

ADDIS ABABA UNIVERSITY
SCHOOL OF GRADUATE STUDIES
ADDIS ABABA INSTITUTE OF TECHNOLOGY
DEPARTMENT OF CHEMICAL ENGINEERING



**OPTIMIZATION AND CHARACTERIZATION OF PERFUME PRODUCTION USING
LEMONGRASS ADDITIVE**

A Thesis Submitted to the School of Graduate Studies of Addis Ababa University in Partial
Fulfillment of the Requirements for the Degree of Masters of Science in Chemical Engineering
(Process Engineering)

By:

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ADDIS ABABA, ETHIOPI

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List of acronyms

AOAC	Associates of Analytical Chemistry
GC	Gas chromatography
MS	Mass-spectrometer
GC-MS	Gas chromatography- Mass-spectrometer
SCFE	Supercritical fluid extraction
FT-IR	Fourier Transform Infrared Resonance
Esdp	Esprit de Parfum
EDP.	Eau de Parfum
EDT	Eau de toilette
EDC	Eau de Cologne
CIF	Cost insurance and freight
ANOVA	Analysis of variance
ASTM	American Society of Testing and Material
MSD	Mass selective detector

ABSTRACT

Lemongrass is perennial herb which has been cultivating in Ethiopia and known as tej-sar which has been using to decorate the house rather than for perfume application. The objective of this study was to develop a perfume at optimum extraction point using steam distillation from tej-sar (*Cymbopogon citratus*). The effect of different parameters, such as extraction time and particle size was determined on the extraction yield of essential oil. The percentage oil yield in dried samples was higher as compared to fresh samples due to solvate formation. These essential oil was further analyzed by gas chromatography/mass spectrometric (GC-MS), Fourier transform infrared spectroscopy (FT-IR) to evaluate the main component which were citral as indicated by the strong intense vibration frequency in the FT-IR and GC-MS spectrograph. A general factorial design was applied to both extraction processes using DESIGN EXPERT software and linear regression model was obtained growing the individual effect of time, particle size as parameter and their interaction in the entire extraction process. For steam distillation extraction, the minimum essential oil yield has been obtained 0.125% for fresh raw material and 0.225% for dry raw material of lemongrass after the extraction time of 1.5 hours with particle size ranges from 25mm-35mm and maximum oil yield 0.81%, 0.345% was found at the extraction time of 2.5hr in a particle size ranges from 8-15mm for dry and fresh raw material respectively. From the experiment what was obtained was applicable for perfume due to it consisted of citral number that has lemony aroma. The extracted oil from tej-sar was developed into perfume using a fixative and carrier solvent and lastly sensory evaluation of perfume was conducted in the nine-point hedonic test scale to see its quality, aroma and the result was accepted. The rough profitability evaluation indicates us this project is feasible as it is clearly observed from the cost estimation, the rate of return on investment was calculated and has 54.35 % and the payback period tells us the plant return its total investment cost in one year and five month.

1. Introduction

1.1. Background

Perfume is a mixture of fragrant essential oils and aroma compounds, fixatives and solvents used to give the human body, objects and living spaces a pleasant smell.

Since the beginning of recorded history, humans have attempted to mask or enhance their own odor by using perfume which emulates nature's pleasant smells. Many natural and man-made materials have been used to make perfume to apply to the skin and clothing to put in cleaners and cosmetics or to scent the air. This is because of differences in body chemistry, temperature and body odors (Auracacia, 2016).

Perfumes and scented articles were in use from pre Vedic and Vedic periods for religious practices, social customs and domestic rituals and later gradually became part and parcel of human life. Perfumes were also used in cosmetics and beauty aids. Medicinal values of many perfume was well known to ancient and were used in both rituals and to treat diseases. Perfumes as luxury accessory or one of the most popular must have items on the aspirational buyers' list as well (Auracacia, 2016).

Perfume is extremely difficult to produce the large required a quantity of different ingredients to produce a very small essence. The difficulty of production resulted in scarcity and hence made the substance even more valuable and expensive. In Greek society, perfume or odor was one of the main symbols of beauty (Donato and Seefried, 1980).

Perfume is presently the most important in Ethiopia that used to provide a pleasant odor, cover the scent of the base of the product, give a product an identity, provide product concept support and signify a change in a product. Ethiopia is one of the countries which have been using perfume traditionally for many years. According to Ethiopian revenue and custom authority 2015, Ethiopia import perfume an average of 112,589.908kg annually from different countries to satisfied the demand of the buyers (ERCA, 2015). Due to this reason Ethiopia loss its foreign currency as much.

Lemongrass is perennial herb largely cultivated in tropical and subtropical countries. The scientific name of lemongrass is *Cymbopogon citratus*. The *Cymbopogon* word derives from the Greek words “kymbe” (boat) and “pogon” (beard) referring to the arrangement of the spike of the flower. The word *citratus* derives from the old Latin meaning lemon-scented leaves (Shah *et*

al., 2011). It is a perennial tropical grass; resistant to different temperatures and can grow in warm, semi-warm and temperate climates. It is from 60 to 120 centimeters high, its leaves are green, long and slats and have pleasant aroma and taste. This grass is native to India (Parikh & Desai, 2011).

The essential oil of the *C. citratus* is one of the most important volatiles oils which is economically important component of lemon grass (Lewinsohn *et al.*, 1998). The oil used in Brazil for medicinal purposes, perfume, food and pharmaceuticals (Koshima *et al.*, 2006). These days the demand for the lemongrass perfume has soared because of its unique fragrance. While it is widely used in the different types of herbal teas and non-alcoholic beverages.

Lemongrass also known in Ethiopia as (tej-sar). There are two common lemongrass species that were registered in the book of Ethiopia and Eritrea flora (Tewolde and Sue Edwards, 1997). Those are *Cymbopogon citratus* and *nardus*. *Cymbopogon citratus* grass has been growing for many years in Ethiopia as backyard plant. This herb plant used by the farmer for decoration of the house during holiday instead of using for perfume and other application. From this Ethiopia has capacity to cultivate this grass due to available land, high cultivation period and good weather condition and that used as raw material for perfume development.

1.2 Statement of Problems

The problem of formulation of perfume involves knowing the proportion in which essential oil and other materials to be mixed to avoid skin irritation and increase the intensity and longevity of the perfume. Most imported perfumes are synthetic odorant which are not pure natural fragrance and it is a mixture of organic compounds that are harmful when it applied.

Today there is a high demand of perfume for various purposes such as medicinal, soap making household, personal hygiene and insecticides even if it is significant, no perfume factories that are erected in Ethiopia rather than imported. Imported perfume are also very expensive to meet the demand of our local consumer industries besides direct human use. In particular perfumes that are usually imported can be produced locally from a vast variety of oil bearing plants yet to be explored. Therefore it becomes necessary to source and extract this oil from local source such as lemongrass that used to develop perfume by extracting the essential oil with steam distillation.

1.3 Objectives

1.3.1 General Objectives

The general objective of this study to develop an economical and indigenous way of producing body perfume using locally available raw material of lemongrass that capture the perfect scent for the average of working women between the age of 23-55 with extraction of essential oil using steam distillation method.

1.3.2 Specific Objectives

The specific objectives of this research work include:

- Characterization and Optimization of essential oil extraction from lemongrass using steam distillation.
- To develop perfume and characterize physical property using sensor analysis.
- To determine techno-economy feasibility of perfume production using lemongrass Additive.

1.4 Significance of the study

- To secure our perfume supply as well as to reduce its perfume import bill through the development of an indigenous perfume production.
- To reduce any side effect resulting from synthetic chemicals by substituting natural one.
- To create awareness for stockholders as it is possible to establish local industries for perfume and oil production because of available land and cheap raw materials.

2. Literature Review

2.1 The History of Perfumery

Perfumery is the art of making perfumes began in ancient Mesopotamia and Egypt and was further refined by the Romans and Persians. Knowledge of perfumery came to Europe as early as the 14th century due partially to the spread of Islam. The first modern perfume made of scented oils blended in an alcohol solution was made in 1370 at the command of queen Elizabeth of Hungary and was known throughout Europe as Hungary Water. The art of perfumery prospered in Renaissance Italy and in the 16th century. Italian refinements were taken to France by Catherine de' Medici's personal perfumer (Elizabeth, 2011).

The most detailed perfume recipes for composite scented preparations were written down ancient Egypt only during the Ptolemaic Period. During this period the doors to the classical World were opened wide to the ancient Egyptians and besides the Egyptian language Latin and Greek were understood. These were inscribed in hieroglyphs on the walls of perfume incense 'laboratories' of temples. Other sources such as actual remains of plants found in excavations and wall paintings and reliefs in temples and tombs provide us with knowledge of the plants used by the Egyptians for perfume production and the methods they used. The bulk of our knowledge about perfume recipes from ancient world and the plants used in them, however, it is derived from various classical authors both Greek and Roman ("The essence and use of perfume in ancient Egypt by Sheila Ann by submitted in accordance with the requirements for the degree of master of arts in the subject ancient near eastern studies at the University of South Africa Supervisor : Professor P S Vermaak joint supervisor : Mrs a Ferreira February 2012," 2012).

