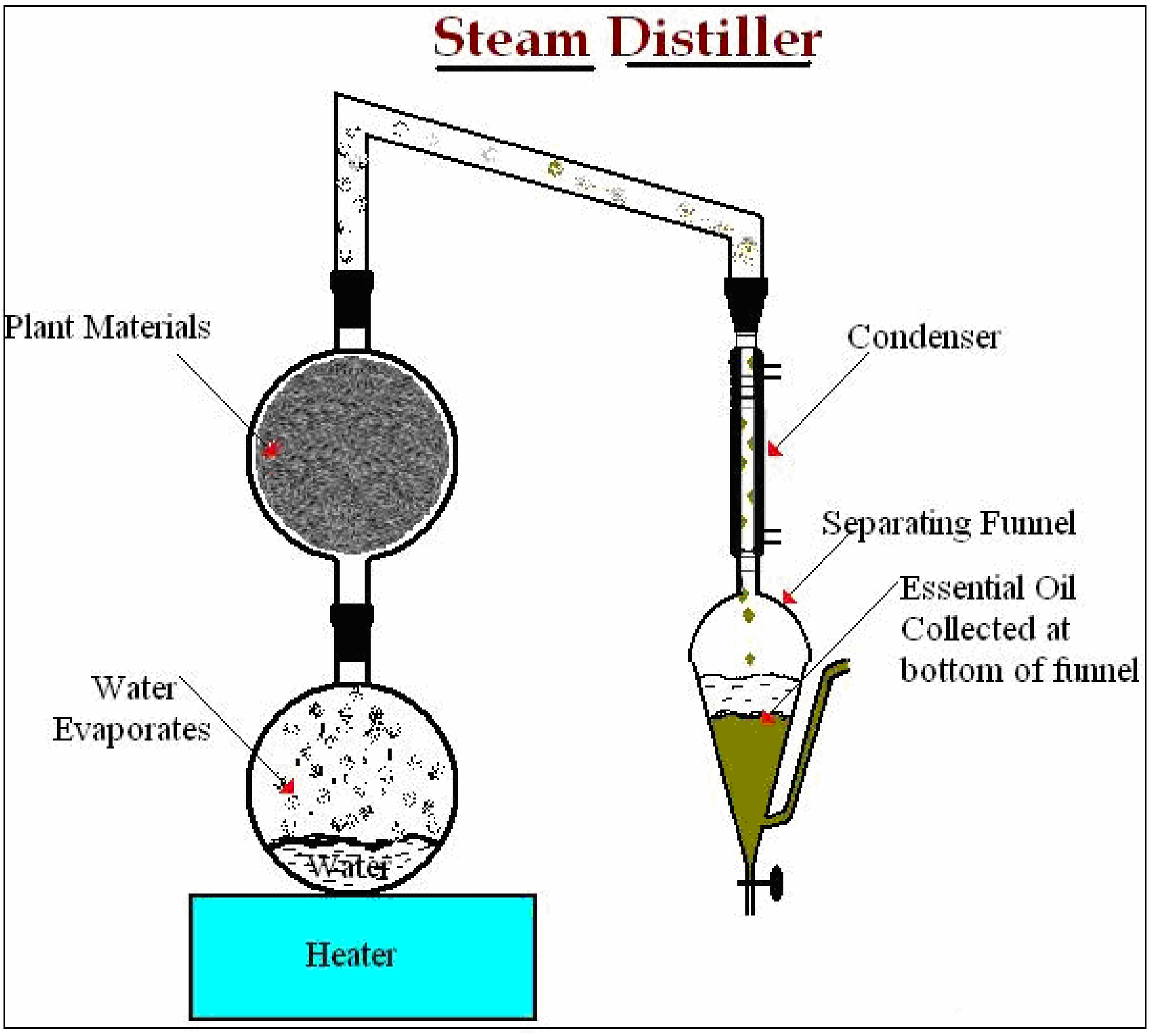


Figure-3.1: Flow sheet for Extraction of Essential oils using Steam Distillation Method

Figure-3.2 Essential Oil Steam Distiller

3.2 Experimental Procedure:

Fresh leaves were 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 appartus 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.

