

ADDIS ABABA UNIVERSITY
ADDIS ABABA INSTITUTE OF TECHNOLOGY
SCHOOL OF CHEMICAL AND BIO ENGINEERING



OPTIMAZATION AND CHARACTERIZATION OF ESSENTIAL OIL
FROM CORIANDER SEED

A Thesis Submitted to Addis Ababa Institute of technology, School of Chemical and Bio Engineering in Partial Fulfillment of the Requirements for the Degree of Master of Science in Chemical Engineering (Process Engineering Stream)

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March , 2021

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This is to certify that the thesis prepared by **Firehiwot Mola**, entitled as: ‘**Optimization and Characterization of Essential Oil from Coriander Seed**’ and submitted in partial fulfillment of the requirements for the degree of Masters of Science in Chemical Engineering (Process Engineering) complies the regulation of the University and meets the accepted standards with respect to originality and quality.

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UNDERTAKING

I certify that research work titled “**Optimization and Characterization of Essential Oil from Coriander Seed**” is my own work. The work has not been presented elsewhere for assessment. Where material has been used from other sources it has been properly acknowledged / referred.

Firehiwot Mola

Abstract

Coriander seed (Dimblal) is both an annual and a perennial herb that has been cultivated in Ethiopia and has been used to flavoring, cooking, and fragrance rather than for medical purpose. The goal of this research was to Optimization and characterization of essential oil from coriander seed (*Coriandrum sativum* L.) using steam distillation method. Umbellifera, a family of coriander seed, was the raw material of experimental work. In this research, the moisture in coriander seed was first dried in an oven at 105°C, then the dried coriander seed were grind in cross beater mill with sieve size of 1mm and the sample was sieved using vibrating shaker. Finally steam distillation was used to complete the extraction. The results from experimental work were analyzed with the aid of DESIGN EXPERT 7.0 software and using general factorial design. The effect of various parameters, such as extraction time and particle size on the extraction yield of coriander oil was investigated. This work was deals with random experimental design consisted of extraction time of 2, 3 and 4hours; particle size of 1, 1.4, 1.8 and 2mm, under constant temperature (85°C), and it was conducted with three replications. In steam distillation, the maximum oil yield of 0.84% was obtained at extraction time of 4 hour and with particle size ranges of 1-1.4mm and at a 2hr of extraction time and range of 1.8-2mm particle size, the minimum oil yield of 0.02% was obtained. This shows that, a higher yield is obtained by increasing the extraction time and decreasing the particle size. Physic-chemical characterization of the oil was carried out. The extracted oil has a refractive index of 1.4699, specific gravity of 0.878, pH value of 6.2, viscosity 35 mpas, boiling point at 131°C and saponification value (16.833ml/g), iodine value (31.725ml/g), acid value (21.0375ml/g). Gas chromatography–Mass spectroscopy was used to determine the composition of coriander oil. The major components were alpha-pinene (13.4253%), Linalyl acetate (54.8565%), gamma-terpinene (4.8523%), Camphor (3.7224%), Limonene (8.3925%), Decanal (1.6571%), and beta-myrcene (4.5846%).

Key words: *Corianderum sitavum* L., steam distillation, Essential oil, Extraction, Gas chromatography-mass spectroscopy, FT-IR.

Acknowledgment

Praise to GOD the most merciful and compassionate for giving me the strength in completing this thesis research.

Next and for most, I would like to express my deepest gratitude to my advisor Dr.Eng. Abubeker Yimam from Addis Ababa University, Addis Ababa institute of technology (AAIT), Dean, School of Chemical and Bio Engineering for his tireless advice, supervision, guidance and patience throughout my research work.

Thanks to Addis Ababa University for providing financial support in the completion of this work. I also would like to convey my gratitude to a great number of laboratory assistant in AAIT chemical Engineering Department who's helping me throughout the work especially Mr.Hintsaselassie Seifu and others who were helping me throughout the work.

My deepest gratitude goes to my classmate yeshidagne Nigussie for his kindness, advice and understanding, at my weakest moment his moral compass is always turned me positive direction during my thesis work.

Last but not least, I want to acknowledge my lovely husband and my family for their incredible support, guidance, encouragement and always wish the best for me.

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List of acronyms

ANOVA	Analysis of Variance
AV	Acid Value
FTIR	Fourier Transform Infrared Spectroscopy
GC-MS	Gas Chromatography Mass Spectroscopy
IV	Iodine value
SG	Specific gravity
SV	Saponification value

1. Introduction

1.1 Background

Coriander seed (Dimblal) which belongs to the family Apiaceae (Umbelliferae) is mainly cultivated from its seeds throughout the year (Mhemdi et al., 2011). The scientific name of coriander seed is *Coriandrum sativum* L. The coriander word derived from the Greek word Koris, meaning a stink bug. Coriander is mainly applied as a spice, utilizing both its leaves, known as cilantro, if the dried fruits are used; the herb is called as coriander. It is used as herbal condiment for many Asian and Mediterranean culinary preparations (Nadeem et al., 2013). This plant is widely distributed and mainly cultivated for its fruits which are used for different purposes such as aromatherapy, food, drugs, cosmetics and perfumery. The fruits also have medicinal uses for, pain in the joints, gastrointestinal complaints and indigestion, glycaemia as well as sleep prolonging. The interest of coriander fruits oil becomes more important since the European Union authorized the use of coriander oil for use as a food supplement (Douglas, 2005).

In Egyptian times, coriander was known as a medicinal herb and its seeds have been found in the tomb of Tutankhamen, revealing its mythological important. Common uses of coriander include its application as a diuretic, an antioxidant present and memory enhancing compound. Coriander oil is frequently used as a flavor or in perfume and soap production and a rich source of several beneficial phytonutrients. Coriander oil is particularly interesting because it is more stable and capable of retaining its odour for a longer period than other oils of its class.

Coriander is classified into two categories of commerce based on the size of the fruit, which defines the volatile oil content and end use. The small fruited type var. *microcarpum* (diameter 1.5-3mm) is grown widely in cooler temperate regions while in tropical and subtropical condition the large fruited type var. *vulgare* (diameter 3-5mm) is grown. The small-fruited types contain higher oil (0.5-2%), which is extracted for its essential oil while the larger fruit with a lower oil (<1%), is used for grinding and blending (diederichsen, 1996; Maroufi et al., 2010).

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Essential oils are aromatic substances produced from aromatic plants with original aroma and flavor of the odoriferous plants. They are colorless or slightly yellow complex mixtures of odorous and volatile compounds. They occur in various site of plant anatomy, in some cases they are found throughout the plant organ and they could be restricted to specific organ or product of the plant. Either of the different part such as flowers, seeds, leaves, twigs, herbs, woods, barks, resinous, sap, or root or the whole part of the single plant species could be sources of essential oil. They were originated in ancient Rome, Greece, and Egypt throughout the middle and Far East had, as a common feature. They were used for many purposes such as perfumes, cosmetics, lotion, food preservation, food flavors, deodorants, pharmaceuticals, medicinal and embalming antiseptic. The most important essential oils in terms of production are citrus and mint oils, with orange oil displaying a production of over 50,000 tones, although rose and jasmine are considered to be the most financially important essential oil. (Beement mengesha, 2009)

Essential oils are extremely volatile substances extracted from plants of a single botanical genus using a physical method or technique. The oils are usually labeled with the name of the plant species from which they came. There are several techniques that can be used to extract essential oil: water distillation, steam distillation, solvent extraction, and expression under pressure, supercritical fluid extractions and subcritical water extractions (Properizi, et al., 2013).

Essential oils are so termed as they are believed to represent the very essence of odor and flavor (Weiss, 1997). Essential oil plants and culinary herbs are a diverse group of plant species used for flavorings in foods and beverages, as well as fragrances in pharmaceutical and industrial products. Essential oils derive from aromatic plants of many general distributed worldwide (Panda, 2011). In the old and new testaments, there are over 200 references to aromatics, incense and ointments. 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 (pandey and Virendra, 2006-2007).

1.2 Statement of the problem

Ethiopia is located in the tropical region, so the weather is conducive to plant growth of coriander seed. There are a lot of coriander seed producing areas which are found in Arsi zone, Oromiya regions. Most Ethiopian people use Coriander as house holding spice preparation. Coriander plays an important role in most Ethiopian peoples' domestic spice trade and its seeds are used for a complete spice mixture called 'berbere'. Its seed is used as flavoring of 'berbere' (which is a spiced, hot red pepper powder used for numerous meat and vegetarian dishes), 'injera', cakes and bread and its leaves added as an aromatic herb to 'wot' and tea. In 'Kefa', seeds are added to cheese and to porridge. World Science approves the use of coriander seed for medical purposes for both internal and external treatment issues, according to the literature. In addition to its medicinal purpose, coriander seed (*Coriandrum sativum* L.) is also used for production of soap, perfumes and lotions, food flavorings, food preservation. Nutraceuticals and cosmeceuticals from the coriander oil (Atta, 2003; Takruri and Dameh, 1998) culinary (food making) (Diederichsen, 1996), and; in perfumery, food, beverage, and pharmaceuticals industries (Jansen, 1981). The seed contains significant quantities of carotene, thiamine, riboflavin, niacin, tryptophan, vitamin B6, folate, vitamin C and E (Holland et al., 1991); iron, manganese, magnesium and dietary fiber to the diet (Ensminger and Esminger, 1986).

Essential oil industry processing is very limited in Ethiopia. The amount of import essential oil increase from day to day. A large amount of coriander seed now a day is exported to united Arab emirate. Producing coriander oil industrially results in a quality product and will be a source of income by exporting it to other countries. Instead of exporting the seed, exporting the oil will bring more income and create additional jobs. Thus, this research is intended to provide scientific results (such as oil yield and the different factors affecting the production) that can be inputs for the establishment of Coriander oil industry in Ethiopia.

1.3 Objectives

1.3.1 General objective

The general objective of the study was to optimization and characterization of essential oil from coriander seed (*Coriandrum sativum* L.).

1.3.2 Specific objectives

- ✚ To extract coriander oil.
- ✚ To investigate the effects of particle size and extraction time.
- ✚ To determine compositions of coriander oil.
- ✚ Characterization of some basic physical and chemical properties of coriander oil.
- ✚ Optimization of essential oil extraction from coriander seed using steam distillation.

1.4 Significance of the study

This study is an experimental study of steam distillation method, extraction, and characterization and optimization of coriander oil using coriander seed as raw material.

- ✓ This study will contribute a significant method of identification of high quality of coriander oil.
- ✓ This work will show the possibility of extraction technology of coriander oil extraction process.

2. Literature review

2.1 Over view

Coriander is one of the oldest herbs and spices. The seeds have been found in ruins dating back to 5000 B.C and Greek Ebers Papyrus earlier 1550 BC have been mentioned from this herb. This is likely a reference to the strong aroma given off by the cilantro plant leaves when they are bruised. On the other hand, interestingly about one-half liter of coriander seeds was found in the tomb of Tutankhamun (Ramesses II) in Egypt. In Egypt, the herb was known as “spice of the happiness”. Coriander appears to have been cultivated in Greece since at least the second millennium BC, where the plant was used in perfumes and cooking, as well as Hippocrates traditional medicine. In the Roman Empire, the herb was also commonly used. As an example, Apicius (a collection of Roman cookery recipes) includes some 70 recipes using coriander in his cookbook.

Until now, coriander has been widely cultivated and used in many parts around the world such as in Russia, Central Europe, Asia, and Africa... According to Ravi, India are the world’s largest Producer and exporter of coriander and refined products (Ravi et al., 2007). For coriander origin, Diederichsen (1996) also mentioned in his report that the origin coriander is derive in the coastal areas North and East of the Mediterranean Sea and from there the plant spread to Europe, Africa and Asia in ancient time. There is evidence that coriander was grown in Germany around 9,000 years ago, and remains of coriander fruits were found in Tombs in Israel dated 8,000 years old. In India, coriander has been grown for the last 5,000 year. It was used as a medicinal plant and as a condiment by the Egyptians since at least 4,500 year ago (Diederichsen, 1996). In a recent report also pointed out that coriander was grown in England as far back as the 1st century A.D (introduced by the Romans), and was known as a vegetable in China in the 5th century A.D (introduced through Persia).In 1885, in the Voronezh district of Russia, the first factory for steam distillation of coriander essential oil was establish.

