

**EXTRACTION AND IDENTIFICATION OF ESSENTIAL OILS
FROM OLEORESINS**

Submitted in partial fulfilment of the requirements for the award of

Master of Science in CHEMISTRY

by

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DEPARTMENT OF CHEMISTRY

SATHYABAMA

INSTITUTE OF SCIENCE AND TECHNOLOGY

(DEEMED TO BE UNIVERSITY)

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APRIL-2022



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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **Ms. I. GRESSHMA DAS (40910001)** who carried out the project entitled “**EXTRACTION AND IDENTIFICATION OF ESSENTIAL OILS FROM OLEORESINS**” under my / our supervision.

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To whom so ever it may concern

This is to certify that Ms. I. Greeshma Das, from Sathyabhama institute of science and technology is perceiving her internship, during this she has completed her project work titled "Extraction and identification of essential oils from Oleoresins" at M/s Symrise Private Ltd.

We wish her all the best.


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DECLARATION

I **MISS.I.GREESHMA DAS** student of **SATHYABAMA INSTITUTE OF SCIENCE AND TECHNOLOGY** hereby declare that the Project Report entitled **“EXTRACTION AND IDENTIFICATION OF ESSENTIAL OILS FROM OLEORESINS”** done by me under the guidance of **Dr.J.KARTHIKEYAN, M.Sc.,M.Phil., Ph.D.(Internal)** Head of the Department, Department of Chemistry, Sathyabama Institute of Science and Technology, Chennai., and **Dr.T.HARIKRISHNA, M.Sc., Ph.D.(External)** Head of the Department, Quality Control, Symrise PVT Ltd, Chennai., is submitted in partial fulfilment of the requirements for the award of **Master of Science degree in CHEMISTRY.**

DATE: 11.05.2022

PLACE: Chennai.

I.GREESHMA DAS

SIGNATURE OF THE CANDIDATE

ACKNOWLEDGEMENT

I am pleased to acknowledge my sincere thanks to Board of Management of **SATHYABAMA** for their kind encouragement in doing this project and for completing it successfully. I am grateful to them.

I convey my thanks to **Dr. J KARTHIKEYAN, Head of the Department, Department of Chemistry** for providing me necessary support and details at the right time during the progressive reviews.

I would like to express my sincere and deep sense of gratitude to my Project Guide's **Dr.J.KARTHIKEYAN, M.Sc.,M.Phil., Ph.D.(Internal)** and **Dr.T.HARIKRISHNA, M.Sc.,Ph.D.(External) Head of the Department, Quality Control, Symrise PVT Ltd, Chennai** for their valuable guidance, suggestions and constant encouragement paved way for the successful completion of my project work.

I wish to express my thanks to all Teaching and Non-teaching staff members of the Department of Chemistry who were helpful in many ways for the completion of the project.

I.GREESHMA DAS

ABSTRACT

Essentials oils are highly concentrated substances extracted from flowers, leaves, stems, roots, seeds, barks, resinoids, and oleoresins. These oils are often used for their flavor and odor properties in a wide selection of products such as foods, medicines, and cosmetics. Rather than being synthetically manufactured in labs, essential oils are extracted from plant materials through removal methods that are suited to the specific plant part containing the oils. Essential oils are the liquids that are isolated from plants when introduced to solvents and they are liquefied versions of the plants. The method of extraction of affects essential oil quality by way of pressure and temperatures applied. There is a wide number of ways to extract the essential oils, but here we are using “**Hydro-Distillation**”. Hydro-Distillation is a well-known and popular method for extracting essential oils from oleoresins. Additionally, it represents an environmentally friendly process. In the current work, **Ginger Oleoresin, Pepper Black Oleoresin, and Cumin Seed Oleoresin** are used to extract essential oils using **Dean-Stark Apparatus** using water as extracting solvent. In this process, heat is allowed to pass through the extraction chamber which contains oleoresin. GC-FID(quantitative) and GC-MS(qualitative) techniques are used to analyze the extracted oil.

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LIST OF ABBREVIATIONS

GC - Gas Chromatography

GC-MS - Gas Chromatography - Mass Spectrometer

CHAPTER – 1

INTRODUCTION

1.1.What are Essential Oils?

Essential oils are organic, concentrated, highly volatile, the hydrophobic liquid that naturally occurs within and is secreted by tiny structures located in a very plant's various parts – the seeds, grasses, roots, barks, stems, leaves, fruits, flowers, resins, zest and wood of plants. They're also stated as volatile oils, ethereal oils, or aetherolea. These oils got the name “essential,” because they were believed to capture a plant's essence, that's its odor and flavor. the character of an oil depends on the plant itself and on the botanical family and species to which it belongs. an important oil gets its name from the plant from which it's derived. as an example, the volatile oil from the Ginger Oleoresin would be called Ginger Oil.

The oils contained within the plants are aromatic, lending plants their distinctive fragrance while also promoting their self-protection and pollination; it's likely that oils from a plant's wood, leaves, and roots help the plant guard itself against attacks from parasites and animals and permit them to adapt to their environments, which are sometimes harsh. As already mentioned, the oil may be a volatile hydrocarbon, which is to mention it's a molecule that rapidly changes states from solid or liquid to gaseous at temperature. the precise ratio of the constituents in a necessary oil gives it specific wellness-enhancing and therapeutic qualities.

1.2.History of Essential Oils

Because of the worldwide usage of essential oils for hundreds of years, it's difficult to pinpoint exactly which culture began using them first, but the primary records of essential oils allegedly come from ancient India, Persia, and Egypt. Greece and Rome also engaged within the widespread trade of aromatic oils and ointments to Eastern countries. Each culture had various uses for them starting from health treatments to spiritual practices. Their herbal preparations included incense, perfume, clothing and fabric fresheners, medicine like pills, powders, and suppositories, ointments, scented baths, and

aromatherapy massages. In many cultures, aromatic oils were believed to form a union with the gods and were held in such high regard that they were used only by a pick group of privileged people, like priests.

Arabs were the primary to develop the technique of plant distillation to extract essential oils. They were able to replace the fatty oils that were used as solvents for extraction with a replacement solvent that they created by distilling ethyl alcohol from fermented sugar. During the center Ages, this information of the distillation technique spread to Europe and its pharmacies specialized in distilled products

Table-1.1

Major Raw Materials 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 Spearment Tea Tree Wintergreen Thyme	Chamomile Clary Sage Clove Geranium Hyssop Jasmine Lavender Manuka Marjoram Orange Rose Ylang-Ylang	Bergamot Grapefruit Lemon Lime Orange Tangerine	Almond Anise Celery Cumin Nutmeg Oil Pepper	Camphor Cedar Rosewood Sandalwood
Berries	Bark	Resins	Rhizome	Root
Allspice Juniper	Cassia Cinnamon	Frankincense Myrrh	Ginger	Valerian

1.3.Terminology

Essential Oils – An Essential Oil is a volatile material, derived by a physical process from odorous plant material of a single botanical form and spices with which agrees in name and odor.