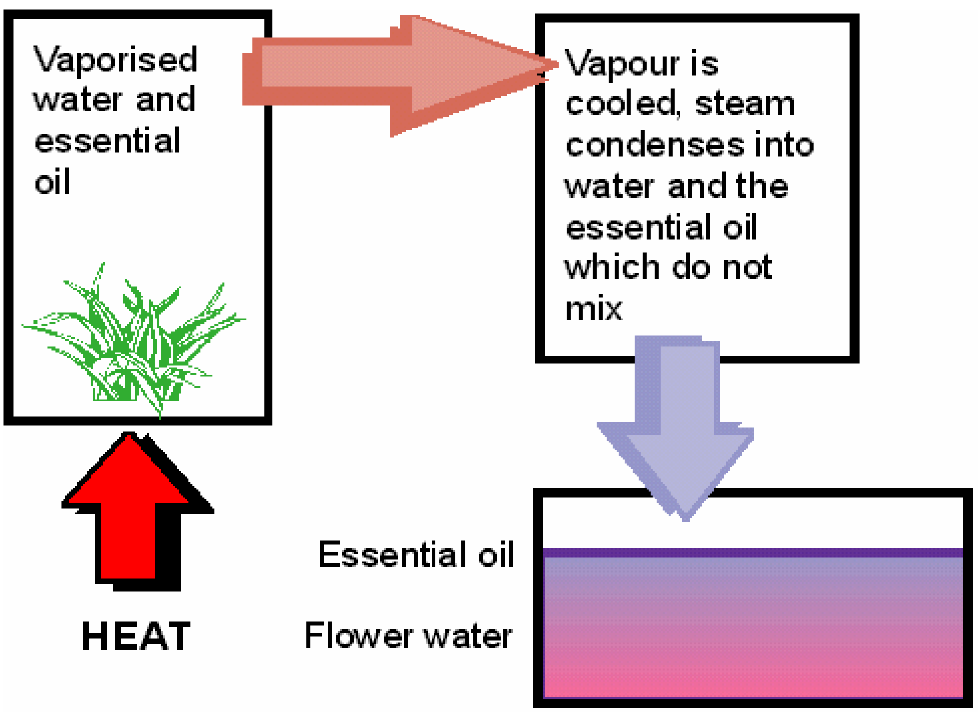


Figure-3.3: Flow diagram for the Steam Distillation

3.2.1Precaution:

- The temperature of the extraction chamber cannot be too high, lest some components of the oil be altered or destroyed. The same is true of the chamber's pressure. Higher temperatures and pressures result in a 'harsh' aroma – more chemical than floral – and lessen the oil's therapeutic effects.

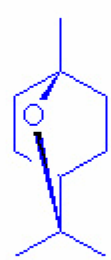
- The extraction period must be allowed to continue for a certain period of time in order to flush all the oil's components from the plant, as some are released more quickly than others.

3.3 Experimental Observation

Table-3.1 Physical and Chemical Properties of Eucalyptus Oil

Colour	Pale yellow liquid
State	Liquid-oil
Odour	Camphoraceous odour
Taste	pungent and cooling taste.
Boiling Point of Cineole (Eucalyptol)	176 ^{0}C to 177 ^{0}C;
Density of oil	0.921 to 0.923
Solubility	1. Insoluble in water; 2. Miscible in alcohol having high concentration or in anhydrous alcohol; 3. Miscible in oil; fats; paraffins; ether ; chloroform and glacial acetic acid.

Major Components: 1, 8-Cineole.



1,8-cineole

Chemical Name: 1, 3, 3- Trimethyl-2- Oxabicyclo [2. 2. 2] – octane or 1, 8- epoxy-p-methane.

Molecular Formula of Cineole: $C_{10}H_{18}O$

Molecular Weight: 154.25

Precaution of Storing: Products containing eucalyptus oil should be stored at a temperature not exceeding 25°C in well filled containers. It should be protect from light. Liquid products containing eucalyptus oil are best stored in child resistant containers.