Most modern perfumes are alcohol-based and contain synthetic scents. While the term perfume usually refers to fragrances in general, in the more technical language of the perfumer, a perfume must contain over 15% of fragrance oils in alcohol. The preferred fragrances for perfumes are no means universal, but differ according to cultural dictates and fashions. In the sixteenth century, for example pungent animal scents such as musk and civet were very popular. In the nineteenth century by contrast such animal scents were generally considered too crude and light floral fragrances were favored (Calkin and Jellinek, 1994).

The ancient Egyptians highly prized their botanical treasures in which Egypt was richly endowed and for the growing of which its climate was admirably suited, being moderate and uniform. The

Egyptians made various types of perfumes from fragrant plants oils and fats employing various methods and involving various professions (Brun, 2000; Dayagí-Mendels, 1989).

There are two main categories of perfumery products which toiletries and household products. Toiletries are defined as fine fragrances, personal care products, cosmetics and deodorants. Household products are considered to be air fresheners, laundry products, surface cleaners and disinfectants.

2.2 Concentration of Perfume

Perfume types reflect the concentration of aromatic compounds in a solvent (which in fine fragrance) is typically ethanol or a mix of water and ethanol. Various sources differ considerably in the definitions of perfume types. The intensity and longevity of a perfume is based on the concentration, intensity and longevity of the aromatic compounds (natural essential oils or perfume oils) used: As the percentage of aromatic compounds increases, so does the intensity and longevity of the scent created. Specific terms are used to describe a fragrance's approximate concentration by percent/volume on perfume oil which is typically vague or imprecise. A list of common terms (Perfume-Classification) is as follows (Burr and Chandler, 2008).

- Perfume extract or simply perfume (Extract): 15-40% aromatic compounds.
- Esprit de Parfum (ESdP): 15-30% aromatic compounds a seldom used strength concentration in between EdP and perfume.
- Eau de Parfum (EdP), Parfum de Toilette (PdT): 10-20% (typical 15%) aromatic compounds.
- Eau de toilette (EdT): 5-15% (typical 10%) aromatic compounds.
- Eau de Cologne (EdC): Chypre citrus type perfumes with 3-8% (typical 5%) aromatic compounds.
- Perfume mist: 3-8% aromatic compounds (typical non-alcohol solvent).
- Splash (EdS) and Aftershave: 1-3% aromatic compounds.

2.3 Perfume Notes

Perfume is described in a musical metaphor as having three sets of notes making the harmonious scent accord. The notes unfold over time with the immediate impression of the top note leading to the keener middle notes and the base notes gradually appearing as the final stage. These notes are created carefully with knowledge of the evaporation process of the perfume (Burr and Chandler, 2008).

Top notes: The scents that are perceived immediately on application of a perfume. Top notes consist of small light molecules that evaporate quickly. They form a person's initial impression of a perfume and thus are very important in the selling of a perfume.

Middle notes: The scent of a perfume that emerges just prior to when the top notes dissipate. The middle note compounds form the "heart" or main body of a perfume and act to mask the often unpleasant initial impression of base notes which become more pleasant with time. They are also called the heart notes.

Base notes: The scent of a perfume that appears close to the departure of the middle notes. The base and middle notes together are the main theme of a perfume. Base notes bring depth and solidity to a perfume. Compounds of this class of scents are typically rich and "deep" and are usually not perceived until 30 minutes after application. The scents in the top and middle notes are influenced by the base notes as well the scents of the base notes will be altered by the type of fragrance materials used as middle notes.

2.4 Component of Perfume

There are three basic parts of perfume that make up components for the formulation:

Basics: The base is the most important component of any perfume. A base can be any fragrance or scent that is not as volatile. This scent can be added to the perfume which will result in a product that meets consumer desires.

Fixatives: Fixatives are ingredients in the perfume that prolong the odor effect and delay the evaporation rate of volatile materials. The component tends to have no odor to be miscible in polar and non-polar solutions and to be at a higher boiling point temperature. The fixative bonds to polar compounds within the perfume through hydrogen bonding reducing the overall vapor pressure of the mixture. They retain a high concentration of the top and middle notes and release them slowly over time. Examples of fixatives are vetiver, dipropylene glycol, diethyl phthalate and glycerin.

Solvents: The solvent is the portion of the perfume in which all the components are dissolved. Solvents are used to dilute the mixture in order to increase the surface area of application without using an excessive amount of the fragrance. It is also used to reduce the intensity of the fragrance of the solution. Solvents decrease the price of the perfume per milliliter while increasing the amount of applications per bottle. Ethanol is the most common solvent that used for perfume formulation.

2.5 Perfume import in Ethiopia

Perfume is very significant in Ethiopia for different purpose in addition to body perfume.

Table 2.1 perfume imports from different countries, 2011-2015

year	Net Wt. (Kg)/yr	Average /yr in (kg)	CIF Value (ETB)/yr	Average in annual(Birr)
2015	98,931	112,589.908	123,723,953.19	129,616,394
2014	164,898.94		200,754,677.02	
2013	126,095.36		126,607,519.23	
2012	86,207.57		95,911,419.74	
2011	86,816.67		101,084,402.80	

(Source: Ethiopian revenue and custom authority, Addis Ababa Ethiopia 2016)

2.6 Classification of Perfumery Materials

The raw materials employed in perfumery have natural origin (animal or plant) or synthetic origin (natural-identical or new artificial fragrant molecules called aroma chemicals).

- ♠ Aroma chemicals synthesized from crude oil making up to 70 to 100% of the perfume concentrate.
- ♠ Natural essential oils and plant extracts-constituting up to 30% of perfume composition.
- ♠ Animal products used in very small amounts up to 0.1% in the formulation.

✚ Aromatics Source

- **Plant Sources:**

Plants have long been used in perfumery as a source of essential oils and aroma compounds. These aromatics are usually secondary metabolites produced by plants as protection against herbivores, infections as well as to attract pollinators. Plants are by far the largest source of fragrant compounds used in perfumery. The sources of these compounds may be derived from various parts of a plant. A plant can offer more than one source of aromatics for instance the aerial portions and seeds of coriander have remarkably different odors from each other.

- **Animal Sources:**

Ambergris: Lumps of oxidized fatty compounds whose precursors were secreted and excreted by the sperm whale. Ambergris should not be confused with yellow amber which is used in jewelry.

Because the harvesting of ambergris involves no harm to its animal source, it remains one of the few animalic fragrance agents around which little controversy now exists.

Castoreum: Obtained from the odorous sacs of the North American beaver.

Civet: Also called civet musk, this is obtained from the odorous sacs of the civets, the world society for the protection of animals investigated African civets caught for this purpose.

Hyraceum: Commonly known as "Africa Stone" is the petrified excrement of the Rock Hyrax.

Honeycomb: From the honeycomb of the honeybee both beeswax and honey can be solvent extracted to produce an absolute.

Deer musk: Originally derived from the musk sacs from the Asian musk deer, it has now been replaced by the use of synthetic musk sometimes known as "white musk".

Synthetic Source

Many modern perfumes contain synthesized odorants. Synthetics can provide fragrances which are not found in nature. For instance, Calone, a compound of synthetic origin imparts a fresh ozonous metallic marine scent that is widely used in contemporary perfumes. Synthetic aromatics are often used as an alternate source of compounds that are not easily obtained from natural sources.

2.7 Essential Oil

Essential oils or volatile oils are found in many different plants. These oils are different from fatty oils because they evaporate or volatilize on contact with the air and they possess a pleasant taste and strong aromatic odor. They are readily removed from plant tissues without any change in composition. Essential oils are very complex in their chemical nature. The two main groups are the hydrocarbon terpenes and the oxygenated and sulphured oils (Gunther, 1994).

2.7.1 Essential Oil plant for perfumery in the world

◆ Lavender

Lavender perfumes are very old and were used by the Romans in their baths. It is still one of the most important scents. It is a low shrub with terminal spikes of very fragrant bluish flowers. The oil is important in the manufacture of Eau de Cologne and other perfumes and is also used in soaps, cosmetics and medicine as a mild stimulant. Lavender water a mixture of the oil in water and alcohol is popular in England (Yardley brand).

◆ Jasmine

A highly esteemed perfume, jasmine is cultivated in southern France and surrounding areas. The flowers are picked as soon as they are open and the oil is extracted by enfleurage (Seminar, 2014).

Table 2.2 Important essential oils (Gunther, 1994).