Absolute – An Absolute is a prepared perfume material. The alcohol-soluble fraction of a resinoid is called an absolute. They are obtained by alcohol-extraction of concretes or other hydrocarbon types of extracts or from fat extracts of plant material.

Concrete –Concrete is a prepared perfumery material. Concretes are extracted from non-resinous or low-resinous materials. The natural raw materials from which concretes are prepared are almost exclusively of vegetable origin example bark, flower, herb, leaf, root, etc.

Balsam – A Balsam is a natural raw material exuded from a tree or a plant; the balsam may be either a physiological or a pathological product of the plant. Balsams are resinous masses, semi-solid materials or viscous liquids, insoluble in water, completely soluble in ethanol, but only partially soluble in hydrocarbons.

Extract – Extracts are prepared materials. The term Extract is used for perfume materials, flavour materials, pharmaceutical products, and many other commercial products.

Resin – A Resin is either a natural or a prepared product. Natural Resins are exudations from trees or plants and they are formed in nature by the oxidations of terpenes. Prepared resins are oleo-resins from which the essential oil has been removed.

Oleo-resins – An Oleo-resin is neither a natural nor prepared material. As the name indicates, oleo-resin consists entirely or mainly of essential oil and resin. Natural oleo-resins are exudations from tree trunks, barks, etc. Prepared oleo-resins are extracted from botanical drugs with solvents that can extract oil and resinous matter from the botanical, yielding the oleo-resin as evaporation residue.

Table – 1.2

Oleoresins used in the Extraction of Essential Oils

Name of Oleoresins	Extracted From	Sensory	Oil Content (Theoretical)
Ginger Oleoresin	Ginger Rhizomes (Zingiber Officinale)	Pungent and Characteristic aroma of fresh ginger	30.3%
Pepper Black Oleoresin	Ground–dried berries of Piper Nigrum L	Pungent and Characteristic aroma of fresh black pepper	21.2%
Cumin Seed Oleoresin	Ground roasted seeds of Cuminum Cyminum	The characteristic aroma of Cumin, with a bitter undertone	15.1%

1.4.Composition of Essential Oils

Essential oils are comprised of a posh mixture of constituents, one oil sometimes containing many them. the particular ratio of the constituents gives the oil its specific wellness-enhancing and therapeutic qualities. the foremost commonly found classes of volatile oil constituents include: Monoterpenes, Sesquiterpenes, Diterpenes, Alcohols, Phenols, Aldehydes, Ketones, Esters, and Oxides. the majority essential oils are anti-septic and lots of even have anti-fungal, anti-viral, and anti-bacterial activities.

The looks of essential oils ranges from being colorless to be any color of the rainbow, and sometimes the colour of the oil points to its therapeutic qualities. as an example, being a blue color, Chamomile Oil is beneficial for aromatherapy massage on an individual experiencing “red hot” emotions, because the blue represents its classic “cooling and soothing” effect and counteracts any negative physical and psychological feelings. Oils like Patchouli, Orange, and Lemongrass are amber or yellow in color and their bright happy colors can easily

help someone determine what they're best used for – as mood boosters! normally, Younger plants yield more essential oils than older plants. Sometimes their colors are a results of the extraction method while at other times the colour of the stuff affects the colour of the ultimate product. While Chamomile isn't blue, it contains a component called Chamazulene, which turns the oil an inky blue color during the distillation process.

Table – 1.3
Chemical Composition of Essential Oils

Name of Essential Oils	Chemical Composition
Ginger Oil	The Ginger Oil contains a mixture of constituents such as Monoterpenes , namely Phellandrene, Camphene, Cineole, Linalool, Limonene, Citral, Geraniol, Citronellol, Borneol and Sesquiterpenes , namely α -Zingiberene, α -Curcumene, β -Bisabolene, β -Sesquiphellandrene, Zingiberol and Zingiberenol
Pepper Black Oil	The major constituent of Black Pepper oil was trans-Caryophyllene , Sabinene, Limonene .
Cumin Seed Oil	The major fatty acids were Cuminic aldehyde , Terpinyl isopropyl ether, Cuminic nitrile , Cymene , Myrcene.

1.5.Methods of Extracting Essential Oils

A single drop of oil will be potent enough to possess powerful health benefits. Microdroplets of oils are stored within a plant's glands. After these droplets are diffused through the gland walls, they are displayed over the plant's surface then evaporate, filling the air with a nice aroma. Plants with the strongest scents are usually found in tropical regions where there are higher temperatures, which causes plants to supply more biogenic volatile organic compounds while also extending the growing period throughout which their fragrant compounds are generated.

The subsequent methods are the ways to extract essential oils: Enfleurage, Expression, Steam Distillation, Solvent Extraction, Carbon dioxide Extraction, Fractional Distillation and Percolation, Phytonic Process, Maceration, Mechanical Pressing, and Distillation.

Enfleurage - Delicate plant parts like flowers, roots, and leaves are soaked in fatty oils to extract their essential oils.

Cold Pressing - A rotating automaton with spikes punctures the fruit rind to release its essential oils. This method is additionally called "Expression". It's specifically meant for citrus essential oils (Lemon, Bergamot, Orange, etc).

Steam Distillation - A current of steam is injected into the still containing botanical material, usually at high pressures and temperatures (eg., Sandalwood).

Solvent Extraction - In an exceedingly one amongst one in every of the components of botanical material dissolves in a particular liquid (solvent) and therefore the non-volatile components, like waxes and pigments, are separated/removed by filtration. This method is additionally called "Liquid-liquid Extraction." It encompasses Enfleurage, Maceration, and greenhouse gas Extraction.

CO₂ Extraction - Pressurized CO₂ turns into a “supercritical” liquid that's pumped into a chamber containing stuff. Despite being a gas it's liquid properties, which allows it to act as a solvent that pulls out the plant matter's essential oils.

Hydro-Diffusion Extraction - This method is comparable to steam distillation except that the steam enters through the highest of the chamber instead of the underside. The stuff lies on top of a grill, therefore the steam “percolates” down through the material, very similar to coffee passes through a filter. This method is additionally called "Percolation."

Phytonic Process - This method uses non-chlorofluorocarbons (non-CFCs) as a solvent. The oils produced are called Phytols. The extraction occurs at or below temperature, which suggests the oil isn't degraded by high temperatures. These oils are pure and as near the natural plant properties as possible. This method is additionally called "Florasol Extraction."

Maceration -A solvent (Menstruum) is added to cut/ground/crushed stuff and also the mixture is allowed to stand for a specific period of your time. The liquid is strained and also the solid residue is pressed for any remaining liquid. Both strained and expressed liquids are mixed then filtered.