2.2 Description of coriander seed

Coriander (*Coriandrum sativum* L.) is a common spice crop that plays an important role in flavoring. The stems, leaves & fruits all have a pleasant aromatic odour. The entire plant when young is used in preparing chutneys, sauces & the leaves are used for flavoring continental curries & soups. The fruits are extensively employed as a condiment in the preparation of curry powder, pickling spices, sausages & seasoning. They are also used for flavoring liquors particularly gin. Coriander seeds are also known for their medicinal properties & are considered carminative, diuretic tonic, stomachic anti-bilious, refrigerant & aphrodisiac, as such, coriander is a frequent ingredient in the preparation of ayurvedic medicines & a traditional home therapy for different ailments. The new value added product obtained from seeds are also in large demand in international markets. The volatile oil is also used in flavoring liquors & for obscuring the bad smell of medicines. (Sharma MM, Sharma RK)

2.3. Geographic Distribution

2.3.1. Overview of coriander in the world

The plant grows all over the world, but originally from the Mediterranean regions, cultivated mainly in the tropical areas such as Ukraine, Russia, Romania, Morocco, Mexico, India, and Argentina. Especially in a cool and comparatively dry frost-free climate at the optimum temperature of germination and early growth of coriander is 20-25 °C (Carrubba et al. 2006). It grows best in dry climates, but it can grow in any type of soil like light, well-drained, moist, loamy soil, and light to the heavy black soil.

2.3.2. Overview of coriander in Ethiopia

Ethiopian coriander does well in areas with an average annual rainfall of 563-1018 mm. Temperatures on the other hand range from 7.9°C and 24.3°C, respectively. The crop is mostly farmed in the Arsi Zone located in the southeast region of Ethiopia. Water is a crucial component for the germination of coriander and farmers seek alternative methods such as irrigation to sustain their crops in instances where there is prolonged drought. Farmers mostly apply traditional farming methods such as the use of organic manure to ensure the end product is natural and conforms to the Good Agricultural Practices (GAP).

2.4 Botanical description

Coriander (*Coriandrum sativum* L.) it is an annual herb belonging to the family of Umbelliferae (Apiaceae). Coriander is a soft plant that growth up to 50cm (20in) tall and is native to southern Europe, Norther Africa, and south west Asia. The leaves vary in form, from thick lobed at the plant's base to slender & feathery higher up on the flowering stems. Coriander is a perennial and an annual herb. It is flowered herb and is usually distinguished with vertical, glabrous, and profusely branching plant. The height of the coriander's plant is in the range from 0.2 to 1.4 m with a well-developing taproot. The stem of coriander usually erect, sympodial and monochasial-branched, sometimes it has several side branches at the basal node. Each branch is finished with an inflorescence. The ribbed stem has the green color, but it turns to red or violet when it is in the flowering period. The stem of full-grown plant is hollow; its basal parts diameter can reach to 2 cm.



Figure 2-1 coriander (*Coriandrum sativum* L.)

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The coriander's leaves are variable in shape, size and number. They have yellow-green color and sheath surrounding and are curiously margined. The supporting stem is up to three quarters of its circumference.



Figure 2-2 coriander leaves

The flowers are in umbels with shortly-stalks and five to ten rays, pale mauve, almost white, delicately pretty. The seed clusters are very symmetrical and the seeds fall as soon as ripe. The fruit is a cremocarp which splits up into two mericarps which remain suspended on the carpophores for some time. The plant is bright green, shining, glabrous and intensely fetid.



Figure 2-3 coriander flower and seed

Table 2-1 Taxonomical description of coriander

Kingdom	Plantae
Subkingdom	Trachaeobionta(vascular plants)
Division	Magnoliophyte(flowering plants)
super Division	Spermatophyte(seed plants)
Class	Magnolipsida(dicotyledons)
Subclass	Rosidae
Order	Apiales
Family	Apiaceae (umbellifera Juss.)
Genus	Corianderum L.

Reproductive biology: Coriander crop requires a cool climate during the growth stage and warm, dry climate when it is ready to harvest. It can be growth in variety of soil types, but it thrives in well-drained loamy soil. Cold climates and high altitudes can result in higher essential oil content and higher seed quality. Coriander crops last approximately 110–140 days. Plants are harvested and dried in the sun for 1-2 days to reduce moisture levels to 18%. The seeds are then thrashed out of the dried plant. Seeds are then dried in the shade until the moisture content is below 9%. Coriander seed is mostly crushed into powder, which is then used as a food ingredient due to its fragrance. The seeds are also used to extract essential oils.

2.5 Importance of coriander

2.5.1 Medical use

From the ancient time, people had known to use coriander as a medicine. The ancient Egyptians had used coriander for medicinal purposes for thousands years (Mathias, 1994). From countries to countries, we can find the coriander in many traditional remedies for different diseases (Azhar and Mazhar, 2003; Duke, 1992; Eddouks et al., 2002; Grieve, 1971; Nadkarni, 1976; Usmanghani et al., 1997). The fruits of the plant are famous for carminative, diuretic effects and used in the treatment of cold, fever, nausea, and stomach disorders. Some of the main importances of coriander are:-

Anemia:-it can be due to lack of sufficient iron in your body. Iron deficiency can also cause tiredness, heart palpitations, shortness of breath, and other symptoms. Coriander, which contains a high amount of iron, is a good choice for dealing with this issue.

Diabetes: - the essential oil of coriander seed can also aid in the treatment diabetes. In a 2009 study, researchers discovered that a coriander seed extract lowered blood sugar levels in diabetic rats. The beta cells in the pancreas were more active after using coriander seed oil. This may help increase insulin levels and reduce blood sugar.

Stimulate Hair Growth:-Hormonal imbalances, stress, weak hair follicles and improper diet can all lead to loss of hair. Using coriander seeds can help in stimulating hair growth and preventing hair fall. Massage your scalp with hair oil which has powdered coriander seeds mixed in it. This will strengthen the hair follicle.

Conjunctivitis:-The antimicrobial properties found in coriander can also protect you from conjunctivitis. Coriander is also a good disinfectant which makes its essential oils an integral part of various eye care products.

Blood Sugar Levels: - coriander used as a natural remedy to lower your blood sugar levels. There are different ways in which you can use coriander essential oil to treat your diabetes. One way is to mix two to three drops of coriander oil with carrier oil and apply it either on your soles or on your pancreas area before you go to sleep.

Cure for Smallpox: - Smallpox can be cured by using the coriander essential oils since they consist of anti-infections, detoxifying, antioxidant and antimicrobial acids which can prevent, cure, reduce the pain and soothe the skin for patients suffering from smallpox.

2.5.2 Used in cooking

All parts of the coriander plant are edible, for example in Thailand, even the coriander root is used as a vegetable in a variety of cooking (Wikipedia), new leaves and dried fruits, on the other hand, are the most commonly used pieces of cooking. Because the flavor is diminished when heated, coriander fresh leaves are usually used raw or added to the dish just before serving. In Indian and central Asian recipes, coriander leaves are used like vegetable and cooked until the flavor degrades.

2.5.3 Flavoring

Coriander fruit is widely used in whole of plant or powder for flavoring purpose. The most coriander goes into curry powder, of which it forms 25-40% (Purse glove, 1981), and this powder is used to flavor liqueurs as being an important flavoring agent in gin production (Jansen and Wageningen, 1981). The coriander essential oil product could be used in the flavoring and aroma industries. Essential oil could be fractionated to provide linalool (usually 60 – 70 %), which could be used as a starting material for synthetic production of other flavoring agents, such as citral and ionone. Besides, the fruits are also used in the preparation of baking, sausages, candies, soups, cookies, buns, cakes, and tobacco products. In Ethiopia, a spice of coriander is widely used to add flavor for meat and vegetarian dishes.

2.5.4 Fragrance

Currently, the beauty industry is the primary consumer of essential oil fragrances. Coriander oil is also used as a fragrance component to impart a fresh and clean aroma in soaps, detergents, lotions and perfumes. It is known for its pungent, intoxicating scent (Kabuba, 2009).

2.6 Coriander seed oil constituents

The raw material of coriander became important for industrial use and further processing. Therefore, coriander fruits are one of the most popular raw materials in oil industry. There exist very different uses of coriander and these are based on different parts of the plant. So, many studies so far investigate the biochemical composition of different parts of coriander. Currently, lot of published studies determine the chemical composition of coriander fruits. According to (Asgarpanah et al 2012), the biochemical composition of ripe fruits of coriander including contents vary from 11.5 to 21.3% (protein); 17.8 to 19.15% (fat); 28.4 to 29.1% (crude fibers); 4.9 to 6.0% (ash); starch (10.53%), sugar(1.92%), minerals (4.98%), water(11.37%), essential oils(0.84%) and pentosans (10.29%) (Asgarpanah and Kazemivash, 2012; Diederichsen,1996).

The essential oil content in dried coriander fruits is in the range of 0.03-2.6%. Linalool varies from (40.9 to 79.9%), geranyl acetate (2.3 to 14.2%), gamma-terpinene (0.1 to 13.6%), and alpha-pinene (1.2 to 7.1%) were identified as the main components in the oil of the coriander fruits (Bhuiyan et al., 2009).

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Many phytochemical studies are done to investigate and compare the chemical composition of the coriander fruits essential oil grown in different regions (Anitescu et al., 1997; Bandoni et al., 1998). The presence of other minerals in fruits of coriander such as Mg, Al, Si, P, S, Cl, K, Ca, Ti, Mn, Fe, Cu, and Zn are reported using X-ray fluorescence analysis (Al-Bataina et al., 2003). The structures of the major compounds identified of essential oil of coriander are represented in figure 2.4.

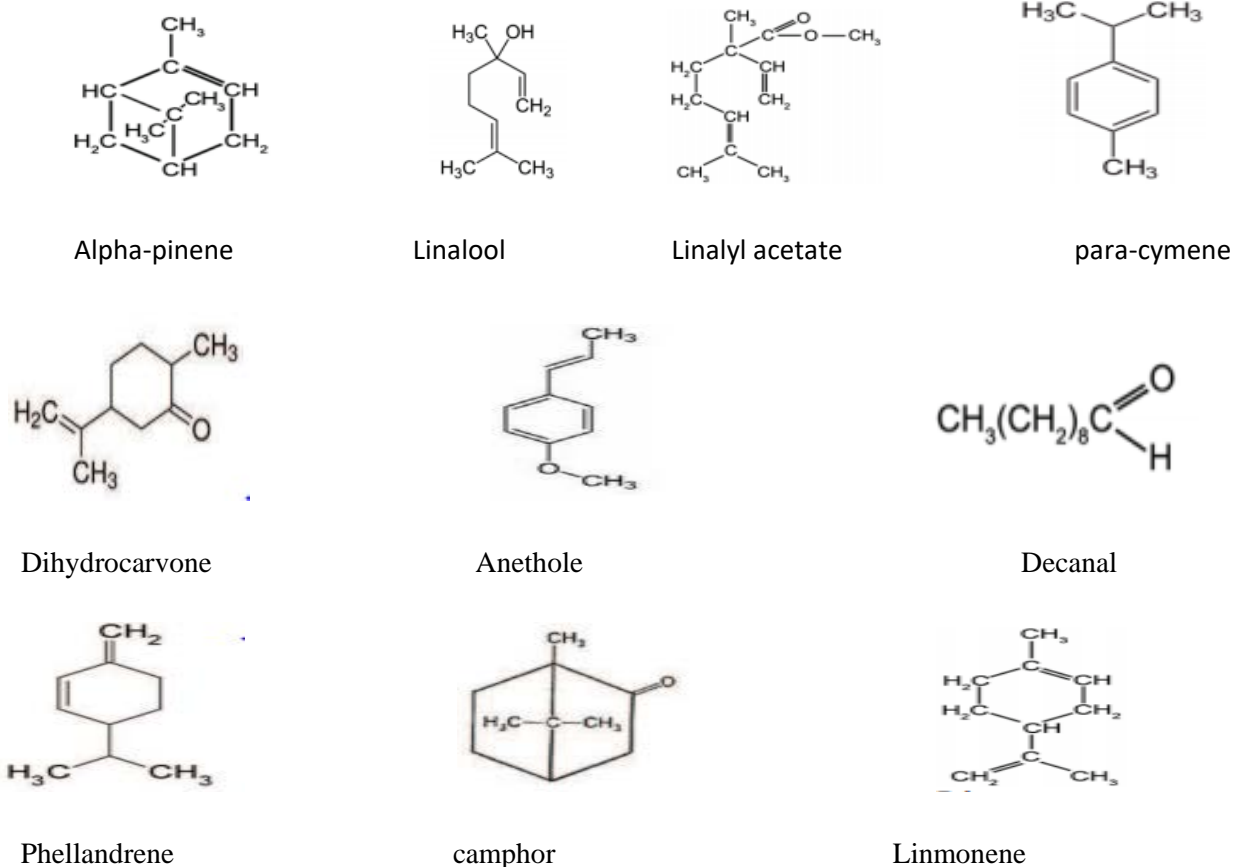


Figure 2-4 structure of the major compound identified in the essential oil of coriander seeds

2.7 Methods of extraction

Essential oils from coriander seed are the volatile, organic constituents of plant matter that play a role in medicinal, cosmetics, and industrial application. They have a low solubility in water but are soluble in fats, alcohol, organic solvents and other hydrophobic substances and are generally liquid at room temperature. The majority of essential oil is obtained from vegetable raw material in different ways.