Name of oil	Method of production	Part of plant used
Almond	Steam distillation	Kernels
Bay	Steam distillation	Leaves
Bergamot	Expression	Peel
Caraway	Steam distillation	Seed
Cassia	Steam distillation	Leaves and twigs
Cedar wood	Steam distillation	Red core wood
Cinnamon	Steam distillation	Bark
Citronella	Steam distillation	Grass
Clove	Steam distillation	Buds
Coriander	Steam distillation	fruits
lemongrass	Steam distillation	leave
Eucalyptus	Steam distillation	Leaves
Geranium	Steam distillation	Leaves
Jasmine	Cold pomade	Flowers
Jasmine	Hydro-distillation	Flowers
Lemon	Expression	Peel
Orange	Expression, distillation	Peel
Peppermint	Steam distillation	Leaves
Rose	Steam distillation, solvent	Flowers
Sandalwood	Steam distillation	Wood
Spearmint	Steam distillation	Leaves
Tuberose	solvent, enfleurage	Flowers
Wintergreen	Steam distillation	Leaves
Ylang-ylang	Steam distillation, solvent	flowers

2.7.2 Essential Oil plant in Ethiopia for perfumery application

Ethiopia has a long history of spice and herb production for the domestic market and has a unique, indigenous product. Modest quantities of several spices have been exported for centuries to countries in the Middle East and exports to Europe have developed over the past twenty years (Wikipedia). In general use, herbs are any plants used for flavoring, food, medicine and perfume. In Ethiopia several culinary herbs are produced: chervil, chives, coriander, dill, green basil, lovage, mint, oregano, rocolla, thyme, vernonia are Aromatic plant (Council & Willems, n.d).

◆ Lavender

Is a genus of 39 species of flowering plants in the mint family Lamiaceae. The genus includes annual or short-lived herbaceous perennial plants and suffrutescent perennials, sub shrubs or small shrubs. Leaf shape is diverse across the genus. They are simple in some commonly cultivated species.

◆ Frankincense

Frankincense and myrrh are phytotoxically safe raw materials in industries like pharmaceuticals and food industries. They are used in folk medicines, flavoring, beverages and liqueurs, cosmetics, detergents, creams and perfumery, paints, adhesives and dyes manufacturing. Both myrrh and frankincense are highly valued for their aromatic fragrances and are common ingredients in incense perfume and potpourris, soaps, detergents, creams and lotions and are often included in meditation blends, as it strengthens the psyche and aids in deepening the meditative state (FAO, 1995). Three types of frankincense products are recognized in Ethiopia: Tigray, Ogaden and Borena Incense are aromatic biotic material which releases fragrant smoke when burned.

◆ Myrrh

Myrrh is the aromatic resin of a number of small, thorny tree species of the genus *Commiphora* which is an essential oil, termed an oleoresin. Myrrh resin is a natural gum. It has been used throughout history as a perfume incense and medicine. It can also be ingested by mixing it with wine. In pharmacy, myrrh is used as an antiseptic in mouth washes, gargles and toothpastes.

◆ Gum Arabic

Gum Arabic is used as thickening, stabilizing, emulsifying and suspending agent in food and drink industries; as tablet-binding agent, cream, lotions- suspending and emulsifying agents in pharmaceuticals, as film forming and sizing agent in printing and textile industries. It is also used

in ceramics, paints, inks, textiles and adhesives. In cosmetics, gum Arabic functions as a stabilizer in lotions and protective creams where it increases viscosity, imparts spreading properties and provides a protective coating and a smooth feel. It is used as an adhesive agent in blusher and as a foam stabilizer in liquid soaps.

◆ Opoponax

Opoponax or sweet myrrh is a cousin of the healing Myrrh *Commiphora*. Myrrh with a warm balsamic and sweet, honey-like aroma. It is a natural oleo-gum-resin like myrrh and frankincense. The color of its resin is brown; however, good quality crude botanical resin is dark red. Opoponax has been a component of incense and perfumes since Biblical times. Talking of perfumery in particular, Opoponax qualities from several *Commiphora* are widely used especially in oriental fragrances to impart sweet balsamic notes.

◆ Lemongrass (*Cymbopogon Citratus*)

A perennial plant with long, thin leaves, is one of the largely cultivated medicinal plants for its essential oils in parts of tropical and subtropical areas of Asia, Africa and America (Ranitha, Nour, Sulaiman, Nour, & S, 2014). *Cymbopogon* (lemon grass) is a genus of about 55 species of grasses (of which the type species is *Cymbopogon citratus*) native to warm temperate and tropical regions of the Old World and Oceania. It has a citrus flavor and can be dried and powdered or used fresh. Lemongrass oil is used as a pesticide and a preservative. Chemical investigations of essential oils in the nineteenth century revealed that many of the compounds responsible for the pleasant odors contained exactly ten carbon atoms. These ten carbon compounds came to be known as terpenes if they were hydrocarbons and terpenoids if they contained oxygen and were alcohols, ketones or aldehydes. Eventually, it was found that there are also minor and less volatile plant constituents with fifteen, twenty, thirty and forty carbon atoms. Because compounds of ten carbons were originally called terpenes, they came to be called terpenoids if they contained oxygen and were alcohols, ketones and aldehydes (Pavia *et al*, 2005). There are two types of lemongrass (*Teji sar*) which are found in Ethiopia *cymbopogon citratus* and *nardus*.

2.8 Chemical Constituents of Essential Oils.

Essential oils are a mixture of fragrant compounds including terpenoids, aldehydes (geranial, citronellal), alcohols (geraniol, citronellol, nerol), esters (linalyl acetate, citronellyl acetate, isobornyl acetate (Burt, 2004), which accumulate in different organs like leaves, barks, woods,

roots, rhizomes, fruits and seeds. Essential oils are synthesized and accumulated in specialized organs (trichomes, secretory cavities or canals) located near surfaces.

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

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

✚ Hydrocarbon

Essential Oils consist of Chemical Compounds that have hydrogen and carbon as their building blocks. Basic Hydrocarbon found in plant is isoprene.

✚ Terpenes

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

◆ Monoterpenes [C₁₀H₁₆]

Properties: Analgesic, Bactericidal, Expectorant and Stimulant. Monoterpenes are naturally occurring compounds, the majority being unsaturated hydrocarbons (C₁₀). But some of their oxygenated derivatives such as alcohols, Ketones and carboxylic acids known as monoterpenoids. The branched-chain C₁₀ hydrocarbons comprises of two isoprene units and is widely distributed in nature with more than 400 naturally occurring monoterpenes identified.

◆ Sesquiterpenes

Properties: anti-inflammatory, anti-septic, analgesic, anti-allergic. Sesquiterpenes is biogenetically derived from farnesyl pyrophosphate and in structure may be linear, monocyclic or bicyclic. They constitute a very large group of secondary metabolites, some having been shown to be stress compounds formed as a result of disease or injury.

◆ Sesquiterpene Lactones

Over 500 compounds of this group are known; they are particularly characteristics of the Composite, but do occur sporadically in other families. Not only have they proved to be of interest from chemical and chemotaxonomic viewpoints, but also possess many antitumor, anti-

leukemia, cytotoxic and antimicrobial activities. They can be responsible for skin allergies in humans and they can also act as insect feeding deterrents. Chemically the compounds can be classified according to their carboxylic skeletons. Thus, from the germacranolides can be derived the guaianolides, pseudoguaianolides, eudesmanolides, eremophilanolides, xanthanolides, etc.

◆ Diterpenes

Properties: anti-fungal, expectorant, hormonal balancers, hypotensive .Diterpenes are made of up four isoprene units. This molecule is too heavy to allow for evaporation with steam in the distillation process, so is rarely found in distilled essential oils. Diterpenes occur in all plant families and consist of compounds having a C₂₀ skeleton. There are about 2500 known diterpenes that belong to 20 major structural types. Plant hormones Gibberellins and phytol occurring as a side chain on chlorophyll are diterpenic derivatives. The biosynthesis occurs in plastids and interestingly mixtures of monoterpenes and diterpenes are the major constituents of plant resins. In a similar manner to monoterpenes, diterpenes arise from metabolism of geranyl geranyl pyrophosphate (GGPP).

✚ Alcohols

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

✚ Aldehydes

Properties: anti-fungal, anti-inflammatory, anti-septic, anti-viral, bactericidal, disinfectant, sedative. Medicinally, essential oils containing aldehydes are effective in treating Candida and other fungal infections disease.Example Citral in lemon, Lemongrass and lemon balm which has been used.

✚ Acids

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

✚ Esters:

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

✚ Ketones

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

✚ Lactones:

Properties: anti-inflammatory, antiphlogistic, expectorant, febrifuge. Lactones are known to be particularly effective for their anti-inflammatory action, possibly by their role in the reduction of prostaglandin synthesis and expectorant actions. Lactones have an even stronger expectorant action than ketones (A, 2012).

2.9 Uniqueness of essential oils

In early work, the term essential oil was defined as the volatile oils obtained by the steam distillation of plants. This definition was clearly intended to make a distinction between "fatty oils" and the oils which are easily volatile.

It was found that the oils contain many classes of organic substances with varying volatility. Although a list of all the known oil components would include a variety of chemically unrelated compounds. It is possible to classify these into main groups of essential oils (Guenther, 1960).

- ♣ Terpenes, related to isoprene
- ♣ Straight-chain compounds, not containing any side branches
- ♣ Benzene derivatives

◆ Essential oils are volatile:

Essential oils are the volatile fragrant components from various indigenous and exotic plants which have been traded internationally for several centuries (Becker, 2005).

◆ Essential oils are aromatic:

Essential oils are highly aromatic and therefore, many of the benefits can be obtained by simply inhaling them. This can be done by breathing in the fragrance from the bottle or they can be diffused into the room. Essential oils when diffused can be the best air filtration system in the world. They will purify the air by removing metallic particles and toxins from the air increase atmospheric oxygen; increase ozone and negative ions in the house which inhibits bacterial growth, destroy mold, cigarettes and animal odours; fill the air with a fresh herbal aromatic scent (Becker, 2005).