Hydro-Distillation - Material is submerged in water that's heated until the material becomes soft. The oil vapors rise, enter a condensation chamber, and funky off. Here, the steam becomes water again, but the vapors become the oil. The oil is then separated from the water component, which becomes the “floral water” (hydrosol).

1.6.Extraction of Essential Oils using Hydro-Distillation method

Hydro Distillation is one among the oldest methods used for the extraction of oils from different parts of the plants like wood and flower petals. it's an extended history of application in various ancient civilizations of Persia, India, China, and Egypt. This process is an alternate to the steam distillation method

when pure oil can not be captured by the plants using traditional methods. During this method, water and oil are exclusively separated through the condensation.

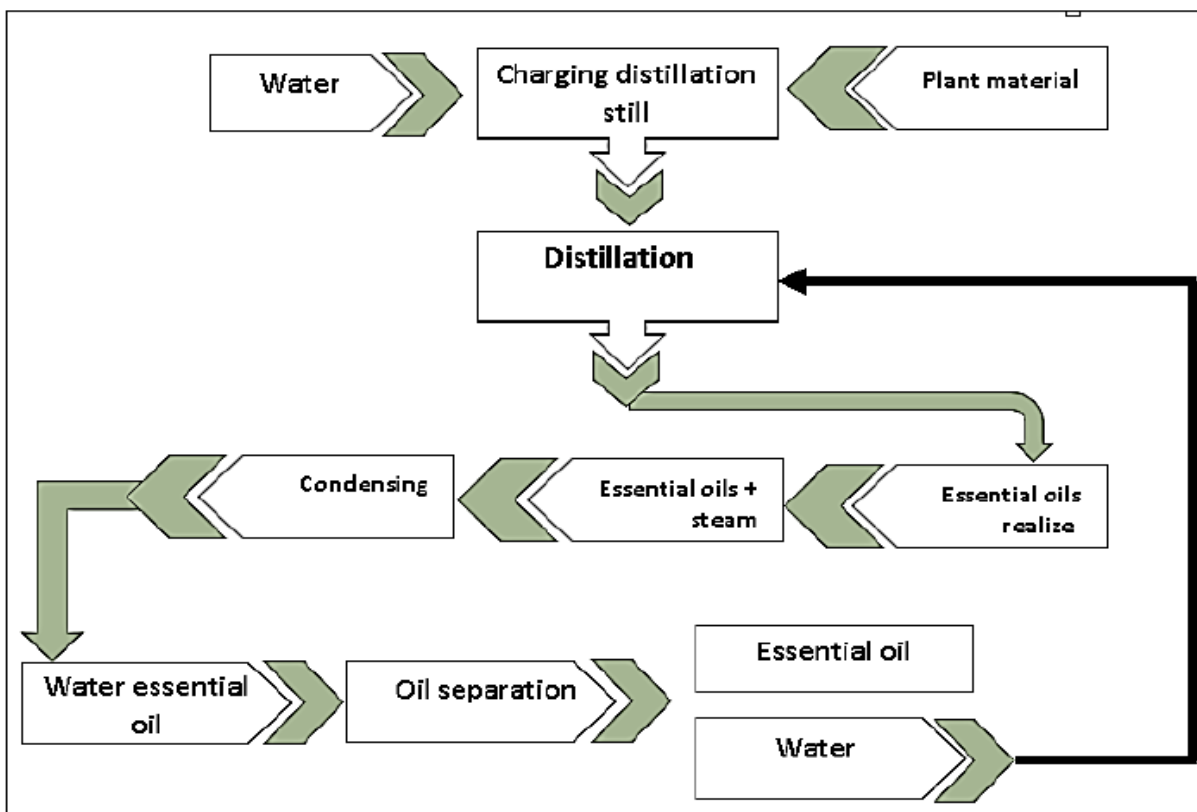


Figure:1.1 Flow Diagram of Hydro-Distillation Method

While extracting oil through this process, the stuff is soaked within the water that's placed over a container over heat. The container material should be manufactured using copper, stainless-steel, or glass together with a condensing unit attached to a receiving flask. During the boiling process, the steam, furthermore as oil vapor, is captured within the condensing apparatus. Excessive water from the resultant mixture is formed to dispense out through a gap within the condensing apparatus. the ultimate product hence obtained within the receiving flask contains only the distillate. Moreover, the excessive water is stated as 'hydrosol' that includes a significant amount of plant essence may also be employed in various cases. for instance, rose hydrosol or perfume is usually used for its pleasing floral aroma, mild antiseptic, and soothing properties.□It is widely utilized in the planet.

1.6.1. Advantages of hydro-distillation

- It is widely used in the world.
- The water distillation method is inexpensive and easy to construct. It is proper for field operation.
- Boiling water causes motion of plant into the distilling flask, which leads to improved heat transfer.
- There is direct contact between plants and boiling water.

1.7. Classification of Essential Oils (based on aroma)

Essential Oil aromas have the potential to boost the mind, body, and atmosphere. While it are often confusing to create a variety when faced with the vast number of volatile oil options because of their limitless benefits, it may be easier to settle on by narrowing down the choices supported the characteristics of their "aroma families." Each volatile oil aroma family exhibits a primary aromatic trait by which it's easily recognized (citrusy, floral, minty, etc.) and a corresponding effect (calming, grounding, cooling, energizing, clarifying, etc), which may help to readily identify the perfect application for it.

TABLE-1.4 Classification of Essential Oils

AROMA FAMILY	ESSENTIAL OILS
CITRUS These light oils often have fruity scents that are characteristic of the rinds from which they are extracted.	Lemon, Orange, Grapefruit Bergamot, Lime, Tangerine, Citronella, Lemongrass, Mandarin, Litsea Cubeba, Tagetes

FLORAL These scents are often reminiscent of the flowers from which they are extracted and can be described as being feminine, powdery, subtle, modest, romantic, and even poetic.	Chamomile, Geranium, Jasmine, Lavender, Neroli, Rose, Rosewood, Ylang-Ylang, Petitgrain
HERBACEOUS Essential Oils that have herbaceous scents can be further described as smelling green or grassy. These Essential Oils often have mild floral yet invigorating spring-like scents that are associated with lush, wet foliage.	Chamomile, Clary Sage, Eucalyptus Radiata, Fennel, Hyssop, Marjoram, Melissa, Rosemary, Thyme, Oregano, Bay Laurel, Catnip, Sage Dalmatian, Parsley, Tea Tree, Yarrow
CAMPHORACEOUS These Essential Oils have strong scents and are known to be beneficial for clearing the respiratory system due to their clarifying, penetrating, energizing, purifying, and almost medicinal aromas.	Camphor, Cajeput, Eucalyptus, Pennyroyal, Laurel Leaf, Lavandin
MINTY Essential Oils with a minty scent are strong-scented and are distinctly known for their bracing, fresh fragrances. They are reputed to be clearing and cooling when used in aromatherapy and topical applications.	Spearmint Wintergreen Peppermint