The extraction of oil from plant materials can be done variety of ways, depending on the oil's stability. Normally, several types of methods can be applied to extract essential oil. Quality of oil depends on the type of extraction. The methods for extracting of essential oils are as follows.

2.7.1 Hydrodistillation

Hydrodistillation is a widely used method is still one of the most economical methods for extraction on essential oil, because of its simplicity, easy construction and more complete production. The aroma plant material is still packed in, and enough water is added and brought to a boil; instead, live steam is injected into the plant charge. By cooling the vapor mixture of water and oil with water, the vapor mixture is condensed. Distillate flows from the condenser into a separator, where oil is immediately separated from the distillate water.

For isolating essential oils from plant materials, there are three types of Hydrodistillation.

1. Water distillation
2. Water and steam distillation
3. Direct Steam distillation

2.7.1.1 Water distillation

In water distillation, the material is completely immersed in water, which is boiled by applying heat by direct fire, steam jacket, closed steam jacket, closed steam coil or open steam coil. The main characteristic of this process is that there is direct contact between boiling water and plant material. To prevent the charge from overheating while the still is heated by direct fire, proper precautions must be taken. When a steam jacket or closed steam coil is used, there is less danger of overheating; with open steam coils this danger is avoided. But with open steam, care must be taken to prevent accumulation of condensed water within the still. Therefore, the still should be

will insulated. The plant material in the still must be agitated as the water boils, otherwise agglomerations of dense material will settle on the bottom and become thermally degraded. Before any field distillation is done, a small-scale water distillation in glassware should be performed to observe whether any charges take place during the distillation process (Baser, K. H.C., 1999).

The yield of oil from a known weight of the plant material can be calculated using the results of this laboratory experiment. Clevenger device is a laboratory apparatus that is recommended for trial distillations. For only this reason water distillation possessed one distinct Advantage, i.e. that it permits processing of finely powdered material or plant parts that, by contact with live steam, would otherwise form lumps through which the steam cannot penetrate. Other practical advantages of water distillation are that the stills are inexpensive, easy to construct and suitable for field operation. These are still widely used with portable equipment in many countries.

The key drawback of water distillation is that it does not allow for complete extraction. Furthermore, certain esters are partially hydrolyzed and sensitive substance such as aldehydes have a tendency to polymerize. Distilling water necessitates a larger number of stills, more space and more fuel. It demands considerable experience and familiarity with the method. The high-boiling and somewhat water soluble oil constituents cannot be completely vaporized or they require large quantities of steam. Thus, the process becomes uneconomical. For these reasons, water distillation is used only in cases in which the plant material by its very nature cannot be processed by water and steam distillation or by direct steam distillation. The Other disadvantage also, as water distillation tends to be a small operation (operated by one or two persons), it takes a long time to accumulate much oil, and so good quantity oil is often mixed with bad quantity oil. The distillation process is treated as an art by local distillers, who rarely try to optimize both oil yield and quality. Water distillation is a slower process than either water and steam distillation or direct steam distillation.

2.7.1.2. Water and steam distillation

In water and steam distillation, the steam can be generated either in a satellite boiler or within the still, although separated from the plant material. In water and steam distillation, the plant material cannot be in direct contact with the fire source beneath the still, however, the walls of the still are good conductors of heat so that still notes can also be obtained from the thermal

degradation reactions of plant material that is touching the sides of the still. As the steam in the water and steam distillation process is wet, a major drawback of this types of distillation is that it will make the plant material quite wet. This causes distillation to take longer because the steam must vaporize the water in order for it to condense further up the still. Using a baffle to prevent the water from boiling too quickly and coming into direct contact with the lower plant material lying on the grid is one way to keep it from being waterlogged.

Advantage of water and steam distillation

- ❖ maximum oil yield obtained
- ❖ The Oil quality obtained by steam and water distillation is more consistent.
- ❖ Steam and water distillation is more energy efficient than water distillation because it is quicker.
- ❖ Component of volatile oil are less susceptible to hydrolysis and polymerization (the still walls affects polymerization).

Disadvantage of water and steam distillation

- ❖ The wet plant content delays distillation because the steam must vaporize the water in order for it to condense further up the still.
- ❖ Due to the low pressure of rising steam, oils of high boiling range require a greater quantity of steam for vaporization, hence longer hours of distillation.
- ❖ A baffle is used to prevent the water from boiling too quickly and coming into direct contact with the lower plant material resting on the lower plant material resting on the grid, causing it to become waterlogged.

2.7.1.3. Direct Steam distillation

Steam distillation is the process of distilling plant material with steam generated outside the still in a satellite steam generator generally referred to as a boiler. A real advantage of satellite steam generation is that the amount of steam can be readily controlled.

Because steam is generated in a satellite boiler, the plant material is heated no higher than 100⁰C and, consequently, it should not undergo thermal degradation. Pressurized steam, generated in a separate chamber, is then circulated through the plant material. The heat of the steam forces open

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the tiny intercellular pockets in which the essential oils are contained releasing the oils. During steam distillation, the temperature of the steam must be moderated so that it is high enough to open the oil pouches without destroying the plants, fracturing or burning the essential oil as has been recommended in the literature (Sheridan, 2000).

Some or most essential oils have been found to be heat sensitive and hence thermo degradable. As the tiny droplets of essential oils are released, they evaporate and mingle with the steam, travelling through a pipe into a condenser. The steam and oil vapour are then condensed to a liquid mixture. As the oil-water mixture has been found to be nearly immiscible at a temperature lower than 65°C (Sheridan, 2005). The mixture can be separated using various gravity related techniques. Due to the immiscibility of the oil and water at low temperature, the essential oil can be separated from the water by either decanting off the water or skimming of the oil from the top, as the oil is less dense than water at these conditions. The water obtained as a byproduct of distillation is referred to as floral water or distillate and retains many of the therapeutic properties of the plant. For this reason, floral waters are valuable in skin care for making facing mists and toners and are also preferred to essential oils when treating a sensitive individual or child or when a more diluted treatment is required (Sheridan, 2000). Steam distillation is the most widely accepted process for the production of essential oils on large scale. Throughout the flavor and fragrance supply business, it is a standard practice.

Advantage of steam distillation

- ❖ No thermal decomposition of oil constituents.
- ❖ Amount of the steam can be readily controlled.
- ❖ Amount of steam can be readily controlled.
- ❖ Most widely accepted process for large-scale oil production, superior to the other two processes.

Disadvantage of steam distillation

- ❖ This operation needed significantly more capital investment than the other two methods.

2.7.2. Solvent extraction

Solvent extraction is the most effective method for the recovery of oil (almost 98%), especially with materials with low oil content, like coriander. Solvent extraction is used on delicate plants to produce higher amounts of essential oils at a lower cost (Chrissie, 1996). From an economical point of view in most cases, n-hexane is used as solvent, because it is cheap, has good oil solubility at relatively low temperature, has an appropriate boiling temperature, is non-corrosive to metal, does not react chemically with the oil, is stable under the process conditions, is not mixable with water, which eases the separation of water from the seeds, and it is easily and completely removed from the residue with low energy input and without impairment of the raw oil. The plant is removed and this renders a solvent. The solvent is then boiled off under a vacuum or in a centrifugal force machine to help separate it from the essential oil.

Because the solvent has a lower boiling point than the essential oil it evaporates and the oil is left. The extraction method entails treating the raw material with hexane and recovering the oil by distilling the resulting oil in n-hexane known as miscella. The n-hexane dissolved in the substance is recovered by evaporation and condensation from the distillation of miscella. The hexane thus recovered is reused for extraction. The low boiling point of hexane ($67^{\circ}\text{C}/152^{\circ}\text{F}$) and the high solubility of oils and fats in it are the properties exploited in the solvent extraction process. This process also sometimes yields an aromatic resinous product known as oleoresin, which is more concentrated than essential oils with an even wider application in the food and other industries (Heath, 1981)

2.7.3. Turbo Distillation Extraction

Turbo distillation is ideal for extracting difficult to extract or coarse plant materials like bark, roots and seeds. In this method used for solvent or water-extraction of numerous solid raw materials, mainly vegetal, used in the perfume, flavor, cosmetic, pharmaceutical and spirit and food industries. The plants are submerged in water and steam is circulated by the mixture of plants and water. The same water is recycled through the plant material during the entire process. This method allows faster extraction of essential oils from hard-to-extract plant material (Kumar, 2010). As cited in Kumar(2010), steam distillation is special types 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 steam temperature must be

high enough to vaporize the oil present, but not so high that the plants are destroyed or the essential oils are burned. (Anitescu *et al*) have studied that ripe fruits of coriander sativum L. were extracted by steam distillation and by supercritical fluid extraction, using CO₂ in a two-stage separation system. An inexpensive thermal expansion procedure for supercritical fluid delivery has been developed. (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 spinning-band distillation column, in which the oil is heated to 100°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.

2.7.4. Maceration

Maceration actually creates more of infused oil rather than an essential oil. Plant matter is soaked in vegetable oil, heated and strained before use as massage oil. This approach is undesirable since it alters the oil's composition.

2.7.5. Expression (cold pressing)

Expression or cold pressing, as it is also known, is only applied to obtain essential oils from orange or citrus peel. During this process, the term expression refers to any physical process in which the essential oil glands in the peel are crushed or broken to release the oil. This classical method is still frequently executed by means of rather primitive techniques, but the essential oil produced this way contains more of the fruit odor character than oil produced by any other method.

2.7.6 Effleurage (cold fat)

The principles of effleurage are simple. Certain flowers (e.g. tuberose and jasmine) continue the physiological activities of developing and giving off perfume even after picking. Some flowers, such as jasmine and tuberose, have such low contents of essential oils and are so delicate that heating them would destroy their blossoms before releasing the essential oils.

In these cases, the essential oils are extracted using a costly and time consuming procedure known as effleurage. The essential oils from flower petals are absorbed by trays of odorless vegetable or animal fat (Stahl *et al.*, 1988). At the end of every day or even after a few hours,

when the vegetable or fat has removed as much of the essential oil as possible, the depleted petals are removed and replaced with fresh ones. The essential oils are separated by adding alcohol to the effleurage mixture.

2.7.8. Supercritical fluid extraction

The method of separating one part (the extractant) from another (the matrix) using supercritical fluids as the extracting solvent is known as supercritical fluid separation. It is a substance at a temperature and pressure above its thermodynamic critical point. Additionally, close to the critical point, small changes in pressure or temperature result in large change in density, allowing many properties to be “tuned”. Super critical fluids are suitable as a substitute for organic solvents in a range of industrial and laboratory processes.

Extraction is usually from a solid matrix, but it can also be from liquids. Supercritical fluid extraction can be used as a sample preparation step for analytical purposes, or on a larger scale to either strip unwanted material from a product (e.g. decaffeination) or collect a desired product (e.g. essential oils). Carbon dioxide and water are the most commonly used supercritical fluids, being used for decaffeination and power generation respectively (Wikipedia, 2008). Extraction conditions for supercritical CO₂ are above the critical temperature of 31⁰C and pressure of 74 bars. Supercritical extraction mostly uses carbon dioxide at high pressure to extract the high value products from natural materials. Supercritical fluids possess excellent extractive properties such as high compressibility, liquid-like density, low viscosity, high diffusivity (Lim *et al.*, 2002).