3.4. Analysis of Essential Oils

Gas Chromatography is one of the best techniques we have to identify the constituents of an essential oil. When properly used it can easily detect and identify major components of essential oils, and give us some indications of the quality and authenticity of the oil. The technique does have limitations however. Many minor components of essential oils (<0.01%) do not register on GC detector systems. The separation of essential oil components is usually carried out by GC with fused-silica capillary columns. The properties and conditions of columns used are variable, depending on the polarity of the components to be separated. It is advantageous to use a more selective phase for a given separation as the overlapping of peaks in the final chromatogram is often a significant drawback of chromatographic techniques in natural samples. The discovery of chiral phases (mostly based on cyclodextrin derivatives) allows the resolution of enantiomers of volatile components. These phases can give different elution sequences for a polarity range and provide a distinct advantage in identification because of large changes in solute relative retention times. The information obtained from high-resolution GC analysis of the volatile fraction of essential oils must be sufficient to determine whether the product is genuine or not. If the product is adulterated, the kind and level of adulteration must be detected. Therefore a selective and accurate separation is absolutely necessary in the case of industrial analysis. On the other hand, GC sometimes permits the separation and further identification of some components of the nonvolatile residue as well.

3.4.1 Major Component Of Eucalyptus Oil

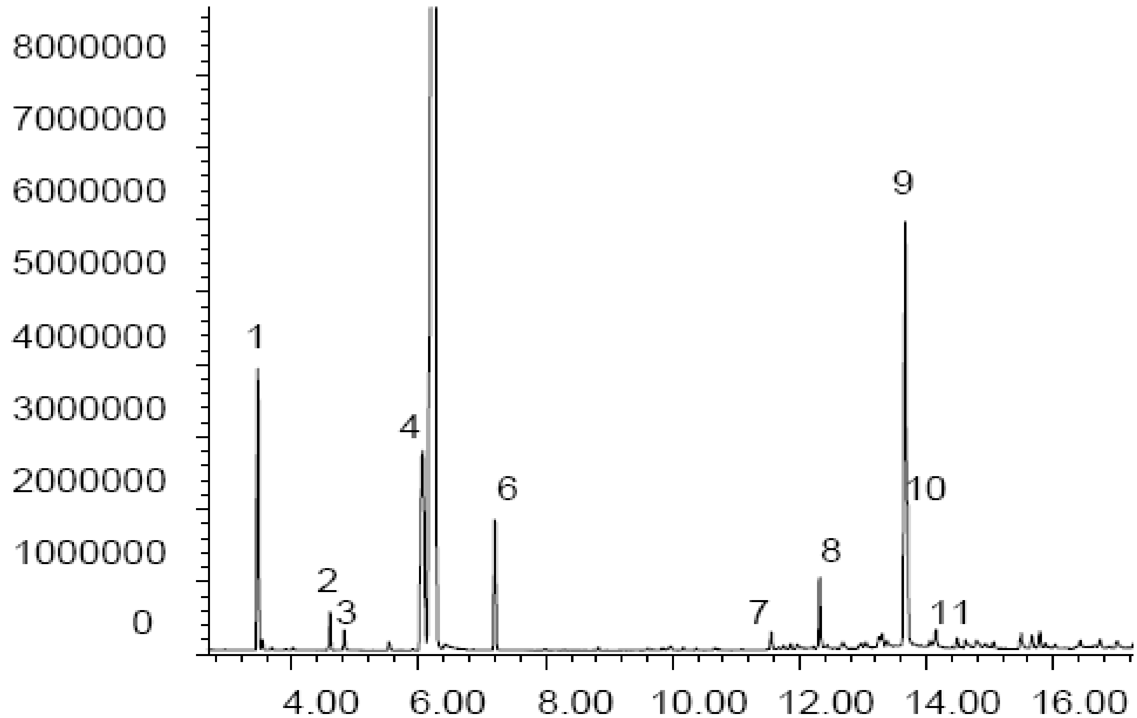


Figure-3.4 Chromatographic Data of Eucalyptus Oil

Componets	%age
α-Pinene	13.85
β-Pinene	0.78
Sabinene	---
Limonene	4.31
1,8-Cineole	67.67
p-Cymene	13.13
Linalool L	---
Terpinen-4-ol	---
α-Terpineol	45.68
α-Terpinenyle Acetate	---
d-Carvone	