SPICY These Essential Oils have exotic, warm, intense aromas that are often reminiscent of baking and other warm memories. With strong scents, they are commonly used to stimulate energy and focus.	Aniseed, Basil, Black Pepper, Cardamom, Cinnamon, Coriander, Cumin, Ginger, Nutmeg, Allspice, Cassia, Clove Bud
RESINOUS/MUSKY These Essential Oils exude deep, rich scents that are smoky, woody, earthy, sweet, leather-like, and warm. Their mellow, alluring, and long-lasting fragrances lend a reassuring quality that makes them ideal for use in spiritual practices.	Benzoin, Elemi, Frankincense, Myrrh, Peru Balsam
WOODY/EARTHY These Essential Oils have deep, warm, lingering scents. Often described as smelling “brown,” Their fragrances are soft, masculine, musky, and sensual. Their seductive, and hypnotic qualities create an atmosphere of mystery.	Cypress, Juniper Berry, Pine, Sandalwood, Fir, Cedarwood, (<i>Atlas & Virginian</i>), Palo Santo, Rosewood, Patchouli, Vetiver, Valerian, Carrot Seed

1.8.Applications 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 plants. Hence essential oils are used in flavorings, perfumes, pharmaceutical preparations like anti-microbial agents, and many other ways.

Ginger Oil: Ginger Essential Oil represents one of the best natural remedies for stomach problems, including colic, indigestion, stomach aches, and spasms. Ginger Oil can be a great remedy for colds, flu, coughs, asthma, bronchitis, and loss of breath.

Pepper Black Oil: Gives relief from spasms and cramps, Good for the digestive system, Relieves sinusitis and nasal congestion, Gives relief from gas problems, Aids in weight loss, Helps to cure vitiligo.

Cumin Seed Oil: Cumin essential oil is a popular spice that can be used to enhance culinary dishes from around the world. It can purify the body's systems, and it is ideal for internal cleansing. It is a great essential oil for supporting digestive health, and it can help relieve occasional digestive discomfort.



Figure – 1.2

Therapeutic uses of Essential Oils

CHAPTER – 2

LITERATURE SURVEY

1.Title : Facile Methods for the Extraction of Essential Oil from the Plant Species.

Authors : J. Ranjitha and S. Vijiyalakshmi.

Journal and Year : Pharmaceutical Sciences and Research, 01 April 2014.

Abstract : Essential oils are a various group of natural products. Oils are the important source of aromatic and flavoring chemicals in food, industrial, and pharmaceutical products. volatile oil may be a concentrated hydrophobic liquid containing volatile aroma compounds derived from the various parts of the plants. Various extraction methods are employed in the manufacture and extraction of essential oils from the plant materials. Essential oils are produced using several techniques like water distillation, steam distillation, hydro diffusion, maceration, solvent-free microwave extraction, solar distillation and CO₂ supercritical fluid extraction, etc. These extraction and expression methods are wont to remove the oils from the dried/fresh plants (or) machines to squeeze the oil out of the plants. Out of all the extraction methods, supercritical CO₂ method is more efficient. the current paper is an effort to list out the assorted methods of extraction of oil from the various parts of the plants and their merits and demerits.

2.Title : Essential Oils from Plants

Authors : Monica Butnariu , Ioan Sarac .

Journal and Year : Journal of Biotechnology and Biomedical Science , 21 Dec 2018.

Abstract : Essential oils, called volatile oils or ethereal oils, are natural metabolic secretions of plants, the role of which isn't yet fully understood by science. Some specialists consider them to be true plant hormones, fluid manifestations of the system of plants, within the sense that they contribute to the removal of pests, attracting, instead, pollinating agents, which are some insects and birds. Small,

light-colored spherical structures, is, in fact, modified filaments, which behave like some secretory glands of essential volatile oils. they're those who give the fragrance of the aromatic plant and have, within the case of salvation, medicinal curative properties, like fever reduction, blood cleansing, and relieving pain. Essential oils are products isolated from plants or organs through a physical process that have a particular volatility (higher or lower) and possesses a pleasurable odour characteristic of the source from which they originate. they're also referred to as volatile or essential oils and their name are usually given by the popular name of the plant from which they're extracted. the foremost important characteristic of those mixtures, which also gives the special measure, is that the specific smell. this is often the premise for his or her use in perfumery, cosmetics and also the food industry. Many essential oils have special therapeutic qualities, a number of which are known and used since antiquity.

3.Title : *Zingiber officinale* Rosc. essential oil, a review on its composition and bioactivity

Authors : Mohaddese Mahboubi

Journal and Year : Clinical Pytoscience , 15 Jan 2019

Abstract : *Zingiber officinale* Rosc. is widely used as spice and medicinal plant in folk and traditional medicines. The aim of this study was to review the chemical composition and biological activities of *Z. officinale* (ginger) oil. Ginger oil is extracted from *Z. officinale* rhizomes, which its chemical composition influences from nation-state, extraction methods, freshness or dryness of rhizomes. The antibacterial, antifungal, analgesic, anti-inflammatory, anti-ulcer, immunomodulatory, relaxant, and warming effects of ginger oil are confirmed in experimental and preclinical studies. the protection problems with ginger oils are well documented and are generally thought to be safe. thanks to wide pharmacological effects of ginger oil, attention to ginger oil as an ingredient of natural formulations in management of gastrointestinal and respiratory diseases is efficacious.

4.Title : Yield, Quality and Composition of Cumin Essential Oil as Affected by Storage Period

Authors : Abdlazim Mohamed Ali , Hana Jumma Ali Jumma

Journal and Year : Analytical Mass Spectrometry and Chromatography , March 2019.

Abstract : Essential oils from newly produced (2017) and former season crop (2016) of cumin (*Cuminum cyminum* L.) seeds collected from local farmers in Dongla area, north Sudan were extracted and analyzed to work out the effect of storage period on essential oil yield and quality. The oil was extracted using hydro distillation method. Identification of the volatile compounds was performed using gas chromatography-mass spectrometry (GC/MS). Results revealed that oil obtained from cumin (*Cuminum cyminum* L.) was yellow. With reference to oil characteristics, the precise gravity, index of refraction, acid value, ester value and other quality characters weren't significantly different between the stored and newly harvested seeds. Thirty-eight compounds were separated and identified in cumin seeds oil in both seasons with some minor differences in some components between 2016 and 2017 samples. Major effective components of cumin seeds essential oil were cumin alcohol (4-isopropylbenzyl alcohol), cumin aldehydes as 4-Isopropylbenzaldehyd and seven,7-dimethylbicyclo (4.1.0) hept-3-ene-4-carbaldehyde called carenal, terpinenes as γ -terpinene and α -terpinene (1,4-Cyclohexadiene, 1-methyl-4-(1-methylethyl) and 1,3-cyclohexadine-1-methanol, 4(1-methethyle)-), β -pinene, o-cymene and p-menthadienol in both stored and newly harvested samples. Differences between the samples season wise were very minor with relation to variety of volatile components but percentage oil yield was largely decreased by storage period.