2.7.9. Microwave extraction

Microwave energy is a superior alternative to several thermal applications owing to its efficient volumetric heat production. Volumetric heating or heating of the bulk is more effective, uniform, and less susceptible to overkill or supererogation than moving heat from the surface inwards. The raw material is heated directly by microwaves and this brings about quality consistency and minimizes the impact on the environment as opposed to using fossil fuels or less efficient, indirect electrical heating systems. Specifically in the essential oil extraction, microwave mediated processes are highly desirable due to their small equipment size (portability) and controllability through mild increments of heating. However, so far the microwave technology

has found application in very few industrial bio-processing installations due to the lack of available data on microwave interaction with heterogeneous natural raw materials.

2.7.10 Hydro-diffusion

Although introduced more recently than carbon dioxide extraction, hydro-diffusion is similar to steam distillation except that the steam is produced above the plant material and percolates down through it (Chrissie, 1996). Hydro-diffusion has the advantage of being faster than distillation, particularly for fibrous materials like woods and barks. The resultant oils are reported to have a superior aroma and a richer color obtained by ordinary distillation. Nevertheless, oils captured by hydro-diffusion process are not widely available.

2.8 selections of extraction processes of essential oils

2.8.1 Steam distillation extraction method

Many essential oils are obtained by steam distillation of leaves, seeds, flowers, and other plant materials. In batch and continuous operation, steam distillation refers to a method in which live steam is in direct contact with the distilling device. Parameters for selecting an appropriate extraction method of steam distillation based on the following criteria.

- ❖ Hydrolysis of oil constituents is good.
- ❖ Quality of oil is good.
- ❖ Amount of steam can be readily controlled.
- ❖ Rate of distillation is high.
- ❖ Yield of oil is good if plant material is properly comminuted.
- ❖ Thermal decomposition of oil constituents.

2.9 Important of physical and chemical properties of essential oil

2.9.1 Physical properties

✓ **Refractive index**

Ray of light is bent or refracted toward the usual as it moves from a less dense to a more dense medium. The use of Refractometers allows for the quick and easy determination of this physical constant.

✓ **Specific gravity**

The specific gravity of an essential oil is a significant criterion for its quality and purity. Values for essential oils vary between the limits of 0.696 and 1.188 at 15°C, in general, the specific gravity is less than 1.000(Guenther, 1960). Hence essential oil can be collected over (floating on) water.

✓ **Solubility**

The majority of essential oils are water insoluble and miscible with absolute alcohol. The solubility of oil may change with age. Most of essential oils of commercial interest are steam volatile, reasonably stable to action of heat and practically insoluble in water and hence suitable for processing by steam distillation.

✓ **Boiling range**

In the case of isolates and synthetics, the boiling range is an important criterion of purity.

2.9.2 Chemical composition of coriander

The chemical composition of essential oils commonly comprises one or two predominating compounds that define the oil characteristics, although minor compounds may constitute an important contribution to the specific scent of the volatile oil. The composition is highly dependent on the plant species but it is also known to be widely variable within one species, rendering several chemo types of essential oils of a specific plant based on its genetic background. These differences in the chemical composition of essential oils are further due to differences in the environment, such as the surrounding plant population, or the plant development stage.

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The coriander constituents have been divided into two major parts based on their chemical properties: (i) Isoprenoids & (ii) Non-Isoprenoids. The former is considered as the main class while the latter comprises less dominant component such as aliphatic compounds, aromatics, alcohols & esters. Even though these compounds may be present in very small amounts, they can represent a great contribution to the flavor or bioactivity of the volatile oil.

i. Non-isoprenoids(Non- terpenoid)

Short-chain aliphatic substances, aromatic substances, nitrogenized substances, and Sulphur-containing substances all under this category. They are less important than terpenoids in terms of uses and applications.

ii. Isoprenoids (terpenoid)

Essential oils are made up of three elements almost exclusively carbon, hydrogen, and oxygen. Terpenes, also known as isoprenes, and a terpene containing oxygen is called terpenoid. Terpenes are chemical compounds found in essential oils, resins, and other aromatic plant materials (pines, citrus fruit, etc.). They are usually found in monoterpene oils (C_{10}), sesquiterpenes (C_{15}) and diterpenes (C_{20}). Their most important structure feature is the presence in them of several skeleton at least two isoprene units to 5 carbon atoms each (C_5H_8).

The oxygenated compound (mainly esters, aldehydes, ketones, alcohols, phenols) and oxides; acids, lactones, Sulphur, and nitrogen compound are sometimes also present.

A. monoterpenes

Monoterpene compounds are presented in nearly all essential oils and are formed from two isoprene units with at least a double bond (structure of ten carbon atoms). They are the major molecules consisting of 90% of essential oil; they contribute to the specific smell of plants. While, hemiterpenes only contain one such unit, they quickly react to air and heat and consequently lack stability and long shelf life as they are quickly oxidized.

B. sesquiterpenes

Sesquiterpenes consist of 15 carbon atoms or 3 isoprene units linked to each other, head to tail. Mono- and sesquiterpenes are the most abundant classes in essential oils but less volatile than

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monoterpenes. This formation can produce more than 300 different hydrocarbon sesquiterpenes. Sesquiterpenes have great diversity in construction containing up to four carbocyclic rings.

C. Diterpenes

Diterpenes are less commonly found in essential oils, although there are some compounds that are most prominent. They have 20 carbon atoms (4 isoprene units) and are derived from geranylgeraniol pyrophosphate. They are of fungal or plant origin and are found in resins, gummy exudates, and in the resinous high boiling fractions remaining after distillation of essential oil.

Table 2-2 structure of terpenes

Terpene classification	No isoprene units	No of carbon atoms
Monoterpenes	2 isoprene	10 carbon atoms
Sesquiterpenes	3 isoprene	15 carbon atoms
Diterpenes	4 isoprene	20 carbon atoms
Triterpenes	6 isoprene	30 carbon atoms
Tetraterpenes	8 isoprene	40 carbon atoms

D. Esters

The most widespread group found in essential oil, which includes linalyl acetate (found in lavender, coriander seed, clary sage) and geranyl acetate (found in sweet marjoram). Methyl salicylate, a derivative of salicylic acid and methanol, is an anti-inflammatory compound similar to aspirin and is found in a certain type of heather (*Gaultheria procumbens* Ericaceae family).

E. Aldehyde

Properties: anti-fungal, anti-inflammatory, anti-septic, anti-viral, bactericidal, disinfectant medicinally, essential oils containing aldehydes are effective in treating candida and other fungal infections disease. Aldehydes have an oxygen atom double-bonded to a carbon atom at the end of a carbon chain. Aldehydes have a slightly fruity odor when smelled on their own. Geraniol and cumin aldehyde are examples of important aldehyde.

F. Ketones

Properties: anti-catarhal, cells proliferate, expectorant, vulnerary. Ketones often are found in plants that are used for upper respiratory complaints. Some of the most common toxic constituents are ketone such as thujone found in mug wort, tansy, sage and wormwood; and pulegone found in pennyroyal and buchu. It can be produced by the oxidation of secondary alcohols. A ketone is extracted from an alcohol by oxygenation and has an oxygen atom double-bonded to a carbon atom, which is also bonded to two other carbon atoms.

G. Alcohol

Alcohols contain the hydroxyl functional group and perhaps the most varied group of terpene derivatives found in essential oils. Monoterpene alcohols are not large in number, but occur in large number of essential oils. There are many sesquiterpenes alcohols, but most of them are found in few essential oils. Alcohols are relatively nontoxic non-mutagenic, and possess low irritancy and allergenicity. Monoterpenes are good antiseptics, with antifungal properties.

H. Phenol

These have a bactericidal and relaxing effect, but they can irritate the skin. Common phenols include eugenol (found in clove and west India bay), thymol (found in thyme), carvacrol (found in oregano and savory).

2.10 Factor affecting yield and quality of essential oil

Various process parameters influence the yield and quality of coriander essential oil generated by steam distillation. Some of the factors that affect the yield and quality of coriander essential oil such as:

2.10.1 Nature of raw material

Nature of raw material is important because some materials like roots and seed will not yield essential oil easily if distilled in their natural state. to show their oil cells these materials must be crushed, powdered or immersed in water. Chopping of plants will also change the packing density of the material when placed in the distillation still.

2.10.2 Material of construction of equipment

Essential oils which are corrosive in nature should be preferably distilled in stills made of resistant materials like Aluminum, copper or stainless steel. The tank still can be made from a cheaper metal like mild steel or galvanized iron, and the condenser and separator can be made from a resistant material like stainless steel. As only vapor is present in tank still, the rust and other products of corrosion may not be carried over into the oil. Although copper was the most common material of fabrication of distillation stills since ancient times, its availability is getting reduced and with the arrival of superior alloys like stainless steel, it is slowly disappearing from the scene.

2.10.3 Mode of distillation

Technique for the distillation should be chosen on basis of oil boiling point and nature of herb as the heat content and temperature of steam can alter the distillation characteristics. For high boiling oils such as root oils (e.g. cyperus) and woody (e.g. sandalwood, cedar wood), the oil should be extracted using boiler-operated steam distillation. Since the heat content and temperature of steam depend upon its pressure, a change in steam pressure can alter the distillation characteristics. High-boiling constituents of essential oils normally require high pressure steam to distill over.

2.10.4 Distillation time

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

2.10.5 Geographical variation

There are many reports in the literature showing the variation in the yield and chemical composition of the essential oil with respect to geographical regions (Uribe-Hernandez 1992). Differences in the yield and chemical profile of essential oils obtained from various locations. Such differences could be linked to the varied soil textures and possible adaptation response of different populations, resulting in different chemical products being formed, without morphological differences being observed in the plant (Hussian *et al.*, 2008).

2.10.6 Improper design of equipment

Improper tank, condenser, or separator design can result in oil loss and high capital costs. The height-to-diameter ratio of the tank is crucial. Similarly the use of a condenser with an improper design and without calculating the heat transfer areas based on the steam generation areas will lead to improper condensation and loss of oil.

2.10.7 Particle size of the raw material loading to the chamber

The size of the seeds has its own contribution on the yield of the extract oil. The particle size should be optimum in order to steam is distributed properly trough the chamber.

2.10.8 Operating parameters

Operating parameters proper control of injection rates and pressure in boiler-operated units is necessary to optimize the temperature of extraction for maximal yield. Generally, high-pressure steam is not advisable for the distillation of essential oils. The temperature of the condensate should not be high as it can result in oil loss due to evaporation.

3. Materials and Methods

3.1 Materials and Equipment

Main Raw material:

Coriander seed (*Coriandrum sativum* L.)

Equipment:

The Equipment used for the experiment work were oven, cross beater mill, sieve ,digital weighting balance , steam distillation set up , separating funnel , flask , thermometer, pH meter, viscometer, Gas chromatography – Mass spectroscopy, refractometer, water bath, beaker and pycnometer.

Chemicals: used for characterization of coriander oil were, distilled water, ethanol, sodium hydroxide, phenolphthalein indicator, oxalic acid, potassium hydroxide, HCl, iodine bromide solution, potassium iodide.

3.2 Raw material preparation

Coriander seed was purchased from Merkato market which is found in Addis Ababa, Ethiopia. The impurities like, stone, soil and dust were removed by hand. The moisture of the seed was removed using oven.

3.3 Sample characterization

3.3.1 Determination of moisture content

15g, 20g,25g of the cleaned sample seed was weighed and dried in an oven at 105°C and the weight was measured every 2hrs. The procedure was repeated until the weight remained unchanged. The percentage moisture in the seed was calculated using the following formula.

$$\text{Moisture \%} = \frac{(W_1 - W_2) * 100}{W_2 * 100} \quad (3.1)$$

Where: W_1 : original weight of the sample before drying;

W_2 : weight of the sample after drying.

3.3.2 Size reduction and sieve analysis of the coriander seed

The dried coriander seed were grind in cross beater mill after the moisture was removed by the oven. The sample was sieved using vibrating shaker with set of sieves sizes arranged in ascending order 1mm, 1.4mm, 1.8mm, 2mm to obtain particular sizes of 1-1.4mm, 1.4- 1.8mm and 1.8-2mm. This was aimed to investigate the effect of particles size on yield and quantity of oil.



Figure 3-1 Cross beater mill

3.3.3 Extraction method of coriander oil

Experimental work of coriander oil is conducted using steam distillation method. There are many process parameters which may affect the extraction of oil using steam distillation such as extraction time, particle size, extraction temperature etc. but it is quite difficult to study the effect of all these parameters. as result this thesis focused on the effect of extraction time and particle size at constant temperature of 85°C and atmospheric pressure of 1atm was used as starting parameter. The procedure of experimental work, 2 kg of coriander seed was used, at three different extraction time and particle size interval; 2hr, 3hr and 4hr with various particle sizes of 1-1.4mm, 1.4-1.8mm, and 1.8-2mm with three replications to get the highest yield.