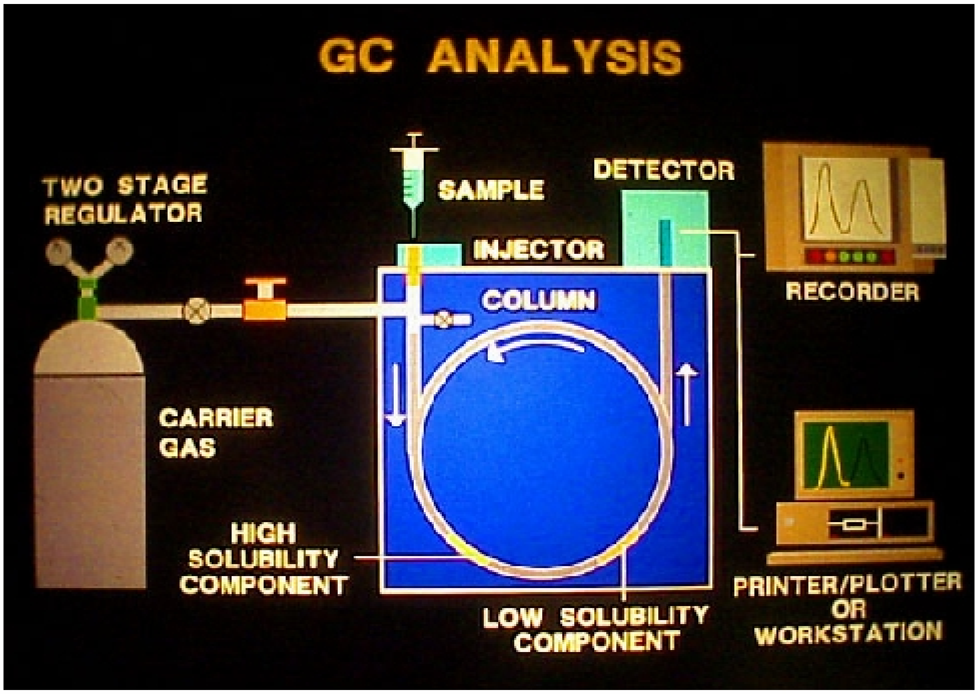


Figure-3.5 : Experimental Setup of Gas Chromatography

3.4.2 Experimental Setup:

A gas chromatograph consists of :

- A supply of a carrier gas (N_2) from a high pressure cylinder having a pressure regulator and flow meters.
- A sample injection system.
- The separation column made from variety of materials including glass, copper, stainless steel, cupro-nickel or organic polymer (Teflon). Packed beds are used.
- The detector is situated at the exist of the separation column which senses and measures the small amount of the separated components present in the carrier-gas leaving the column. Commonly used detectors are thermal conductivity detectors, Wheatstone bridge circuit, and flame ionization detector.
- The recorder is fed by the output of the detector.
- Thermo stated compartment for the column and detector.

CHAPTER-4

RESULT AND DISCUSSION

4.1 Inferences:

The amount of essential oils obtained by steam distillation, contains more than 2 ml of essential oil per 150 gms of oven dried Euclayptus leaves, where as Essential oil obtained from the Bay leaves contains very small amount of oil in comparison to the oil obtained from Eucalyptus. 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. The oil obtained is pale yellow in colour , where as oil obtained from Bay leaves is colourless.

Fresh, 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. As the time increases the steam production increases which extract the oil from the leaves at the boiling point of the water. After 4-5 hours of operation the extraction of oil ceased.