5.Title : The Study on Extraction Process and Analysis of Components in Essential Oils of Black Pepper (*Piper nigrum* L.) Seeds.

Authors : Thien Hien Tran, Le Ke Ha , Duy Chinh Nguyen , Tan Phat Da.

Journal and Year : Agriculture , 22 January 2019

Abstract : Black pepper (*Piper nigrum* L.) could be a tropical crop with extensive medicinal potential in ethnomedicine and nutraceutical applications. The volatile oil of black pepper finds wide applications in inhabitation of respiratory infections and soothing of muscular pains because of its warming and energizing property. The pungent bioactive piperine is liable for this function, and thus, efficient technology is required for an optimal extraction process of this compound. within the present article, we've got developed a procedure for extracting black pepper oil from Vietnam, optimizing conditions that affect the extraction process.

The effect of process parameters, namely material size, preservation method, the concentration of binary compound, the concentration of soak time, the ratio of fabric to water, temperature extraction, time extraction on the extraction yield, and relative efficiency were investigated. Results demonstrated that 20 g of black pepper milled with a mesh size of 160 obtained 0.48 g of oil (2.4%) at a staple to water ratio of 1/21 (g/mL) at 150 °C during a time of 5.2 h. GC-MS (Gas chromatography–mass spectrometry) spectra showed that 3-carene (29.21%), D-limonene (20.94%), caryophyllene (15.05%), and β -pinene (9.77%) were present as major components. These results suggested that the volatile oil extracted from Vietnamese black pepper is applicable within the manufacturing processes of insecticides and air deodorizers.

CHAPTER – 3

AIM AND SCOPE

- Our project aims to extract oils from oleoresins like Ginger Oleoresin, Pepper Black Oleoresin, and Cumin Seed Oleoresin by the Hydro Distillation method. And also study the chemical composition of the extracted essential oils using GC-MS.
- Essential oils have an cost-effective value, marketing value, and industrial value in both local and international markets. Essential oils are aromatic substances widely employed in the Perfume industry, Food and Flavouring, Cosmetics, and Pharmaceutical Products. They need high value in economic since they enter a good range of industries.
- This paper focuses on essential oils and their applications by employing essential oils as natural preservatives that are suitable to be utilized in food preservation, pharmaceutical, cosmetics, and other industries.
- So far as factors that are liable for food spoilage and other health-related problems are still in living, there is the requirement to develop sustained preservation and public health relief technique.
- The recent advances within the application and alternative means of fruits and food decay, especially natural products as a preservative for fruits, essential oils as a drug likewise as their antimicrobial characteristics are reviewed. Other applications within the food, cosmetic and pharmacological industries will be conferred.

CHAPTER – 4

EXPERIMENTAL WORK

4.1.Experimental Setup:

The schematic diagram of the experimental setup is shown below. The experiment was conducted in a Dean-Stark Apparatus. The apparatus consists of one round bottom flask of 500 ml which contains sample and water. The top flask is connected with the condenser through the connector. There is a measuring trap to collect the water and oil in the sample. The separating funnel is used for the separation of essential oil and water.

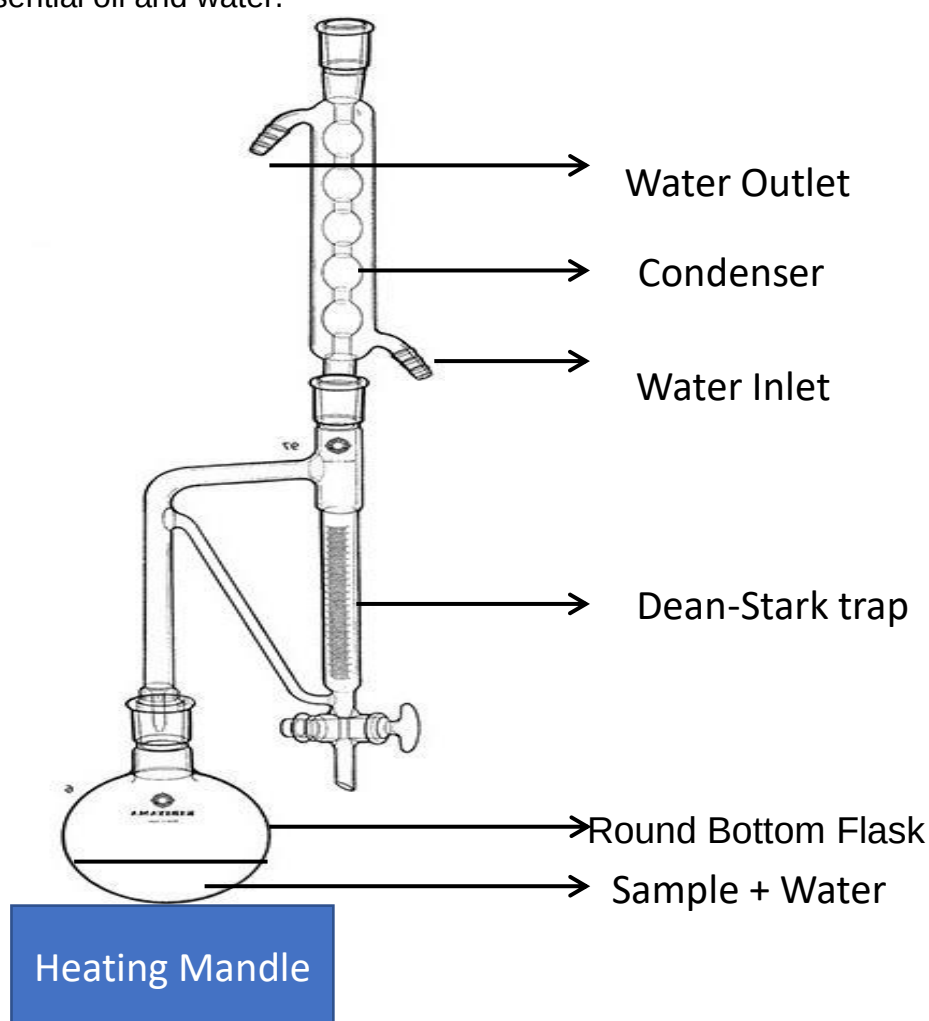


Figure-4.1 Flowsheet for Extraction of Essential Oils using Hydro-Distillation Method

4.2.EXPERIMENTAL PROCEDURE

- Approximately measure 10ml of the sample into a round-bottomed flask(RB flask). To that add 100ml (accurately) of water.
- Attached is the flask to the Dean-Stark condensing and collecting system. The temperature of the flask was adjusted at such a rate that the boiling point of water so that the oleoresin mixture boils without bumping.
- Continued the distillation for 3 hours as per the internal procedure.
- Washed droplets of water that adhere to the lower end of the condenser tube into the receiver with solvent or carrier liquid using the spray tube.
- Inserted the loose plug of cotton wool into the top of the condenser tube to prevent the condensation of atmospheric moisture in the condenser tube.
- After the completion of the distillation, the distillate was collected in the separating funnel. The distillate in the separating funnel was kept for 24 hours.
- Allow to separate two layers (aqueous & oil), collect the oil, and stored it in an air-tight container.
- Then the oil is preserved for characterization studies. The resultant oil is known as Essential Oil.