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The extraction chamber was cleaned before the procedure began, and then the coriander seed was added to the column. After feeding the raw material and opening the condenser water valve. Steam was generated by heating water in steam boiler, and it was then passed through the first still, with contained the raw materials. The boiler was used for to generate steam and let to enter into the column to extract the required yield of coriander oil. Condenser consist two connecting tubes. The outer tube was filled with water while the inner spiral tube was filled with a mixture of steam and oil. In the collector, the diluted mixture of oil and water was collected. Finally in the separating funnel, the oil and water mixture was segregated. The steam distillation set up show below.



Figure 3-2 Steam distillation Used

3.4 Determination of the yield of coriander oil extraction

$$\% \text{ Yield} = \frac{W_1 \cdot 100}{W_2} \quad (3.2)$$

Where: W_1 : weight of oil and

W_2 : weight of coriander seeds

3.5 Characterization of coriander oil

3.5.1 Physicochemical properties of coriander oil

The physical properties of extraction oil are specific gravity, refractive index, viscosity; pH etc. acid value, iodine value, saponification value, and oil composition are chemical properties of coriander oil that can be identified using GC-MS. All the parameters are determined using European pharmacopeia system (European Pharmacopoeia Commission, 2001).

3.5.1.1 Determination of specific gravity

Density bottle methods were used to calculate the density of the oil. A 25ml clean and dry bottle was weighted, then the bottle was filled with the oil, the stopper was inserted, and the bottle was reweighed to give (W_1). The oil was substituted with water after washing and drying the bottle and weighed to give (W_2). The expression for specific gravity is:

$$\text{Sp. gr} = \frac{(W_1 - W_0)}{(W_2 - W_0)} = \frac{\text{mass of sample}}{\text{mass of an equal volume of water}} \quad (3.3)$$

3.5.1.2 Determination of pH

2ml of coriander oil was poured into a clean dry 25ml beaker and 13ml of hot distilled water was added into the beaker and stirred slowly. Then it was cooled to 25⁰C in cold water. After standardizing the pH electrode with a buffer solution, the electrode was immersed in the sample and the pH was read and registered.

3.5.1.3 Determination of Refractive index

The ratio of the speed of light in vacuum to the speed of light in the material is the refractive index of a substance. Coriander oil of Refractive index was measured using refractometer at 25⁰C. first the cuvet was cleaned next filled with coriander oil then put on the prism for refractive index determination. The value is calculated as:

$$N = \tan(\theta b) \quad (3.4)$$

Where: N is refractive index and θ is angle of the rotation

3.5.1.4 Determination of viscosity of the oil

45ml of oil was poured into a test tube and a viscometer was used to measure the viscosity at a temperature of 35⁰C.

3.5.1.5 Determination of boiling temperature of the oil

25ml of coriander oil was poured into beaker, and a thermometer was inserted and put on heating mantle. It was found that the oil in the beaker began to circulate, causing the oil to boil and thermometer to read the temperature.

3.5.1.6 Solubility of coriander oil

By dissolving 2ml of coriander oil in 10ml of alcohol and water, the solubility of the oil was determined.

3.5.1.7 Determination of saponification value

1g of coriander oil was placed into a 500ml conical flask Indicator method was use as specified by ISO 3657(1988).2g was accurately weighted then and 25ml of alcoholic potassium hydroxide (0.5N) was added and the mixture allowed to boiling gently for about 60min.with shaking ,at regular intervals of 5min.the mixture was reflux for 2hrs then cooled. Few drops of phenolphthalein indictor were applied to the warm solution, and the end point was reached by titrated with 0.5 M HCl before the pink color became colorless. After that, saponification value was determined using the following equation:

$$\text{saponification value(SV)} = 56.11 * \frac{N*(V_0-V_1)}{M} \quad (3.5)$$

Where V_0 : The volume of the solution used for blank test; N: Actual normality of the HCl used; V_1 : the volume of the solution used for determination Saponification value;

M: Mass of the sample

3.5.1.8 Determination of acid value

2g of coriander oil was placed in to a 250ml conical flask. Then 25ml of absolute ethanol alcohol and 1ml of phenolphthalein indicator solution were added. The resulting mixture was heated while shaking in water bath set at 65⁰C for 10minutes to dissolve the oil. Once the oil was cooled, it was titrated against 0.1N potassium hydroxide solution until dark pink color appears. Finally, the volume of KOH solution used was recorded. The acid value is determined using the following equation.

$$\text{Acid value(A. V)} = \frac{56.11*V*C}{m} \quad (3.6)$$

Where: - 56.11: Molecular weight of potassium hydroxide, m: Sample weight

V: Volume of potassium hydroxide (ml), C: Concentration of potassium hydroxide,

3.5.1.9 Determination of Iodine value

The amount of iodine consumed is determined by titrating the iodine released (after adding KI) with a standard Thiosulphate. procedure: 0.1gm of the sample was weighed into a conical flask and 20ml of carbon tetra chloride was added to dissolve the oil. After that, 25ml of iodobromide solution was applied and left to stand in the dark for 30 minutes. Again 30ml of 15ml of 10% potassium iodide and 100ml of distilled water were added and the liberated iodine was titrated with 0.1 Normality solution of sodium thiosulphate with constant shaking. When iodine color became quite pale, few drop of 1% starch solution was added and the titration continued by adding thiosulphate drop wise until the blue color was discharge. The same procedure was used for blank test and other samples. The iodine number was determined using the formula:

$$\text{Iodine value(I.V)} = 12.69 * \frac{C*(V_1-V_2)}{M} \quad (3.7)$$

Where:-C: Concentration of sodium thiosulphate used; V₁: Volume of sodium thiosulphate used for blank; V₂: Volume of sodium thiosulphate used for determination, M: Mass of the sample

3.6 Fourier transform infrared ray (FT-IR)

Fourier transform infrared ray (FT-IR) one of the most important analytical techniques available to analyze various substances. The FT-IR spectrum of the coriander oil was collected at Addis Ababa University using Perkins Elmer spectrum 65 FT-IR spectrometer in and functional groups were calculated using IR correlation charts. The % transmittance of the IR spectrum was reported. The wave number region for the analysis was 4000-400 cm⁻¹ in the mid-infrared range (nurrulhidayah et al., 2011).

A FTIR spectrophotometer was used to determine the chemical functional groups present in the surfactant. Different functional groups are susceptible to absorb characteristic frequencies of IR radiation.

3.7 Gas chromatography – mass spectroscopy

The components of a mixture are separated by gas chromatography, and each component is individually characterized by mass spectrometry. The combination of the two techniques allows for both qualitative and quantitative evaluations of a sample containing a number of organic compounds. The GC-MS analysis using HP 5890 series GC equipped with mass selective detector (MSD), HP 5972 series (German) in LIDI, Addis Ababa, Ethiopian. Helium was used as carrier gas at a constant flow of 1ml/min and an injection volume of 1 μ l will be employed, injector temperature 250⁰C and ion source temperature 280⁰C. The detected compounds were classified by analyzing the raw GC-MS data and comparing in to a mass spectral database maintained by the national institute of standard and technology (NIST, USA), as well as retention times and mass spectra of standard compounds. Relative amounts of detected compounds were calculated based on GC peak areas (Akolade, Jubril Olayinka et al. 2012).

3.8 Design of the Experiment

Data analysis has performed by DESIGN EXPERT 7.0 software using general factorial design methods. Factorial designs are the most efficient for experiments that involve two or more factors. For steam distillation extraction there were two factors; extraction time and particle size with three levels and three replications each. This experiment design allows us to distinguish between the importance of the key and interaction variables. The mathematical model that will explain the effects of the key and interaction factors on the response was also developed using this program software.

4. RESULTS AND DISCUSSION

4.1 Determination of moisture contents

By taking coriander sample 15g, 20g and 25g the average moisture content was calculated using equation (3.1). The following table shows the moisture content of the sample.

Table 4-1 Moisture content determination of coriander seed

	Drying time(hours)							% moisture content
	0	2	4	6	8	10	12	
Sample	15	13.764	13.635	13.602	13.582	13.543	13.543	9.711
Weight	20	18.48	18.351	18.201	18.153	18.102	18.101	9.485
(g)	25	22.744	22.695	22.631	22.540	22.523	22.523	9.908

The moisture content of the coriander seeds weighing 15, 20, and 25gms was 9.711, 9.485 and 9.908%, respectively. The three samples will have an average moisture content of 9.701%.

4.2 Experimental design analysis result

The experimental design selected for this study is the general factorial method design-expert 7.0 software was used to determine the optimal conditions of coriander oil extraction. There two selected extraction factors were extraction time and particle size. Other variables, such as temperature and pressure, were held determined by the literature. These factors were taken as constant due to the uncontrollability of the parameters in the equipment used during the experiment. Every experiment in this study used 2kg of coriander seed. The yields of coriander oil are show in the table below.

Optimization and Characterization of Essential Oil from Coriander Seed

Table 4-2 Experimental result of coriander oil yield extracted using steam distillation

Std	Run	Block	Factor 1 A:time hr	Factor 2 B:particle size mm	Response 1 Yield %
4	1	Block 1	3	1-1.4	0.67
19	2	Block 1	2	1.8-2	0.05
18	3	Block 1	4	1.4-1.8	0.7
1	4	Block 1	2	1-1.4	0.47
16	5	Block 1	4	1.4-1.8	0.68
27	6	Block 1	4	1.8-2	0.38
17	7	Block 1	4	1.4-1.8	0.69
20	8	Block 1	2	1.8-2	0.04
26	9	Block 1	4	1.8-2	0.4
8	10	Block 1	4	1-1.4	0.84
5	11	Block 1	3	1-1.4	0.66
21	12	Block 1	2	1.8-2	0.02
15	13	Block 1	3	1.4-1.8	0.5
13	14	Block 1	3	1.4-1.8	0.51
22	15	Block 1	3	1.8-2	0.21
12	16	Block 1	2	1.4-1.8	0.32
7	17	Block 1	4	1-1.4	0.85
3	18	Block 1	2	1-1.4	0.48
9	19	Block 1	4	1-1.4	0.83
25	20	Block 1	4	1.8-2	0.39
23	21	Block 1	3	1.8-2	0.22
10	22	Block 1	2	1.4-1.8	0.33
2	23	Block 1	2	1-1.4	0.49
11	24	Block 1	2	1.4-1.8	0.31
24	25	Block 1	3	1.8-2	0.2
6	26	Block 1	3	1-1.4	0.65
14	27	Block 1	3	1.4-1.8	0.52

From the table, The maximum extraction of coriander oil is 0.85 % was obtained when the particle size ranged from 1-1.4mm and the extraction time was 4hrs, and the minimum yield was obtained at maximum particle size and minimum extraction time.

4.3. Statically Analysis

Table 4-3 Analysis of variance ANOVA for coriander oil using steam distillation extraction

Source	Sum of Square	DF	Mean square	F- value	P-value Prob>F	
Model	1.52	4	0.38	3557.79	<0.0001	Significant
A-time	0.59	2	0.29	2748.92	<0.0001	
B-particle size	0.93	2	0.47	4366.65	<0.0001	
Residual	0.002348	22	0.0001067			
<i>Lack of Fit</i>	0.0002815	4	0.00007037	0.61	0.6588	<i>Not significant</i>
<i>Pure Error</i>	0.002067	18	0.0001148			
Cor Total	1.52	26				

The value 3557.79 for model is significant. Due to noise, there is only a 0.01% probability that a "Model F – value" this high would occur. Values of "Prob >F" less than 0.0500 indicate model terms are significant. In this case A-time, B-particle size is significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model. the "Lack of Fit F-value" of 0.61 indicates that there is Lack of Fit. In comparison to the pure mistake, this is not significant. a "Lack of Fit F-value has a 65.88% risk of occurring. This huge difference may be due to noise. A minor difference in fit is acceptable --we want the model to fit.

Table 4-4 Model adequacy measures

Std.Dev.	0.010	R-Squared	0.9985
Mean	0.46	Adj R-Squared	0.9982
C.V.	2.25	Pred R-Squared	0.9977
PRESS	0.003537	Adeq Precision	181.943

The model was tested for adequacy by analysis of variance. the "Pred R- Squared" of 0.9977 is in reasonable agreement with the "Adj R-Squared" of 0.9982. "Adeq Precision" measures the

signal to noise ratio. It is preferable to have a ratio of more than 4. The ratio of 181.943 suggests that the signal is sufficient in this case. The design space can be navigated using this model.