◆ Essential oils have penetrating characteristics:

The penetrating characteristic of essential oils greatly enhances their ability to be effective. Essential oils will penetrate into the body when applied to the skin. Essential oils rubbed into the feet will be distributed to every cell in the body in minutes. They will even penetrate a finger or toe nail to treat fungal infection underneath. Other vegetable oils do not have this propensity to penetrate (Becker, 2005).

◆ Pure essential oils have very high frequency:

The effectiveness of essential oils is sometimes also described in terms of frequency. It has been reported that the human body has an electrical frequency and that much about a person's health can be determined by frequency. Tainio has determined that the average frequency of the human body during the day time is 62-68 MHz (a healthy body frequency is 62-72). When the frequency drops, the immune system is compromised. If the frequency drops to 58 MHz, cold and flu symptoms appear, at 55 MHz, diseases like Candida take hold, at 52 MHz, Epstein bar and at 42 MHz, cancer. According to Dr. Royal R. Every disease has a frequency He found that certain frequencies can prevent the development of disease and that others would destroy disease. Substances with higher frequency will destroy diseases of a lower frequency. The study of frequencies raises important questions, concerning the frequencies of substances we eat breath and absorb. Many pollutants lower healthy frequency. Processed canned food has a frequency of zero. Fresh produce has up to 27 MHz Essential oil start at 52 MHz and go as high as 320 MHz, which is the frequency of rose oil. Clinical research shows that essential oils have the highest

frequency of any natural substance known to man, creating an environment in which disease; bacteria, virus, fungus, etc. cannot live (Becker, 2005).

2.10. Essential Oil Extraction Processes for Perfume Formulation

There are a few conventional and modern methods of extracting essential oils. It can be extracted by hydro-distillation, cold pressing, enfleurage, hydro-diffusion, supercritical fluid extraction, vapo-cracking, turbo-extractor and microwave extraction.

A. Hydro-distillation

Essential oils can be extracted by hydro-distillation; water, steam and water / steam distillation

i. Steam distillation

Many of the essential oils presently used in perfumery are obtained by steam distillation of flowers, leaves, bark; etc (John, 1982). Steam is widely used because of its high latent heat of evaporation, relatively cheaper and widely available. There are two types of steam distillation: water / steam distillation and steam distillation. This process involves the use of steam to percolate and vaporize out the essential oils from the plant material, with the subsequent condensation of steam and essential oil prior to their separation. It can be seen from the experimental work done that there is an art to distillation and that especially for low yield plants much skill is needed. The role of the distiller is to achieve oil as close as possible to the oil as it exists in the plant. During distillation, only very tiny molecules can evaporate so they are the only ones which leave the plant. These extremely small molecules make up an essential oil. The most advanced type of distillation is by direct steam provided from a separate boiler. The advantages of this type of "dry" steam distillation are that it is relatively rapid, therefore charging and emptying the still is much faster and energy consumption is lower. The rapid distillation is also less likely to damage those oils which contain reactive compounds, e.g. Esters. As a general rule all stills should be insulated ("lagged") to reduce heat losses. Their design and construction should also facilitate loading and unloading (Guenther, 1972).

ii. Water distillation

In this method the charge (which is usually comminuted) is immersed totally in water, which is boiled. The stills are of the simplest type and are used extensively by small holder producers of essential oils (Guenther, 1972). Often they are heated over an open fire. The disadvantages are that the heat is difficult to control and hence the rate of distillation is variable. Also the possibility exists for local overheating and "burning" of the charge which can lead to poorer

quality oil. Improved distillation control can be obtained by using steam from a separate boiler, which is passed into a jacket around the still or through a closed coil in the bottom of the still, to heat the contents of the still. A further disadvantage of this system is that it requires the heating of a large quantity of water adding to costs and time needed for each distillation. However, it is necessary for the efficient distillation of certain woody materials e.g. sandalwood and cinnamon barks (Noor Azian, 2001).

iii. 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. Like water distillation, water and steam distillation is widely used in rural areas. Moreover, it does not require a great deal more capital expenditure than water distillation. Also, the equipment used is generally similar to that used in water distillation, but the plant material is supported above the boiling water on a perforated grid. In fact, it is common that persons performing water distillation eventually progress to water and steam distillation. It follows that once rural distillers have produced a few batches of oil by water distillation, they realize that the quality of oil is not very good because of its still notes (subdued aroma). As a result, some modifications are made. Using the same still, a perforated grid or plate is fashioned so that the plant material is raised above the water. This reduces the capacity of the still but affords a better quality of oil. If the amount of water is not sufficient to allow the completion of distillation, a cohobation tube is attached and condensate water is added back to the still manually, thereby ensuring that the water, which is being used as the steam source, will never run out. It is also believed that this will, to some extent, control the loss of dissolved oxygenated constituents in the condensate water because the re-used condensate water will allow it to become saturated with dissolved constituents, after which more oil will dissolve in it.

B. Cold Pressing

Cold pressed expression or scarification is used to obtain essential oils for the peels and seeds of citrus's, such as bergamot, grapefruit, lemon, lime, mandarin, orange and tangerine oils (Arnould, 1981). In this process, the outer layer of the fruit peel contains the oil are removed by scrubbing. Then the whole fruit is pressed to squeeze the juice from the pulp and to release the essential oil from the pouches. The essential oil rises to the surface of the juice and is separated from the juice by centrifugation.

C. Enffleurage

Enffleurage is an expensive process and is rarely used today except in a few places in France and India (Arnould, 1981). Some flowers, such as jasmine or tuberose have very low Contents of essential oil and are extremely delicate; heating them would destroy the blossoms before releasing the essential oils (Furia, 1972). In such cases, enffleurage is sometimes used to remove the essential oils (Guenther, 1972). Flower petals are placed on solid sheets of warm which will absorb the flowers' essential oils. When all the fragrance is transferred from the flowers to the fat, they are removed and replaced with fresh ones (Billot and Wells, 1975). This process is repeated several times until the fat becomes saturated with the essential oil. A solvent most of the time alcohol is then added which separates the essential oil from the fatty substance. The alcohol will then evaporate leaving only the essential oil. This method is no longer commercially viable (Poucher, 1974).

D. 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). The advantage of hydro-diffusion over distillation is that the process is quicker, especially for fibrous material such as woods and barks. The resultant oils are reported to have a superior aroma and a richer colour obtained by ordinary distillation. Nevertheless, oils captured by hydro-diffusion process are not widely available.

E. Supercritical Fluid Extraction (SFE)

Carbon dioxide is a new method of extraction using carbon dioxide gas, which is kept under high pressure and at a constant temperature (Kelly *et al*, 2002). Plants are placed in a stainless steel tank and as carbon dioxide is injected into the tank, pressure inside the tank builds. Under high pressure, the carbon dioxide turns into a liquid and acts as a solvent to extract the essential oils from the plants. When the pressure is decreased, the carbon dioxide returns to a gaseous state, leaving no residues behind. The equipment for this process is very expensive and so are the resulting oils. Carbon dioxide extractions have fresher, cleaner, and crisper aromas than steam-distilled essential oils and they smell more similar to the living plants because high heat is not used. This extraction method produces higher yield. Many essential oils that cannot be extracted by steam distillation can be obtainable with carbon dioxide extraction. Nevertheless, this technique is very expensive and it is not easily handled.

F. Vapo-cracking

This is a new continuous process, which is French patented (Martel, 1978) allows the recovery of the most volatile aromas directly from solids, like fruits, vegetables and other aromatic raw materials. As a first step in processing, either batch wise or continuously, it simultaneously replaces a press, particularly when that conventional process would yield very little juice. The main plant divides in two parts: Heating is obtained by direct steam injection by steam jacket and hollow screws or by any suitable range of radiations. Condensates may be recovered from that “bleaching” if some preliminary extraction has occurred. A flash chamber is kept under vacuum, where the pre-heated solids fall after passing a sluice. At this point 10 percent of the water content of the material is vaporized. The recovered condensates are remarkably rich in the most volatile aromatic fractions. They are further concentrated by continuous distillation or any other suitable process.

G. Turbo-Extractor

This method is 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 turbo-extractor unit patented by DCF Aroma process (Martel, 1978). It is energy and time saving production of natural extracts. The equipment is equipped with a high blade turbine. It is used for crushing materials within the solvent and fluidizing the comminuted pieces. Extraction is accelerated by the intimate and turbulent contacts between the solid and the liquid phase. Nevertheless, this type of equipment is very expensive and not economical. The Microwave Assisted Process is a revolutionary method of extraction that reduces the extraction time to as little as a few seconds with up to a ten-fold decrease in the use of the solvents. The target material is immersed in solvent that is “transparent” to microwaves, so only the target material is heated. Since the microwave tends to heat the inside of the material quickly, the target chemical is expelled in a few seconds. This process allows for direct extraction of fresh material without the need to dry them prior to the extraction (Soud *et al*, 2002).