Figure-4.2 *Extracting Essential Oils using Dean-Stark Apparatus*

4.2.1.Precaution:

- The temperature of the extraction chamber cannot be too high, lest some components of the oil are 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 to extract all the oil components present in the oleoresin.

4.3.EXPERIMENTAL OBSERVATION

Table-4.1 Physical / Chemical Properties of Essential Oils

Physical/Chemical Properties	Ginger Oil	Pepper Black Oil	Cumin Seed Oil
Colour	Light Yellow to Yellow	Colorless to Bluish Green	White to Tan
Yield	30 %	21 %	15 %
Sensory	Spicy, woody, terpene, warm, ginger, citrus.	Peppery, warm, terpy, woody, pungent odor.	Strong, somewhat fatty, and green odor.
Taste	Spicy, ginger	Spicy, woody, earthy terpenic, peppery, herbal.	Spicy, faintly pungent flavor.
Boiling Point	254 ⁰ c	166 ⁰ c	232 ⁰ c
Flash Point	150 ⁰ F	130 ⁰ F	219 ⁰ F
Density	0.871 g/mL at 25 °C	0.873 g/mL at 25 °C	0.908-0.958 @ 20 °C
Refractive Index	<i>n</i> ₂₀ /D 1.49	<i>n</i> ₂₀ /D 1.483	<i>n</i> ₂₀ /D 1.4940-1.5160
Solubility	Soluble in most fixed oils, mineral oil, and alcohol, but it is insoluble in glycerin and propylene glycol.	Soluble in most fixed oils, mineral oil, and propylene glycol.sparingly soluble in glycerin.	Soluble in fixed oils, mineral oil; very soluble in glycerin, propylene glycol.

CHAPTER-5

RESULTS AND DISCUSSIONS

5.1.EXPERIMENTAL RESULTS

- The experiment was repeated with varying the volume of extraction medium (water) to compare the volume of extracted oils.
- Below table represents the volume of oil extracted from oleoresins with varying water quantity.

Table 5.1

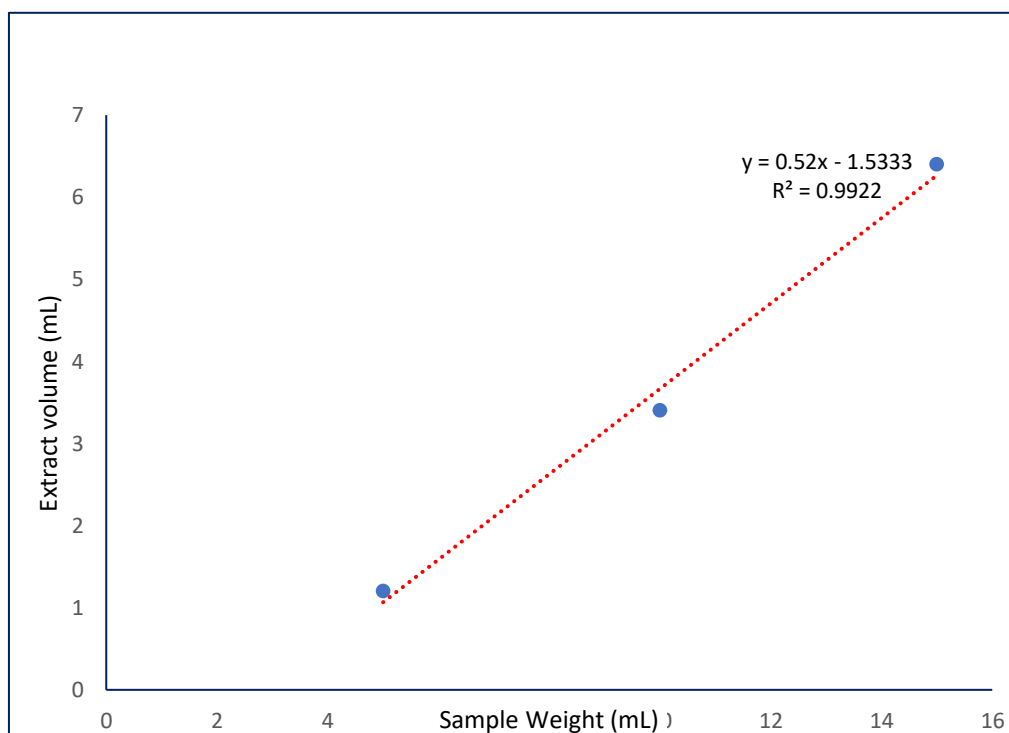
Volume of oil extracted from different oleoresins.

No	Material	Weight of Sample (ml)	Weight of Water (ml)	Time for Heating (min)	Temperature (°C)	Volume of Oils (ml)
1	Ginger Oleoresin	10	50	180	100	3.0
		10	100	180	100	3.1
		10	150	180	100	3.2
2	Pepper Black Oleoresin	10	50	180	100	1.9
		10	100	180	100	2.0
		10	150	180	100	2.1
3	Cumin Seed Oleoresin	10	50	180	100	1.4
		10	100	180	100	1.5
		10	150	180	100	1.6

- To check the linearity i had taken three different concentrations of the sample.

Material	Weight of Sample (ml)	Volume of Oils (ml)
Ginger Oleoresin	5	1.2
	10	3.4
	15	6.4

- The below graph represents the plot between sample weight (ml) and Extract Volume (ml) of Ginger oil



5.2. Characterization of Essential Oils

The initial screening of oil extract is carried out with Gas chromatography technique, which provided only quantitative information on peak pattern of the extracted oil. In recent years, gas chromatography-mass spectrometry (GC–MS) has commonly been employed for identification of various active compounds that are present in oil extract. GC–MS is one of the best, fast and accurate techniques to detect various compounds, including alcohols, alkaloids, nitro compounds, long chain hydrocarbons, organic acids, steroids, esters and requires a small volume of oil extracts. Hence, in the present study, GC–MS technique was adopted for detection and identification of compounds present in the essential oils.