Their coefficients of determination could be used to assess the consistency of the model produced. The value of R-Squared for the developed correlation is 0.9985. It implies that 99.85% of total variation in the percentage yield of coriander seed oil is attributed to the experimental variables studied.

Final equation in terms of coded factors:

$$\text{Yield} = 0.46 - 0.18 * A[1] + 0.0003704 * A[2] + 0.20 * B[1] + 0.047 * B[2] \quad (4.1)$$

Where: A [1] = the difference of time level -1 from the overall average.

A [2] = the difference of the time level-2 from the overall average.

B [1] = the difference of particle size level-1 from the overall average.

B [2] = the difference of particle size level-2 from the overall average

Diagnostics plot

Normal probability plot versus the residuals

Normal probability plot versus the residuals results shows us that the regression model equation provided a very accurate description of experimental data, in which all the data points are very close to the line of perfect fit.

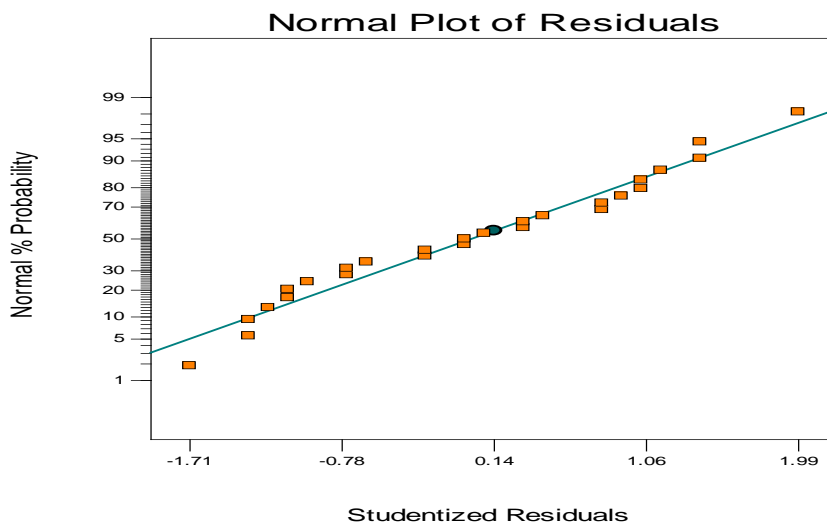


Figure 4-1 Normal plots of residuals

Studentized Residuals versus predicted values

From the figure was show that the Studentized Residuals versus the predicted response value tests the assumption of constant variance. The constant range of residuals across the graph is appealing, as it eliminates the need for transformation in order to reduce personal error.

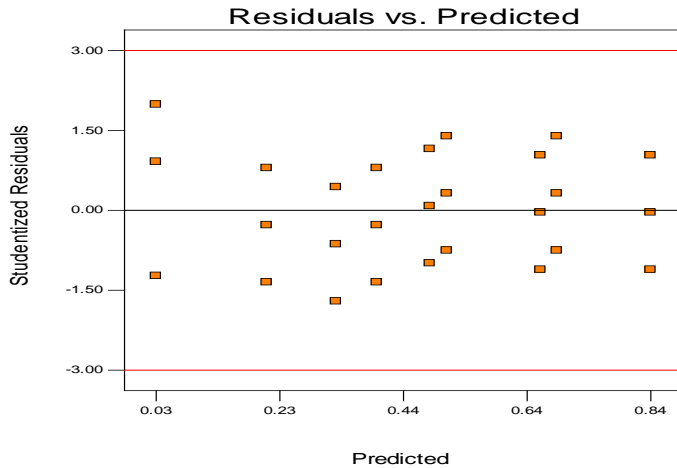


Figure 4-2 Studentized Residuals versus predicted values of yield coriander seed oil

Predicted value versus Experimental value

The graph below depicts the relationship between the real value of the experiment and the value expected by the design expert software model equation.

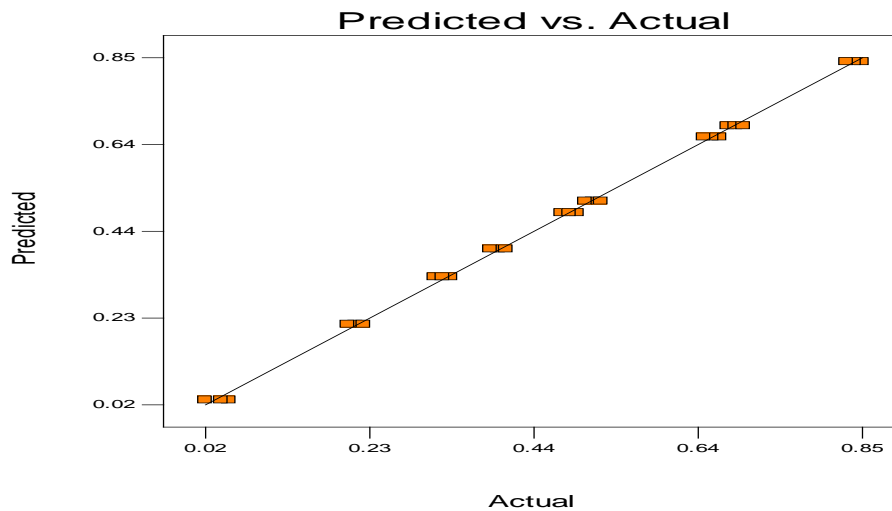


Figure 4-3 Predicted vs. Experimental value of yield for steam distillation

✚ Studentized residuals versus run numbers

The following figure shows the relation between Studentized Residuals versus Run numbers were not have a uniform structure which tells us that the experiment was conducted at a randomized design.

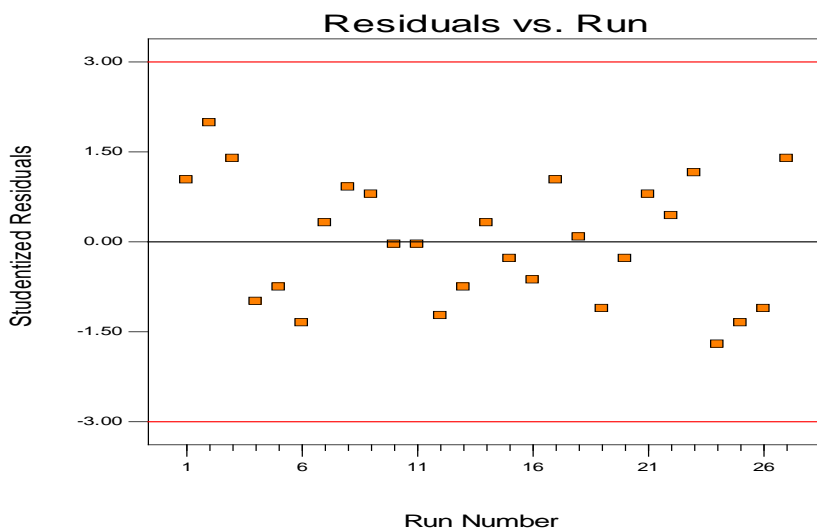


Figure 4-4 Studentized residuals versus run numbers for yield of coriander seed oil

4.4. Individual effect of each factor on the yield of extracted oil

4.4.1. Effect of particle size on percent yield of oil

The yield of coriander oil produced by steam distillation is heavily influenced by particle size. There was an increasing in the oil yield to maximum value due to reduce in particle size and a further increase in the particle size results in a drop in oil yield. That is why larger particle size with smaller contact surface area has more resistant to steam entrance and carried out. Therefore, in contrast to smaller ones, less oil would be moved from within to the surrounding vapor. As a result increasing particle size reduces oil yield. Nevertheless, when the particle is very fine particle size i.e., below <1mm, the extracted oil become small in its amount, even though the contact surface area for small particle is supposed to be significantly higher than that for the larger particles. in the figure it was observed that the minimum particle size has had maximum oil yield whereas the maximum particle size has had minimum oil yield.

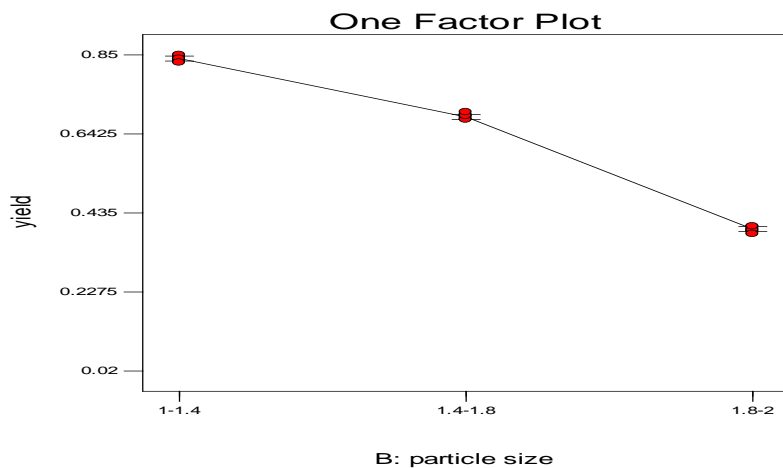


Figure 4-5 Effect of particle size on the yield of extraction oil

4.4.2. Effect of time on percent yield of oil

Extraction time is one of the factors that affect the percent yield of coriander oil. The experimental result shows extraction time influences greatly the percentage yield of coriander oil using steam distillation. The contact time increase, so does the oil yield, until the oil transfer from the coriander seed to the steam reaches zero. In other word, when the maximum amount of extracted oil is obtained, the oil yield level remains invariable even by extending the reaction time. In general; the rate of extraction was high at first but it gradually slowed as time went on.it was found that 4hr has the optimum extraction time for this study. The percentage oil yield was directly related to extraction time. Therefore, as extraction time increase the yield also increase.

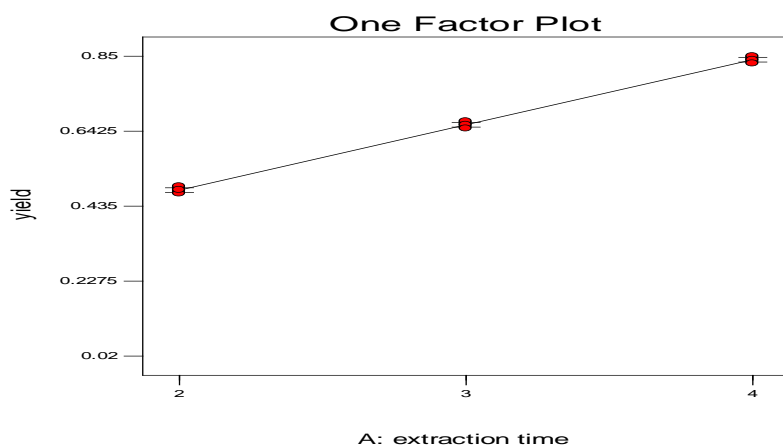


Figure 4-6 Effect of extraction time on the yield of extraction oil

4.5 Interaction effect of factors on the yield of extracted oil

From design expert software the output of interaction effect between particle size and extraction time

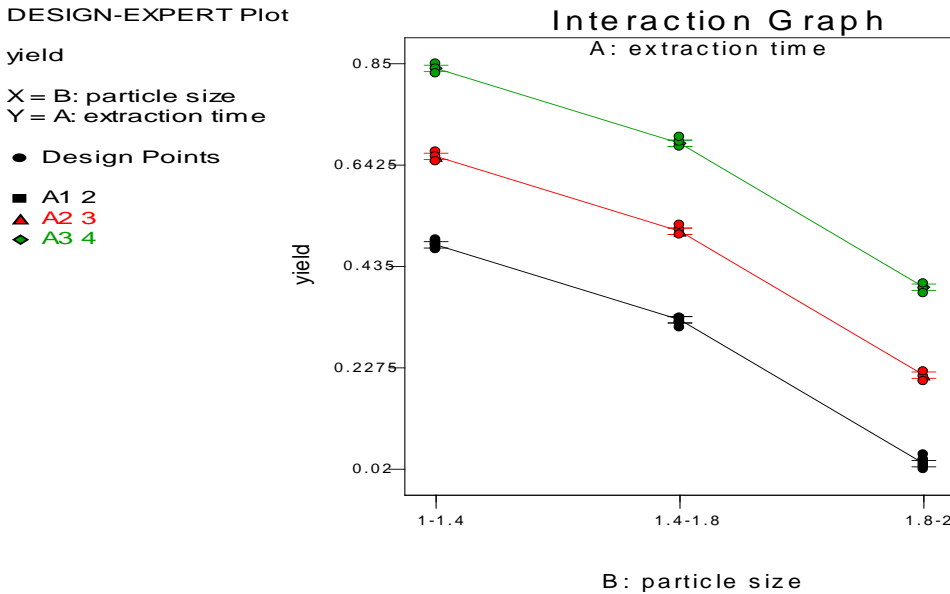


Figure 4-7 Effect of time, particle size and their interactions on coriander oil yield

From the graphical representation Figure 4-6 shows that the extraction time and particle size have significant effect on the extraction yield and also there were no interaction among each factor. This shows us higher extraction time with lower particle sizes give a higher yield and higher particle size with lower extraction time can give lower yield.