H. Solvent extraction

Solvent extraction uses very little heat so it's able to produce essential oils whose fragrance would otherwise be destroyed or altered during steam distillation. Solvent extraction is used on delicate plants to produce higher amounts of essential oils at a lower cost (Chrissie, 1996). In this process, a chemical solvent such as hexane is used to saturate the plant material and pull out the

essential oils. 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 solvent is cooled back into liquid and reclaimed. Along with the essential oil, the fats, waxes and heavier oils can be extracted. This produces a substance called a concrete. The process continues by dissolving oils into warm alcohol. The alcohol is removed under vacuum and pure essential oil is left. Although more cost-efficient than enfleurage, solvent extraction is more expensive than steam distillation so it is reserved for costly oils which cannot be distilled. A solvent extracted essential oil is called an absolute.

I. Micro wave extraction

Microwave radiation interacts with dipoles of polar and polarizable materials. The coupled forces of electric and magnetic components change direction rapidly (2450 MHz). Polar molecules try to orient in the changing field direction and hence get heated. In non-polar solvents without polarizable groups, the heating is poor (dielectric absorption only because of atomic and electronic polarizations). This thermal effect is practically instantaneous at the molecular level but limited to a small area and depth near the surface of the material. The rest of the material is heated by conduction. Thus, large particles or agglomerates of small particles cannot be heated uniformly, which is major drawback of microwave heating. It may be possible to use high power sources to increase the depth of penetration. However, microwave radiation exhibits an exponential decay once inside a microwave-absorbing solid.

In microwave-assisted extraction (MAE): 1) the heat of the microwave irradiation is directly transferred to the solid without absorption by the microwave-transparent solvent; 2) the intense heating of step 1 causes instantaneous heating of the residual microwave-absorbing moisture in the solid; 3) the heated moisture evaporates, creating a high vapor pressure; 4) the vapor pressure generated by the moisture breaks the cell; and 5) breakage of cell walls releases the oil trapped within it (Sukhdev Swami *et al*, 2008).

2.11 Selection of Extraction Processes of Essential Oils

2.11.1 Steam distillation extraction method

Many of the essential oils presently used in the perfumery are obtained by steam distillation of flowers, leaves, bark, etc. According to John J. McKetta (1982) steam distillation refers to a

process where live steam is in direct contact with the distilling system in either batch or continuous operation.

Parameters for selecting an appropriate extraction method of steam distillation based on the following criteria.

- ◆ Hydrolysis of oil constituents is good.
- ◆ Rate of distillation is high.
- ◆ Yield of oil is good if plant material is properly comminuted.
- ◆ Quality of oil is good.
- ◆ Amount of steam can be readily controlled.
- ◆ No thermal decomposition of oil constituents.

2.11.2 Mechanism of steam distillation.

In steam distillation, after the dried and fresh lemon grass has been carefully prepared by comminution only part of the lemon grass oil is present on the surface of the material and immediately vaporized by steam. Heating is the first mechanism in distilling the lemon grass oil as shown in Figure 2.1. Heating in this case is the heating of the plant material at the temperature of boiling water. This is so because higher temperature gives rise to decomposition products which result in a disagreeable “burnt” odour to the oil (Guenther, 1972). At this point, a part of the lemon grass oil dissolves in the water present within the oil glands. This oil in water solution permeates by osmosis through the swollen membranes. Finally reaches the outer surface where the oil is vaporized by passing steam. When this happens, additional quantities of oil go into the solution and permeate the cell membranes while hot water enters. The osmosis and diffusion mechanism overlap each other. In other words, both mechanisms occur simultaneously. These processes continue until all volatile substances are diffused from the oil glands and are vaporized by passing steam.

Heating \implies osmosis \implies diffusion \implies vaporization \implies condensation \implies separation

Figure 2.1: The flow chart for the variation of mechanism of hydro distillation during steam distillation.

2.11.3 Parameters affecting yield and quality of steam distillation extraction

The yield and quality of essential oil from steam distillation is affected by the various process parameters. It is advisable to keep them in mind while designing such systems. Some of the important parameters are being listed below (Padriciano, 2008).

◆ Mode of distillation

The technique for distillation should be chosen considering the boiling point of the essential oil and the nature of the herb, as the heat content and temperature of steam can alter the distillation characteristics. For high boiling oils such as woody oils (e.g. sandalwood, cedar wood) and roots (Cyperus), 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. For oil of rose and other florals, the material is generally immersed in water, hydrodistillation, as flowers tend to aggregate and form lumps which cannot be distilled using water and steam distillation or direct steam distillation.

◆ Proper design of equipment

Improper designing of tank, condenser or separators can lead to loss of oil and high capital investments. The design of the furnace and chimney affects the firing and heat control of the distillation rates. Tank height: diameter ratio is important. 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.

◆ Material of fabrication 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 the tank still, the rust and other products of corrosion may not be carried over into the oil. This can result in considerable savings in the capital cost of the equipment. Expensive, high-value essential oils like rose, agar wood, kewda, sandalwood and lavender should be distilled in stainless steel systems. 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.

◆ Condition of raw material

The condition of the raw material is important because some materials like roots and seeds will not yield essential oil easily if distilled in their natural state. These materials have to be crushed, powdered or soaked in water to expose their oil cells. Chopping of plants will also change the

packing density of the material when placed in the distillation still. One can pack up to 50% more plant material in the same still after chopping of some aromatic herbs like mint. Air drying and wilting the herb prior to distillation also has considerable effect on distillation. If required, drying of the herbs prior to distillation should be done in shaded areas and the dried material should not be kept in heaps.

◆ Time for distillation

Different constituents of the essential oil get distilled in the order of their boiling points. Thus, the highest boiling fractions will be last to come over when generally very little oil is distilling. If the distillation is terminated too soon, the high-boiling constituents will be lost. In many aromatic plants, like vetiver, patchouli, chamomile, sandalwood and agar wood, these high-boiling fractions are valuable due to the quality of their aromas. Thus, the time of distillation must be chosen with due care.

◆ Loading of raw material and steam distribution

Improper loading of the herb may result in steam channeling, causing incomplete distillation. The herb should be evenly and uniformly loaded in the tank without leaving any voids. Excessive filling of plant material may also lead to formation of “rat holes” which may allow steam to escape without vaporizing the oil. For powdered herbs, a proper stainless steel wire mesh or muslin cloth should be put at the false bottom to prevent plant material from falling into the tank base.

◆ 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.

◆ Condition of tank and equipment

The tank and other equipment should not be rusted. If rusted, the tank should be cleaned with dilute caustic solutions. The perforated grids should not be corroded or have large gaps permitting the plant material to settle to the bottom of the tank and emit a burnt odor. The distillation tanks should be well steamed prior to distillation for multiple crop distillation.

2.12 Raw material selection

Aromatic and Perfume Plants: There are number of indigenous and introduced aromatic and perfume plants in Ethiopia. Some of these are Commiphora spp, Boswellia spp, Cinnamomum cassia, Juniperus procera, Echinops spp, Olea europaea subsp. Africana, Otostegia spp, Ocimum spp, Artemisia spp, Cymbopogon citratus, Cyperus bulbosus, Myrtus communis (Genetic, 1996).

2.12.1. Lemongrass as raw material for perfumes.

Lemongrass oil which is used in a range of industrial products where a lemon flavor is required, can also provide citral that can be modified into β -ionone and methyl-ionone and serves as a starting precursor for vitamins A and E synthesis (Robbins 1983). Lemongrass is one of the best important perfumery material and flavoring. Large quantities of lemongrass generated as result of the high demand of flavor in worldwide. Most of which are native to south Asia, Southeast Asia and Australia. Two major types have considerable relevance for commercial use: East Indian lemongrass (cymbopogon flexuous) is native to India, Sri Lanka, Burma and Thailand, whereas the West Indian oil is extracted from cymbopogon citratus (Dc) stapf that is mainly cultivated in central and South America and also known in parts of Africa, south East Asia and the Indian Ocean islands. There are two types of lemongrass species that are cultivated in Ethiopia. Those are cymbopogon citratus and nardus that were experimentally recognized by the book of Ethiopian and Eritrea flora.

2.12.2. Chemical composition of lemongrass essential oil

The main chemical components of lemongrass oil are myrcene, citronellal, geranyl acetate, nerol, geraniol, neral and traces of limonene and citral. The major terpenes in C citratus include citral- α or geranial (10–48%) and citral- β or neral (3–43%), borneol (5%), geraniol (2.6–40%), geranyl acetate (0.1–3.0%), linalool (1.2–3.4%) and nerol (0.8–4.5%) (Akhila *et al*, 2010).

2.13 Production Process of essential oil from lemongrass

Lemongrass essential oil was extracted by steam distillation of the fresh or dried leaves of lemongrass; the oil of lemongrass was yellow in colour with a citrus grass (lemon fragrance).

◆ Drying

Lemongrass was obtained from garden, were dried as soon as possible so as to diminish the risk of fungal growth before oil extraction or mushroom colonization. Results were achieved at room temperature with exposure to direct sunlight and frequent spreading.