5.2.1. Gas Chromatography-Mass Spectroscopy (GC-MS)

GCMS is often said to be the gold standard in the chemical analysis of essential oils. GCMS report being the unique 'fingerprint' of essential oil. GCMS analysis gives us an insight into the qualitative composition of essential oil, which in turn helps us understand key aspects and implications surrounding using that essential oil. This includes its **purity, quality, safety, and therapeutic applications.**



Figure-5.1 Analysing Essential Oils Using GC-MS

GC-MS can be used to study liquid, gaseous or solid samples. Analysis begins with the gas chromatograph, where the sample is effectively vaporized into the gas phase and separated into its various components by employing a capillary

column coated with a stationary liquid phase. The compounds are propelled by an inert carrier gas like helium. As components of the mixture are separated, each compound elutes from the column at a different time supported on its boiling point and polarity. The time of elution is named to as a compound's retention time. GC has the capacity to resolve complex mixtures or sample extracts containing many compounds.

Once the components eluted from the GC column to MS detector, they're ionized and fragmented by the mass spectrometer using electron ionization sources. Ionized molecules and fragments are then accelerated through the instrument's mass analyzer, which very often may be a quadrupole or ion trap. It's here that ions are separated based on their different mass-to-charge (m/z) ratios. GC-MS data acquisition may be performed in either full scan mode, to hide either a large range of m/z ratios, or selected ion monitoring (SIM) mode, to collect data for specific masses of interest.

The final steps of the process involve ion detection and analysis, with fragmented ions appearing as a function of their m/z ratios. Peak areas, meanwhile, are proportional to the amount of the corresponding compound. When a complex sample is separated by GC-MS, it'll produce many alternative peaks within the gas chromatogram and every peak generates a novel mass spectrum used for compound identification. Using extensive commercially available libraries of mass spectra, unknown compounds and target analytes can be identified.

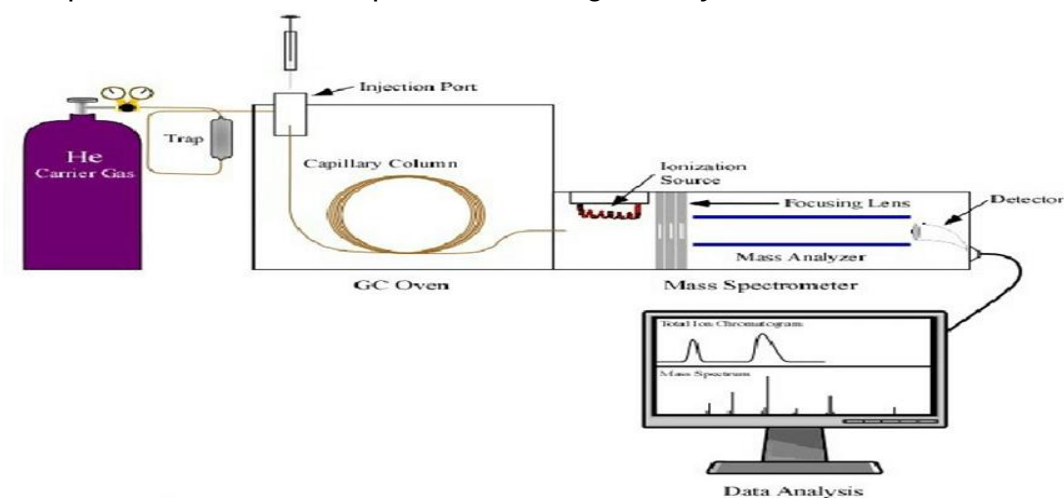


Figure-5.2 Instrumental Setup of GC-MS

5.2.2. Analysis of Ginger Oil

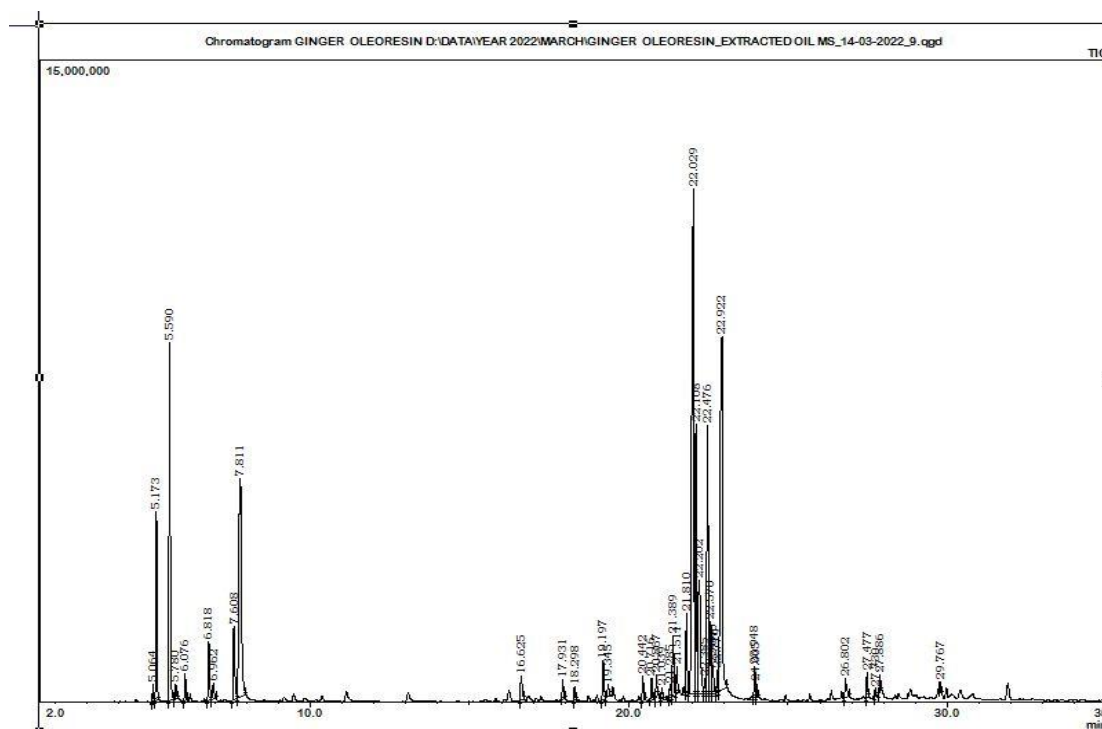


Figure-5.3 GC-MS analysis of Ginger Oil

Table-5.2 Major components present in Ginger Oil

Retention Time	Name	Area %
5.173	α -Pinene	3.9
5.590	Camphene	8.6
6.818	Myrcene	1.6
7.608	Limonene	2.2
7.811	β -Phellandrene	10.9
21.810	Germacrene	1.9
22.029	Gingiberene*	20.1
22.570	β -Cubebene	2.1
22.922	Sesquiphellandrene*	15.1

*active ingredient.