Where: A1, A2 and A3 are codes for time of 2, 3 and 4 hours respectively.

B1, B2 and B3 are ranges of particle size from 1-1.4, 1.4-1.8 and 1.8-2mm respectively.

Difference between actual (experimental) value and predicted value

Describe the difference between the actual values of the model and the predicted one was shown in the following table 4.5

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Table 4-5 Difference between actual (experimental) value and predicted value

standard	Actual value %	Predicted value %	Residual
1	0.47	0.48	-0.0092
2	0.49	0.48	0.011
3	0.48	0.48	0.0074
4	0.67	0.66	0.0096
5	0.66	0.66	-0.00037
6	0.65	0.66	-0.01
7	0.85	0.84	0.0096
8	0.84	0.84	-0.00037
9	0.83	0.84	-0.01
10	0.33	0.33	0.0040
11	0.31	0.33	-0.016
12	0.32	0.33	-0.0059
13	0.51	0.51	0.0029
14	0.52	0.51	0.013
15	0.50	0.51	-0.0070
16	0.68	0.69	-0.0070
17	0.69	0.69	0.0029
18	0.70	0.69	0.013
19	0.05	0.031	-0.011
20	0.04	0.031	0.0085
21	0.02	0.031	-0.011
22	0.21	0.21	-0.0025
23	0.22	0.21	0.0074
24	0.20	0.21	-0.013
25	0.39	0.39	-0.0025
26	0.40	0.39	0.0074
27	0.38	0.39	-0.013

4.6 Optimization

It was predicted that at the following operating condition 1-1.4mm particle size, 4hr extraction time, a maximum oil yield was obtained using the optimization feature in design expert software 7.0.0. The aim is to maximize economic benefit or increasing the yield of coriander oil by minimizing process cost.

Table 4-6 optimum possible solutions

Number	Time	Particle size	Yield	Desirability	Remark
1	4	1-1.4	<u>0.84037</u>	<u>0.988</u>	<u>Selected</u>
2	4	1.4-1.8	0.687037	0.804	
3	3	1-1.4	0.66037	0.772	
4	3	1.4-1.8	0.507037	0.587	
5	2	1-1.4	0.479259	0.553	
6	4	1.8-2	0.392593	0.449	
7	2	1.4-1.8	0.325926	0.369	
8	3	1.8-2	0.212593	0.232	
9	2	1.8-2	0.0314815	0.014	

The desirability scale ranges from 0 to 1, and it measures how close an answer is to its ideal value. The desirability of a response is 0 if it falls within the unacceptable intervals, and 1 if it falls within the optimal intervals or the response reaches its ideal value. The more closely the response approaches the ideal intervals or ideal values, the closer the desirability is to 1. the above analysis maximum yield coriander oil were 0.84037% at a time of 4hr, particle size 1-1.4mm and the value of desirability obtained was 0.988%

4.7 Determination of physical properties of the oil

✓ Refractive index

The value of refractive index coriander oil was determined in bio-engineering lab using refractometer to determine the refractive index. The lower the refractive index is the higher the quality of essential oil (Anhwange *et al*, 2010).

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✓ Specific gravity

Weight in gram of empty pycnometer (W_0) at 25°C = 16.5g

Weight in gram of pycnometer with coriander oil (W_1) at 25°C = 42.84g

Weight in gram of pycnometer with coriander water (W_2) at 25°C = 46.5g

$$\text{Sp. gr} = \frac{W_1 - W_0}{W_2 - W_0} = \frac{42.84 - 16.5}{46.5 - 16.5} = 0.878$$

✓ pH value

The pH of coriander essential oil was determined using a pH meter in environmental laboratory of chemical Engineering Department. Its value was recorded as 6.2

✓ Boiling point of coriander oil

The boiling point of coriander oil was measure by procedure described on the methodology. Its value was 131°C.

The physical properties of coriander oil was summarized in table

Table 4-7 physical properties experimental result of coriander oil

Physical property	Value	Unit
Refractive index at 25°C	1.4699	-
Boiling point at room temp.	131	°C
Viscosity	35	Mpas
PH value	6.2	-
Specific gravity	0.878	-
Color	Pale yellow liquid	-
Odor	Pleasant smell	-
Solubility in water	Insoluble	-
Solubility in alcohol	Soluble	-

4.8 Quality evaluation of the coriander oil

✓ Saponification value determination

The saponification value is the number of 0.5N alcoholic potassium hydroxide required to saponify 1 g of oil/fat. Analytical importance of saponification value is used as a measure of the average molecular weight of the fatty acids in the glycerides that make up a fat. Lower saponification value means the higher the fatty acid molecular weight, the higher the glyceride molecular weight, and vice-versa. Saponification value of oil is important parameter in determining the suitability of oil in soap making.

Saponification value of coriander oil calculated by equation 3.5

$$\begin{aligned} \text{S.V} &= 56.11 * \frac{N * (V_0 - V_1)}{M} \\ &= 56.11 * \frac{0.5 * (26.4 - 25.8) \text{ml}}{1\text{g}} = 16.833 \text{ ml/g} \end{aligned}$$

✓ Iodine number determination

The iodine value is used to determine the unsaturated of oils and in assessing the stability of oil in industrial application. The lower the iodine value of oil, which represents its characteristics such as higher oxidation resistance, longer shelf life, and higher quality, the higher the quality, while the higher the iodine value of oil, the lower the quality. The iodine number value was calculated using equation 3.7.

$$\begin{aligned} \text{iodine value(I.V)} &= 12.69 * \frac{C * (V_1 - V_2)}{M} \\ &= 12.69 * \frac{0.1 * (11.5 - 9) \text{ml}}{0.1\text{g}} = 31.725 \text{ ml/g} \end{aligned}$$

✓ Acid value determination

Acid value is the measure of total acidity of the lipid involving contributions from all the constituent fatty acids that make up the glyceride molecule (Ekpa and Ekpe, 1995). The acid value was determined using the titration process.

The acid value calculated by equation 3.6 as follow:

$$\begin{aligned}\text{acid value(AV)} &= \frac{56.11 * V * C}{m} \\ &= \frac{56.11 * 7.5 \text{ml} * 0.1}{2 \text{g}} = 21.0375 \text{ ml/g}\end{aligned}$$

$$\begin{aligned}\text{Free fatty acid} &= \frac{\text{Acid value}}{2} \\ &= \frac{21.0375}{2} = 10.51875 \text{ ml/g}\end{aligned}$$

Chemical properties of coriander oil such as saponification value, iodine number and acid value are summarized in table.

Table 4-8 Chemical properties of coriander oil extracted using steam distillation

Chemical properties	Value	Unit
Saponification value	16.833	ml/g
Iodine number	31.725	ml/g
Acid value	21.0375	ml/g

4.9 Determination of the functional groups present using FT-IR

By comparing the vibration frequencies in wave numbers of the sample spectrograph obtained from an FT-IR spectrophotometer with those of an IR correlation chart, the functional groups present in the essential oil, were determined.

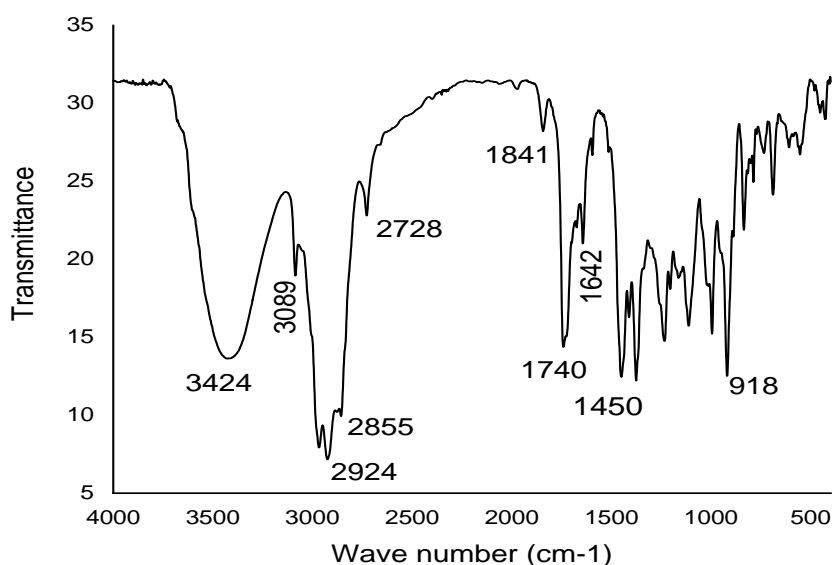


Figure 4-8 FT-IR graph analysis of functional group result

From the above graph based on the peaks the functional groups the unsaturated fatty acids $C=C$ stretches, The $O=C-H$ stretches in both aldehydes in the region $2830-2695\text{ cm}^{-1}$, the carbonyl stretch $C=O$ of a carboxylic acid appears as an intense band from $1760-1690\text{ cm}^{-1}$. $N-H$ stretch $3400-3250\text{ cm}^{-1}$. 1° amine: two bands from $3400-3300$ and $3330-3250\text{ cm}^{-1}$. 2° amine: one band from $3350-3310\text{ cm}^{-1}$. 3° amine: no bands in this region. $N-H$ bend (primary amines only) from $1690-1715\text{ cm}^{-1}$, $R_2C=CH_2$ Acids, fatty acids (aromatic amines) from $1335-1250\text{ cm}^{-1}$, $C-N$ stretch (aliphatic amines) from $1250-1020\text{ cm}^{-1}$. $N-H$ wag (primary and secondary amines only) from $910-665\text{ cm}^{-1}$. The region from $900-650\text{ cm}^{-1}$. Aromatics, alkyl halides, carboxylic acids, amines, and amides show moderate or strong absorption bands (bending vibrations) in this region. All spectra contains peaks attributable to unsaturated fatty acids in $1690\text{ cm}^{-1} - 1715$ and peaks attributable to unsaturated $C-H$ asymmetric stretching for fatty acids, which shows the acidity of the oil highly concentrated (Sharma et al. 2004; Xing et al. 2007).

4.10 GC – MS analysis

A total of eleven components, with different retention time were eluted from the GC column, as indicated by the chromatogram and were further analyzed with an electron impact mass spectroscope voyager detector. The mass spectrographs of the identified constituents are given in table. The relative amount of individual components was calculated based on GC peak areas.