◆ Size Reduction

The dried plant material was disintegrated by feeding it into a rotary cutter that was used to reduce the size of the leaves into desired size. This can increase the yield of the oil during the process. Cutting of leaves enhance and help catalyze the production of the oil during the process. Furthermore, size reduction maximizes the surface area, which in turn enhances the mass transfer of active principle from plant material to the steam. It is an advantage also because cutting can increase the quantity of the grass fed during the operation.

◆ Still tank

The still or retort serves as a container for the plant material. It is also a vessel in which the water or steam contacts the plant material and vaporizes its essential oils. The retort consists merely of a cylindrical container or tank with a diameter equal to or slight less than its height. The height of the still for direct steam distillation should be greater than the diameter so that the rising steam passes as much plant material as possible. It is equipped with a removable cover, which can be clamped upon the cylindrical section. A pipe (gooseneck) is attached to the top of the cylindrical section for leading the vapors to the condenser.

◆ Condenser

The condenser serves to convert all the steam and the accompanying oil vapor into liquid. This requires the removal of an amount of heat equivalent to the heat of vaporization of the vapor plus steam and a small additional amount of heat to cool the condensate below its boiling point.

◆ Separator

The third part of the distillation equipment consists of the condensate receiver, decanter or oil separator. It separates the oil from the condensed water. Since the total volume of water condensed will be greater than the quantity of the oil, it is necessary to remove the water continuously. The condensate flows from the condenser into the oil separator, where the distillation water and volatile oil separate automatically.

◆ Steam Generator

This is the part of the distillation plant that generates heat due to boiling water. It is a cylindrical container in which 9KW heater is placed. The steam outlet (which is at the top) is attached directly to the bottom of the Still. Below the container are the contacts for the wire which will be connected to the circuit box.

◆ Storage and packaging

The oil should be stored in glass bottles or containers made of stainless steel or aluminum or galvanized iron, depending on the quantity of oil to be stored. The oil should be filled up to the brim, and the containers should be kept away from direct heat and sunlight in cool or shaded places. The oil should be stored in well-sealed glass bottles, at 5°C–25°C, and in a dry, well ventilated area away from direct heat and sunlight. Lemongrass oil can be stored for up to 3 years without affecting the quality of oil if kept in aluminum containers sealed airtight using wax. Containers should be completely filled to exclude any air and protect the oil from sunlight as air and sunlight affect the citral content.

2.14 Perfume Formulation from lemongrass essential oil.

2.14.1 Production Process of perfume from lemongrass essential oil

◆ Blending:

Once all of the oils for the perfume have been extracted, they need to be blended together. The oils are blended accorded to a formula that has been predetermined by a master in the perfume industry, often referred to as a "nose." One perfume can contain hundreds of different ingredients and can take multiple years to develop. Once the perfume oils have been blended, the scent is mixed with alcohol. The amount of alcohol added to the perfume oils varies depending on what the final product will be being. Eau de parfum will have a lower concentration of alcohol while scents such as body mists will have a much higher concentration of alcohol. Most perfumes are made of about 10-20% perfume oils dissolved in alcohol and a trace of water.

◆ Aging:

Higher quality or fine perfumes are oftentimes aged for months or potentially even years after the scent has been blended. This is done to ensure that the proper scent has been created. Aging allows the different scents or notes to really blend together. Perfumes are made up of top notes that provide the scent with body as well as base notes which create an enduring fragrance.

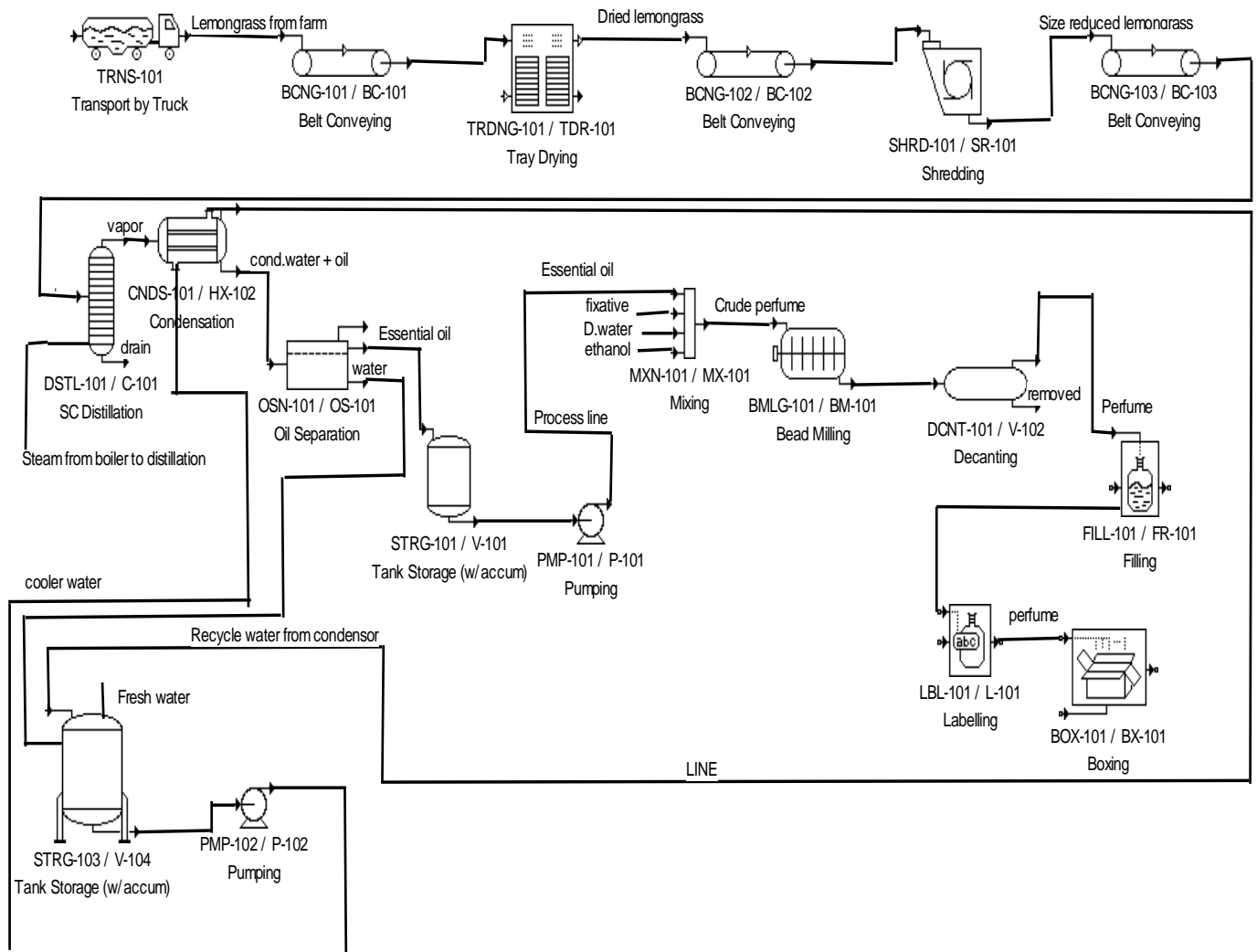


Figure 2.2 Process flow diagram of perfume formulation.

3. Materials and Methods

The experimental work has been done in laboratory of Addis Ababa institute of Technology School of Chemical and Bio-Engineering and college of natural science of Addis Ababa University school of physics and chemistry department, Addis Ababa Ethiopia.

3.1 Materials and Equipment

3.1.1 Characterization and Optimization of essential oil extraction from lemongrass

Materials used during extraction and characterization of lemongrass essential oil were: 1000ml Separation funnel, 250ml and 1000 Beakers, Electronics weighting balance, Water bath, 2000ml Round bottom flask, Knife, Electric heater, Distilled water, Anhydrous sodium sulfate, sodium bisulphite, steam distillation, centrifugal miller, Scissors, aluminum foil, density separator, black bottle, centrifugal separator, Gas chromatography - Mass spectroscopy, FT-IR, UV visible spectroscope, oven, sieve, Polarimeter, Spectrometer, Cuvet, density bottle (pycnometer) etc.

3.1.2 To develop perfume and characterize physical property using sensor analysis.

Materials that used during perfume development and sensor analysis were: Micro-Pipette, Funnel, 50ml and 120ml beakers, black glass, perfume bottle, shaker, centrifuge separator, (glycerin and castor oil), carrier solvent ethanol (96%), Distilled water, Lemon grass essential oil, panalists, paper and pen.

3.2 Experimental methods

3.2.1 Characterization and Optimization of essential oil extraction from lemongrass.

3.2.1.1 Optimization of essential oil extraction from lemongrass using steam distillation

3.2.1.1.1 Raw material preparation

Lemongrass (*Cymbopogon Citratus*) leaves were purchased from home garden in Wonji sugar factory and farmer who live around camp in south of Addis Ababa, Ethiopia.