5.2.3. Analysis of Pepper Black Oil

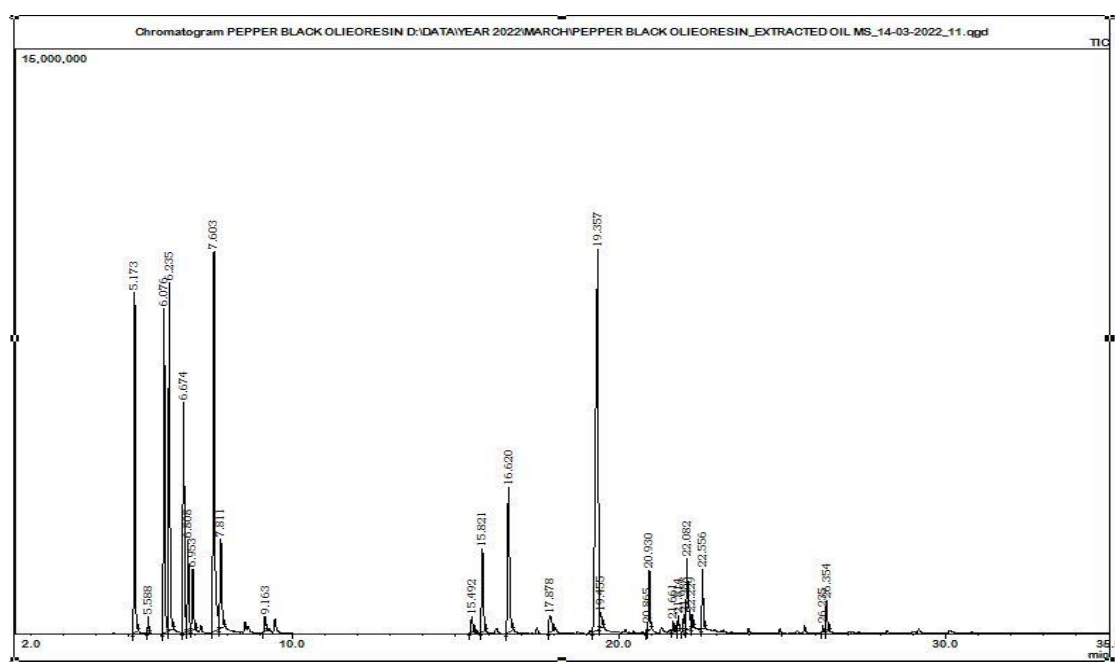


Figure-5.4 GC-MS analysis of Pepper Black Oil

Table-5.3 Major components present in Pepper Black Oil

Retention Time	Name	Area %
5.173	α -Pinene	8.8
6.076	β -Pinene	9.3
6.235	Sabinene*	10.2
6.674	δ -Carene	7.1
6.808	Myrcene	2.6
7.603	Limonene	14.1
7.811	β -Phellandrene	3.3
16.620	α -Copaene	5.9
19.357	Caryophyllene*	21.7

*active ingredient.

5.2.4. Analysis of Cumin Seed Oil

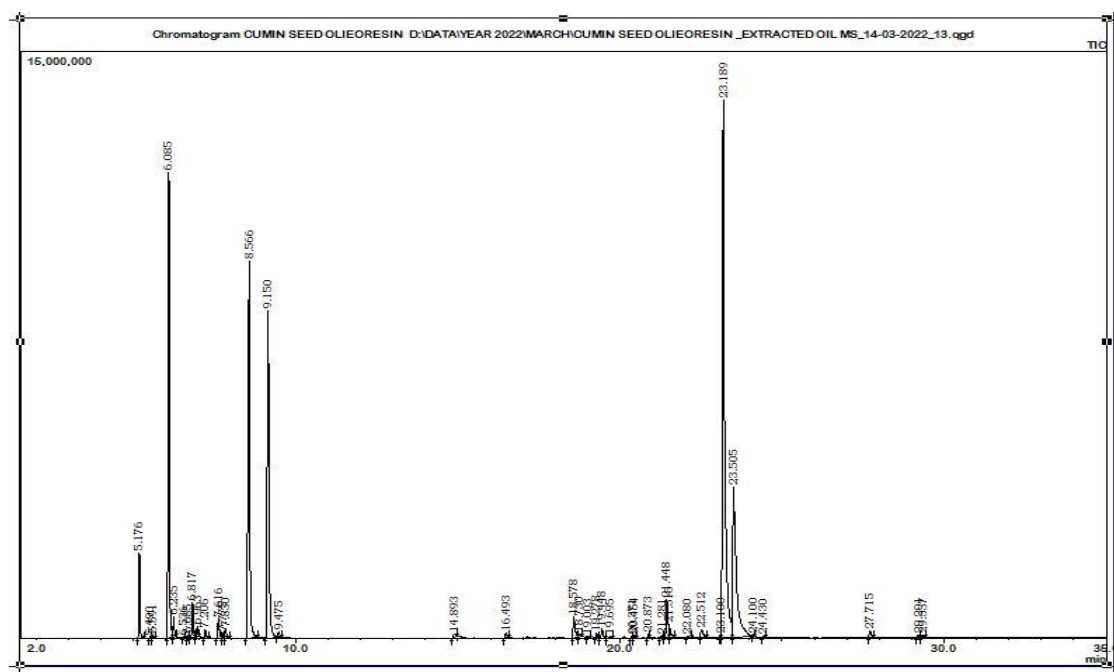


Figure-5.5 GC-MS analysis of Cumin Seed Oil

Table-5.4 Major components present in Cumin Seed Oil

Retention Time	Name	Area %
5.176	α -Pinene	2.3
6.085	β -Pinene	15.3
6.817	Myrcene	1.1
7.616	Limonene	0.8
8.566	γ -Terpinene	16.2
9.150	m-Cymene	14.4
23.189	Cuminic Aldehyde*	29.1
23.505	Dihydro-Cuminic Aldehyde	13.2
27.715	Cuminic Alcohol*	0.3

*active ingredient

CHAPTER-6

SUMMARY AND CONCLUSION

This investigation has given detailed information on extraction and identification of chemical components present in essential oil of oleoresin. The essential oils are isolated mostly from the hydro-distillation method which is more suitable for this process and simple to hold. The experiment has been allotted for the extraction of oil from Oleoresins namely Ginger oleoresin, Pepper Black oleoresin, and Cumin Seed oleoresin which have high essential oil content. The impact of extraction medium(water) in extraction process was studied by altering water quantity and located the recovery of volatile oils from oleoresin were same for all three oleoresins no matter water content employed in the study at same experimental condition. During this study, all three essential oil Ginger oil, Pepper Black oil and Cumin Seed oil were screened using GCMS technique.

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