Optimization and Characterization of Essential Oil from Coriander Seed

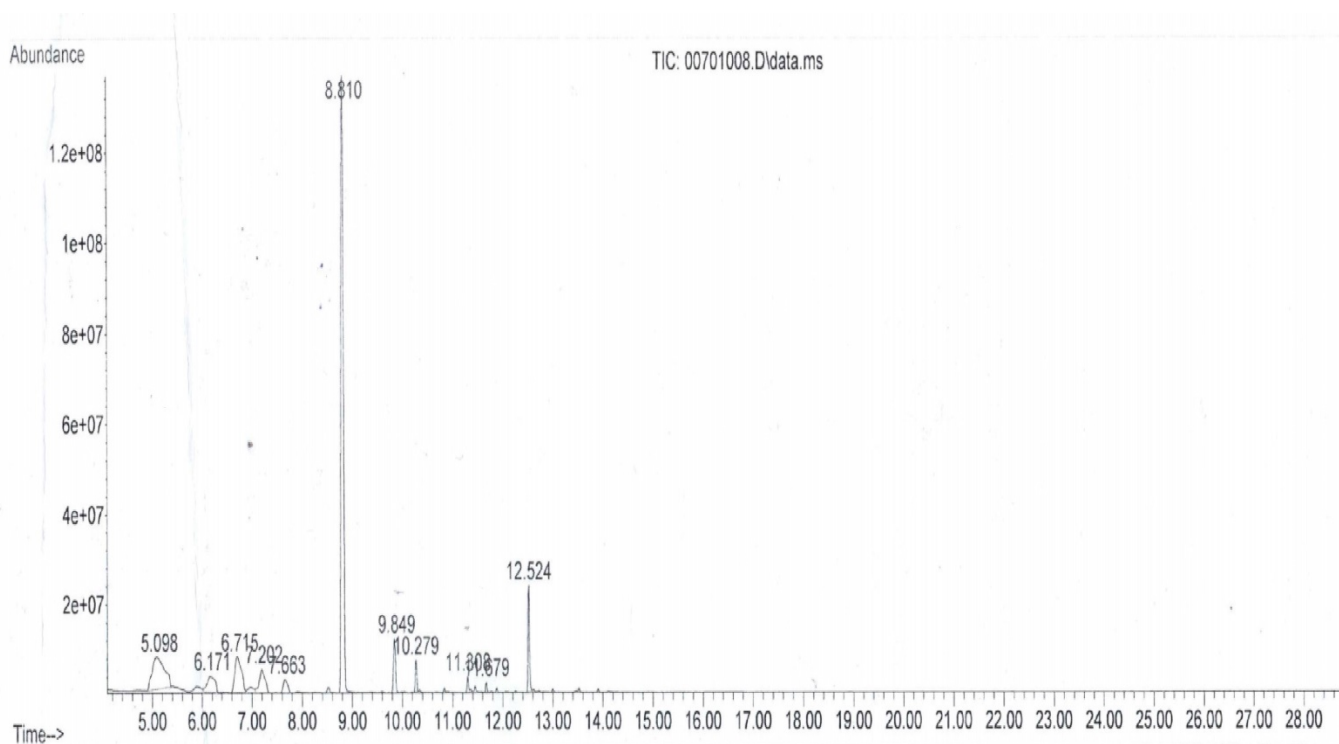


Figure 4-9 GC-MS result profile

Table 4-9 chemical composition of coriander oil

Chemical compound	Library match Quality (%)	Retention time(min)	Area (%)
2,6,6-Trimethylbicyclo[3.1.1]hept-2-ene	96	5.0982	13.4253
Beta-Myrcene	96	6.1707	4.5846
D-Limonene	98	6.7151	8.3925
Gamma-Terpinene	97	7.202	4.8523
Cyclohexane,1-methyl-4-(1-methylethy Lidene)	97	7.6627	2.1209
Linalyl acetate	64	8.8102	54.8565
Camphor	98	9.8487	3.7224
Decanal	99	10.2787	1.6571
3-carene	95	11.3081	0.91
Undecanal	95	11.6791	0.4495
(+)-3-carene	95	12.5242	5.0289

Optimization and Characterization of Essential Oil from Coriander Seed

The identification of the components of the essential oil extracted by steam distillation process was carried out by comparison of their mass spectra and a retention time to those of reference standards. This oil contains a lot of monoterpene hydrocarbons, among other things. There are eleven different compounds was discovered in coriander essential oil. Monoterpene hydrocarbons were found to be the major group of compounds these are alpha-pinene (13.4253%) and linalyl acetate (54.8565%). Other predominate component were, camphor (3.7224%), gamma-terpinene (4.8523%), Limonene (8.92%), decanal (1.6571%), beta-Myrcene (4.5846%), -3-carene (5.0289%).

5. Conclusion and Recommendations

5.1 Conclusion

This research work was intended to study the influence of extraction time and particle size factors on the quality and quantity of essential oil from coriander seed. Variability of these operating conditions is the pre-dominant factors for the quality and quantity of the oil. The extraction of essential oils from coriander seed can be done in a variety of ways. The extraction method used in this study was steam distillation. With Steam distillation extraction method maximum oil yield obtained was 0.84% at the particle size 1-1.4mm and extraction time of 4hr the observed quantitative difference in the quantity of the oil was due to particle size and extraction time variability. As a result, determining the acceptable particle size and the optimal time for the prescribed particle size must be taken into account in order to obtain the maximum amount of the desired product.

The percentage of coriander oil composition and even the chemical composition could be affected by many factors such as climates, geographical regions, sowing time, operating condition, maturity stage, type of soil and extraction equipment. So many physical and chemical properties like specific gravity, refractive index, pH value, solubility, iodine value, acid value and saponification value of the coriander essential oil was determined and obtained. GC-MS analysis was used to identify the major components of coriander essential oil. Monoterpene hydrocarbons were discovered to be the most abundant. The major components were alpha-pinene (13.4253%), Linalyl acetate (54.8565%), gamma-terpinene (4.8523%), Camphor (3.7224%), Limonene (8.3925%), Decanal (1.6571%), and beta-myrcene (4.5846%). Determine the functional group of coriander oil by using FT-IR. From design expert software the variance of analysis (ANOVA) P-value < 0.001 for particle size and extraction time indicate that operating parameter have significant effect on oil yield.

5.2 Recommendations

Recommendation for further work:-

- Comparison of different extraction methods such as solvent extraction, supercritical fluid extraction and single-screw extraction with solvent extraction.
- In this research, the effects of temperature and pressure were not investigated. This is due to uncontrollability of the steam distillation equipment used for essential oil extraction; thus, further research should be performed in order to achieve a much higher quantity and quality of coriander essential oil.
- Further detail researches on feasibility studies are required before implementing this research result.

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APPENDICES

Appendix

A1. Library search report of GC-MS for essential oil of coriander seed

Library Search Report

Data Path : D:\MassHunter\GCMS\1\5977\Nov 19, 2020\
 Data File : 00701008.D
 Acq On : 19 Nov 2020 16:05
 Operator : Estif
 Sample : Firehiwot
 Misc :
 ALS Vial : 7 Sample Multiplier: 1

Search Libraries: D:\MassHunter\Library\NIST14.L Minimum Quality: 0

Unknown Spectrum: Apex
 Integration Events: ChemStation Integrator - autoint1.e

Pk#	RT	Area%	Library/ID	Ref#	CAS#	Qual
1	5.098	13.43	D:\MassHunter\Library\NIST14.L (1S)-2,6,6-Trimethylbicyclo[3.1.1] hept-2-ene	16223	007785-26-4	96
			(1R)-2,6,6-Trimethylbicyclo[3.1.1] hept-2-ene	16224	007785-70-8	96
			(1R)-2,6,6-Trimethylbicyclo[3.1.1] hept-2-ene	16226	007785-70-8	95
2	6.171	4.58	D:\MassHunter\Library\NIST14.L .beta.-Myrcene	16066	000123-35-3	96
			Bicyclo[3.1.0]hex-2-ene, 4-methyl- 1-(1-methylethyl)-	16276	028634-89-1	87
			.beta.-Phellandrene	16088	000555-10-2	87
3	6.715	8.39	D:\MassHunter\Library\NIST14.L D-Limonene	16046	005989-27-5	98
			D-Limonene	16044	005989-27-5	95
			D-Limonene	16045	005989-27-5	93
4	7.202	4.85	D:\MassHunter\Library\NIST14.L .gamma.-Terpinene	16074	000099-85-4	97
			(1R)-2,6,6-Trimethylbicyclo[3.1.1] hept-2-ene	16224	007785-70-8	96
			.gamma.-Terpinene	16077	000099-85-4	96
5	7.663	2.12	D:\MassHunter\Library\NIST14.L Cyclohexene, 1-methyl-4-(1-methyle thylidene)-	16238	000586-62-9	97
			Cyclohexene, 1-methyl-4-(1-methyle thylidene)-	16230	000586-62-9	96
			(+)-4-Carene	16052	029050-33-7	96
6	8.810	54.86	D:\MassHunter\Library\NIST14.L Linalyl acetate	61526	000115-95-7	64
			Pentanoic acid, 1-ethenyl-1,5-dime thyl-4-hexenyl ester	100389	010471-96-2	58
			Bicyclo[3.1.0]hexane, 6-isopropyl dene-1-methyl-	16260	024524-57-0	56
7	9.849	3.72	D:\MassHunter\Library\NIST14.L Camphor	25791	000076-22-2	98
			(+)-2-Bornanone	25850	000464-49-3	98
			(+)-2-Bornanone	25851	000464-49-3	98
8	10.279	1.66	D:\MassHunter\Library\NIST14.L Decanal	29133	000112-31-2	99
			Decanal	29128	000112-31-2	97
			Decanal	29131	000112-31-2	91
9	11.308	0.91	D:\MassHunter\Library\NIST14.L 3-Carene	16028	013466-78-9	95

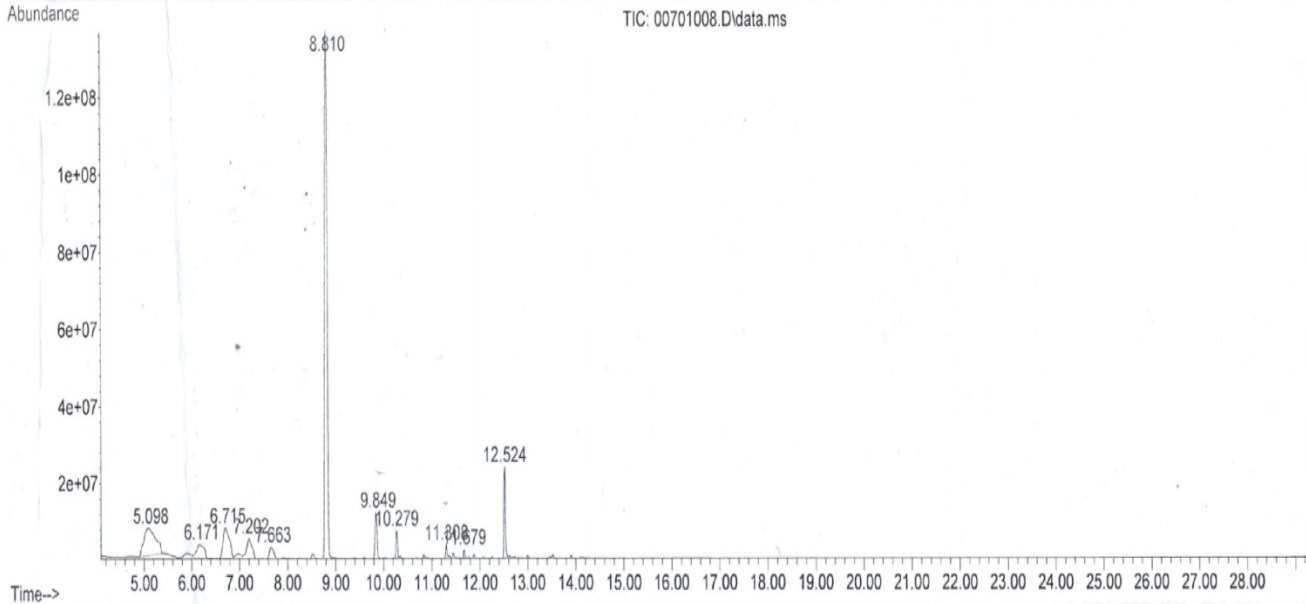
Optimization and Characterization of Essential Oil from Coriander Seed

			3-Carene	16035 013466-78-9 87
			.beta.-Myrcene	16065 000123-35-3 72
10	11.679	0.45	D:\MassHunter\Library\NIST14.L	
			Undecanal	39873 000112-44-7 95
			Undecanal	39871 000112-44-7 91
			1,1-Dodecanediol, diacetate	145886 056438-07-4 87
11	12.524	5.03	D:\MassHunter\Library\NIST14.L	
			(+)-3-Carene	16050 000498-15-7 95
			3-Carene	16036 013466-78-9 92
			3-Carene	16035 013466-78-9 91

Essential o... splitless.M Tue Dec 08 09:38:07 2020

Optimization and Characterization of Essential Oil from Coriander Seed

File :D:\MassHunter\GCMS\1\5977\Nov 19, 2020\00701008.D
Operator : Estif
Acquired : 19 Nov 2020 16:05 using AcqMethod Essential oil 29.33 splitless.M
Instrument : AAU
Sample Name: Firehiwot
Misc Info :
Vial Number: 7



Appendix B. Laboratory Photos

Raw coriander seed



Sieving



Sieved Coriander seed



Steam Distillation



Optimization and Characterization of Essential Oil from Coriander Seed

Refractive Index



Viscosity Measurement



Extracted Essential Oil