Figure 3.1 Lemon grass from a domestic backyard garden in Wonji

Species was investigated in biology department of herbal in Addis Ababa University and then the plant sample was freshly cut 10cm from the root in the morning of the day. They were collected according to (Edwin *et al.*, 2012).Some dirty material was removed. The plant material divided in two parts to do an experiment, those were fresh and dry. Half fresh *Cymbopogon citratus* leaves were dried at room temperature for five days. Leaves were extended on trays, turned them three times daily for ventilation; accelerate drying and preventing the growth of microorganisms. Lastly it was kept in plastic bag after drying, whereas the fresh lemongrass was spreaded to avoid micro-organism and for both size reductions were done based on required size, then experiment was done by putting it into still to extract the oil based on the weight size which was decided.

3.2.1.1.2 Determination of moisture content of the lemongrass

Fresh lemongrass was collected on December 2015; after it was collected half of them were dried by sun for 5 days. According to Buggle *et al.* 1997 carried out an experiments to study the effects of drying temperature on the amount and quality of essential oils extracted from *C. citratus* and Leaf blades were cut into small parts (about 1 –1.5 cm in length) and dried for several days at 30°C, 50°C, 70°C, or 90°C, until a constant weight was achieved the higher amount of oil was collected at lower drying temperatures. So drying atmospheric temperature is good. But,other as it was wet. Six sample of the lemongrass randomly were weighted for wet 33.3, 43.8, 44.4, 45, 44.7, 74.44 grams and four samples for dry 17.04, 45.87, 41.42 and 41.30 were taken randomly and then dried in oven at 105⁰C and the weight was measured every two hours. The procedure was repeated until a constant weight was obtained and the percentage moisture content of the lemongrass was determined (General, 2005). The moisture content of the sample was determined using equation (3.1).

$$\text{Moisture \%} = \frac{w_1 - w_2}{w_1} \quad (3.1)$$

Where, W_1 = Original weight of the sample before drying

W_2 = weight of the sample after drying

3.2.1.1.3 Size reduction

After the moisture was removed by sundry then it was milled in crush miller for dry and fresh lemongrass with sieve size 10mm and cut with scissors for size of 20mm and 30mm.

3.2.1.1.4 Essential Oil extraction

The oil extraction was conducted using steam distillation with duplicate with two different conditions: dry and wet raw material.



Figure 3.2 Steam distillation of Chemical and Bio engineering of AAiT

The extraction was carried out at different size and time with extraction temperature of 94.5°C and at atmospheric pressure. The set-up for steam distillation of lemon grass oil consists of two distillation stills and condenser. The distillation still has a capacity to hold 1-6 kg of plant material per batch. One kilogram of lemongrass with two different leave conditions, fresh and dry were fed in steam distillation at four different times: 1.5 hour, 2hour, 2.5 hour, 3hour with different particle size to compare their efficiency both on the oil yield. Steam was generated by heating water in boiler. Steam thus generated was passed in the first still that held the raw material. The two still were connected by connecting tubes. A condenser is provided with second by connector. Condenser consisted two connecting tubes. Water was passed through outer tube and mixture of steam and oil passed through inner spiral tube. The condensed mixture of oil and water was collected in the collector. The whole apparatus was properly sealed to avoid leakage. Oil being lighter floats on the surface of water. The mixture of oil and water was separated in the separating funnel. Anhydrous sodium sulphate was used for drying. The products were weighted and the essential oil physicochemical properties were determined.

3.2.1.1.5 Determination of percentage of oil extracted

The percentage yield was calculated in two forms i.e. oil yield and extraction yield using the formula below.

Since lemongrass have an oil content of 1-2%, average 1.5% oil content was taken for calculating the yield, Therefore:

$$\text{Percentage oil yield} = \frac{\text{mass oil}}{0.015 \times \text{mass of the sample}} \times 100\% \quad (3.2)$$

The second form that the yield calculated was

$$\text{Percentage extraction yield} = \frac{\text{mass oil}}{\text{mass of the sample}} \times 100\% \quad (3.3)$$

3.2.1.2 Characterization of essential oil extracted from lemongrass

The specific gravity at 25⁰C, density, dynamic viscosity, kinematic viscosity, PH value, flash point, refractive index, optical rotation, were analyzed using standard procedures. The result was compared with standard lemongrass oil. The acid value, Saponification value, total aldehyde content expressed as citral and ester were performed and compositional analysis also was done by GC-MS, FT-IR, and UV-visible spectrometer.

3.2.1.2.1 Determination of some of physical properties of lemongrass oil

3.2.1.2.1.1 Determination of kinematic viscosity of lemongrass oil

A dynamic viscosity of the essential oil was measured directly using vibro-viscometer which was available in laboratory of school of Chemical and Bio Engineering. Initially, a sample was heated at a temperature of 20⁰c. A sample of 35ml oil was measured and fed to a sample holder of the vibro-viscometer. A sensor of the viscometer was immersed to the oil and then a dynamic viscosity of oil was displayed on the vibro-viscometer screen at a temperature of 20c⁰ and then the kinematic viscometer was calculated.

3.2.1.2.1.2 Determination of PH

Two gm of the sample was taken and putted in a clean dry 25ml beaker and 13ml of hot distilled water was added to the sample in the beaker and stirred slowly. And then it was cooled in cold water bath to 25⁰c. The PH electrode was standardized with a buffer solution first and then immersed in the sample and the PH was read (A.O.A.C official method analysis 960.19, 2000).

3.2.1.2.1.3 Determination of boiling temperature of the essential oil

25ml of lemongrass oil poured in to beaker and a thermometer was inserted and placed on a heating mantle, it was observed that the oil in the beaker started circulating leading to boiling of oil and read temperature on thermometer.

3.2.1.2.1.4 Determination of moisture and volatile matter of essential oil

Moisture content of oils and fats is the loss in mass of the sample on heating at 105⁰C under operating conditions specified. 5gm of oil was weighted and putted in a dish and then dried in an oven at 105⁰C for 1hour. The dish was removed from the oven and cooled in a desiccator and

weighed. The process was repeated until a constant weight was observed and the moisture and volatile matter of the oil was determined (Hand book of food analysis, 1984).

3.2.1.2.1.5 Determination of the specific gravity

The density of the oil was determined by using density bottle. A clean and dry bottle of 25ml capacity was weighed (W_0) and then the bottle was filled with the oil, stopper inserted and reweighed to give (W_1). The oil was substituted with water after washing and drying the bottle and weighed to give (W_2) (General, 2005).

The expression for specific gravity (Sp.gr) is:

$$\text{Sp.gr} = \frac{(X_2 - X_1)}{(X_3 - X_1)} = \frac{\text{mass of sample}}{\text{mass of an equal volume of water}} \quad (3.4)$$

3.2.1.2.1.6 Determination of refractive index of lemongrass oil

The refractive index of a substance is the ratio of the speed of light in a vacuum to the speed of light in the substance. The scales of standard instruments indicate refractive indices with respect to air rather than vacuum. Refractive Index of the sample was determined indirect method using prism spectrometer, at physics department of natural science of Addis Ababa University. It was certain that the temperature of the spectrometer is 25.0°C at room temperature, wave number 632nm and the prism was cleaned and completely dry. Placed the prism of the sample (cuvet) then it was adjusted the instrument and light to obtain the most distinct reading and then determined the refractive index using Brewster's angle. Brewster's angle can be calculated from the two refractive indices of the interface as:

$$\theta b = \arctan(n_2/n_1) \quad (3.5)$$

Where, n_2 = Refractive index, θb angle of refraction, n_1 the refractive index of air is 1.

The index of refraction of oils is characteristic within certain limits for each kind of oil. It is related to the degree of saturation particularly to the extent of conjugation, but it is affected by other factors such as free fatty acid content, oxidation and heat treatment.

3.2.1.2.1.7 Determination of optical rotation of lemongrass oil

Polarimeter with a standard 10 cm tube (cuvet) and a sodium vapor lamp was adjusted and volatile oil sample and instrument temperature was 25°C (98°F). The Polari meter tube was filled with oil and wipe off excess oil on the exterior. Then Placed the tube in the Polari meter then Slowly turned the analyzer until both halves of the field, viewed through the telescope, Reading was observed (General *et al.*, 2005).

3.2.1.2.2 Characterization of the chemical properties of lemongrass oil

3.2.1.2.2.1 Acid value

25ml of toluene and 25ml of ethanol was mixed in a 250ml beaker. The resulting mixture was added to 2g of oil in a 250ml conical flask and few drops of phenolphthalein were added to the mixture. The mixture was titrated with 0.1M KOH to the end point with consistent shaking for which a dark pink colour was observed and the volume of 0.1M KOH (V_0) was noted. The Acid value was calculated as (General *et al.*, 2005).

$$\text{Acid value} = \frac{56.1 \times V \times N}{M} \quad (3.6)$$

Where V = Volume in ml of potassium hydroxide solution, N = Normality of the potassium hydroxide solution, M = Mass in gm of the material taken for the test.

3.2.1.2.2.2 Determination of Saponification Value

Indicator method was use as specified by ISO 3657 (1988). 2g of the sample was weighed into a conical flask; 25ml of 0.5N Ethanolic potassium hydroxide was then added. The content which was constantly stirred was allowed to boil gently for 30min. A reflux condenser was placed on the flask containing the mixture. Few drops of phenolphthalein indicator was added to the warm solution and then titrated with 0.5M HCl to the endpoint. Until the pink colour of the indicator just disappeared. The same procedure was used for other samples and blank. The expression for saponification value (S.V.) is given by:

$$\text{Saponification value} = \frac{56.1 \times N \times (V - W)}{M} \quad (3.7)$$

Where, N = normally of hydrochloric acid used for titration, V = Volume in ml. of hydrochloric acid used for the blank, W = Volume in ml. of hydrochloric acid used in determination = Mass in gm of the material take(General *et al.*, 2005).