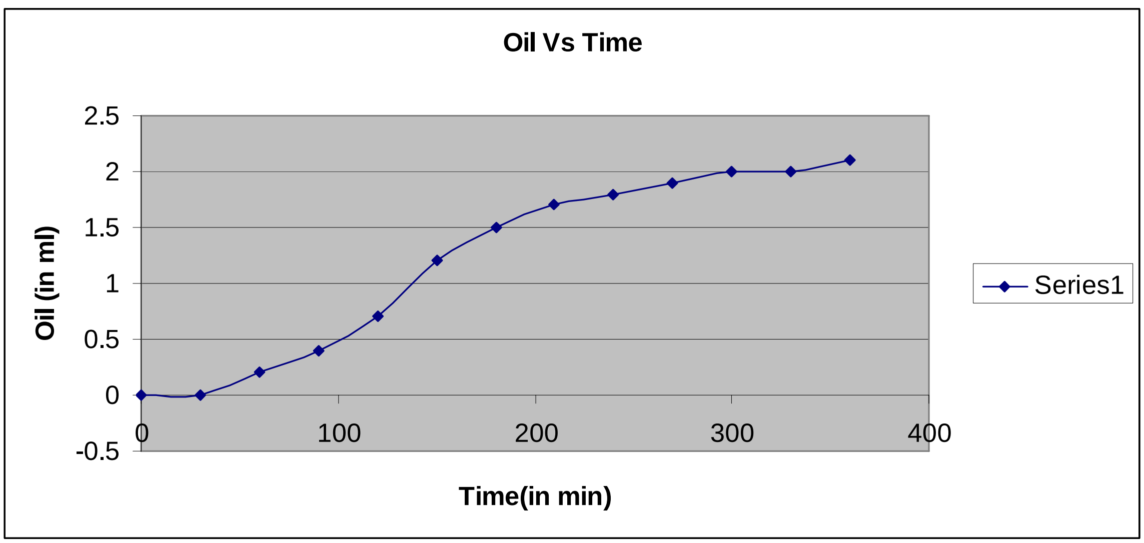


Figure 4.1 : Eucalyptus Oil obtained Vs Time

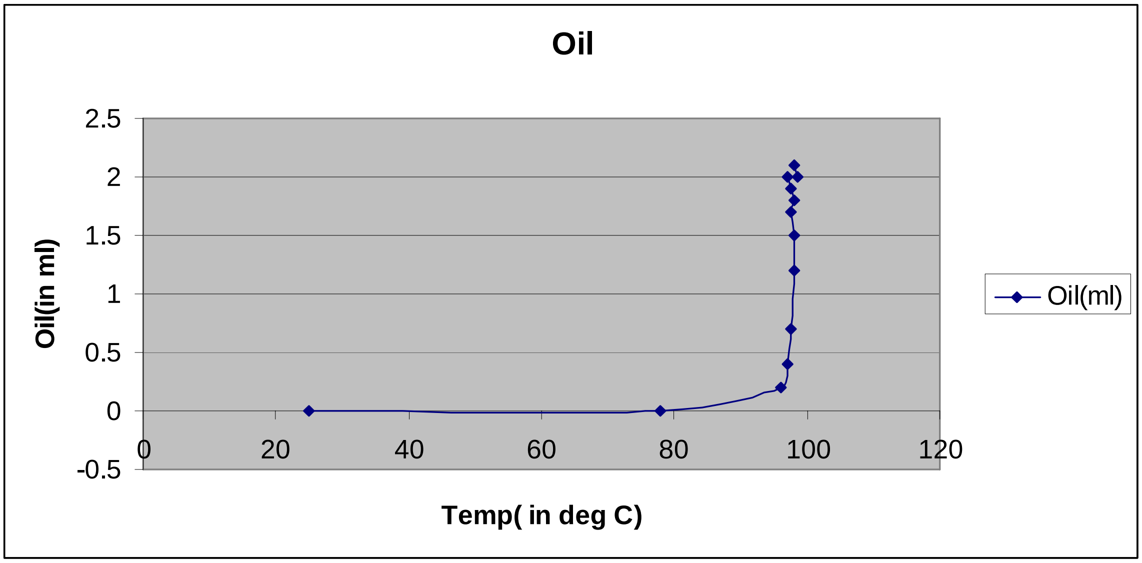


Figure 4.2: Eucalyptus Oil obtained Vs Temperature of the water

CHAPTER-5

CONCLUSION

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 as been carried out for the extraction of oil from Eucalyptus which have 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.

Future Work

Oil contained in Eucalyptus is higher and can be easily extracted. The Eucalyptus oil contains more than 65% of 1,8 cineole. Hence the incorporation of small amount of Eucalyptus oil as cosolvent in aqueous ethanol and petrol mixture improved the water tolerance of the system. Therefore the work can be extended for the study of ternary phase equilibrium of the water ethanol and 1,8-cineole or Eucalyptus oil.

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