3.2.1.2.2.3 Determination of aldehyde content

Into a 150 cc cassia flasks, having a thin neck graduated in 0.1cc divisions, 75 cc of a freshly prepared, saturated, aqueous solution of sodium bisulfate was poured into graduated cylinder. Then 10 cc of the oil was added into the flask. Upon thorough shaking, a semisolid mass frequently was resulted. It was immersed the flask in a beaker of boiling water until the solid compound has gone completely into solution. It was shacked flask repeatedly to assure complete reaction of the aldehyde with the bisulfite solution. A further addition of 25 cc of bisulphite solution was made, and the flask was again repeatedly shaken. After standing undisturbed in the

beaker of boiling water for 10 min, it was permitted the unreacted oil to rise to the surface. It was added sufficient sodium bisulfate solution to force the unreacted oil into the neck of the flask and any droplets of oil adhering to the sides are made to rise into the neck by gently tapping the flask, and by rotating it rapidly between the palms of the hands. It was cooled the flask to room temperature and measured the amount of unreacted oil. The aldehyde content calculated by means of the following formula.

$$\text{Percentage of aldehyde} = 10(10 - \text{No. of cc of unreacted oil}) \quad (3.8)$$

As mentioned above, this result is a volume percentage. It may be converted into a weight percentage if the specific gravity of the original oil and of the aldehyde is known (A.O.A.C official method, 2000).

3.2.1.2.2.4 Ester value, content of esters and combined alcohols

The determination of the ester content is of great importance in the evaluation of many essential oils. Ester value may be defined as "the number of milligrams of potassium hydroxide required to neutralize the acids liberated by the hydrolysis of esters present in 1 g m of the perfumery materials". The value of ester can be calculated as follow

$$\text{Ester value} = \text{Saponification value} - \text{acid value} \quad (3.9)$$

3.2.1.2.2.5 Gas Chromatography-Mass Spectroscopy

The component identification was achieved by the GC-MS analysis using HP 5890 series GC equipped with mass selective detector (MSD), HP 5972 series (German) in Addis Ababa University. Helium was used as carrier gas at a constant flow of 1 ml/min and an injection volume of 1 μ l was employed, injector temperature 250 °C; Ion-source temperature 280 °C. The oven temperature was programmed from 50 °C (isothermal for 4min.), with an increase of 3 °C/min, to 280 °C and held for 10min. isothermal at 280 °C. Total GC running time was 90.67 min.

3.2.1.2.2.6 Fourier Transform Infrared Spectroscopy (FT-IR)

The FT-IR spectrum of the essential oil was obtained using prinks Elmer spectrum 65 (FT-IR) Spectrometer in Addis Ababa University and functional groups were determined with the help of IR correlation charts. The IR spectra were reported in % transmittance. The wave number region for the analysis was 4000-400 cm⁻¹(in the mid-infrared range).

3.2.2 To develop perfume and characterize physical property using sensor analysis

3.2.2.1 Perfume development from lemongrass essential oil

Procedure:

lemongrass essential oil extract was measured and placed in 120ml beaker with measured containing carrier solvent of ethanol then it was shaken by shaker and again fixative was added in solution mixture to improve the longevity of the perfume. In addition to that distilled water was also added to the mixture to homogenized undissolved ingredient. The mixture was poured into black bottle then it was placed in the dark area to concentrate the perfume for one month. Finally it was filled in the bottle of perfume.

3.2.2.2 To characterize physical properties of perfume using sensor analysis.

Sensory attributes such as colour, aroma and overall acceptability of all the three incorporated in perfume formulation from lemongrass additive were evaluated by method recommended by Ranganna (1994) using a Hedonic Rating test. Women panelists were selected to evaluate samples through properly planned experiment. Their judgments were recorded and appropriate analysis was carried out to determine the significance of variations of average score and the contribution of individual parameter. Samples were served to the panelists and they were asked to rate the acceptability of the product on 1–9 points scale, ranging from the extreme like (9) to dislike extremely (1).

3.3 Design of the Experiment

3.3.1 Full Factorial Design

Data analysis has performed by design expert software using general factorial design Method. For steam distillation extraction we had two factors, time and particle size with three levels for size and four levels for extraction time for dry and fresh raw material of lemongrass while temperature was fixed due to uncontrollable of steam distillation. Even if temperature and pressure are the main factor for steam distillation in addition to time and size of the particle, the best recommended temperature around boiling point of water and pressure is atmospheric.

This design of the experiment helps us to differentiate the significance of the main and the Interaction factors. This program software also used to develop the mathematical model that will describe the effects of the main and interaction factors on the response.

4. Results and Discussion

4.1 Characterization and Optimization of essential oil extraction from lemongrass

4.1.1 Optimization of essential oil extraction from lemongrass using steam distillation.

4.1.1.1 Moisture content of the lemongrass

Table 4.1 Moisture content of dry lemongrass

	Sample weight in gram			
	Sample 1	Sample 2	Sample 3	Sample 4
Initial mass	17.04	45.87	41.42	41.30
Mass after 2hrs	16.60	43.84	40.41	40.72
Mass after 4hrs	16.21	43.56	40.11	40.57
Mass after 6hrs	16.01	43.44	40.01	40.31
Mass after 8hrs	16.01	43.31	40.0	40.02
Mass after 10hrs	16.01	43.31	40.0	40.02
Final moisture content	6.10%	5.5%	3.42%	3.09%

The moisture content of the four lemongrass sample having mass of 17.04, 45.87, 41.42, 41.30 gm were 6.10, 5.5, 3.42 and 3.09 percent respectively. The mean average moisture content of the four samples will be 4.52%.The result obtained was different from Vázquez-briones *et al.*, 2015 which moisture content of the dried *C.citratrus* $9.14 \pm 0.70\%$ due to geographical variation of it.

Table 4.2 Moisture content of fresh lemongrass.

	Sample weight in gram					
	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
Initial mass	10.5	15.2	12.4	10.6	16.1	8.9
Mass after 2hrs	9.5	13.5	11.5	9.9	14.8	7.62
Mass after 4hrs	8.9	13.13	10.7	8.7	14.1	7.25
Mass after 6hrs	8.73	12.6	10.13	8.6	13.8	7.23
Mass after 8hrs	8.72	12.31	10.11	8.5	13.55	7.21
Mass after 10hrs	8.72	12.31	10.11	8.5	13.55	7.21
moisture content	16%	19.01%	18.52%	19.82%	15.83%	18.98%

The moisture content of the six lemongrass sample having mass of 10.5, 15.2, 12.4, 10.6, 16.1 and 8.9 grams were 16, 19.01, 18.52, 19.82, 15.83, 18.98 percent respectively. Thus, the mean average moisture content of the six samples were 17.83 %.The recommended moisture content of fresh lemongrass was $73.28 \pm 4.98\%$ but, The result obtained was not in agreement to The moisture content of the fresh *C. citratus* due to dryness of lemongrass during transportation and extraction period.

4.1.1.2 Essential Oil extraction

4.1.1.2.1 Percentage yield of extraction for dry lemongrass

In this section outline of the results obtained from steam distillation lemongrass essential oil experiments are discussed. The percentage extraction yield was calculated by using equation 3.3 and the result is summarized in the table 4.3 below.

Table 4.3 Total %yields of essential oil for different particle size and time

Factors					
Time(minute)	Particle size (mm)	Replicate 1 (grams)	Replicate 2 (grams)	Average (grams)	Extraction yield (%)
90	0.8-15	4.3	3.5	3.9	0.39
	15-25	2.7	3.3	3	0.3
	25-35	2.4	2.1	2.25	0.225
120	0.8-15	6.8	6.3	6.55	0.655
	15-25	5	5.4	5.2	0.52
	25-35	3.8	4.3	4.05	0.405
150	0.8-15	8.3	7.9	8.1	0.81
	15-25	7.2	6.7	6.95	0.695
	25-35	5.7	5.3	5.5	0.55
180	0.8-15	7.8	8.1	7.95	0.795
	15-25	7.1	6.9	7	0.7
	25-35	5.8	5.4	5.6	0.56

Two factors were selected because of uncontrollable of equipment especially temperature. Even if temperature was not taken as factor, the recommended temperature was around boiling point (Guenther, 1972). Due to that the extraction temperature was 94.5⁰C and water boil at 92.5 ⁰C in Addis Ababa. So it was not that much difference based on the recommended. Based on that the maximum extraction of lemongrass oil was 0.81% at particle size ranges from 8-15mm for the extraction time of 2.5 hours and the minimum yield obtained was 0.225 % at maximum Particle size and minimum extraction time. According to Boruah *et al.*1995 Healthy and uninfected leaves yielded 0.80% oil, while leaves with a disease index of (60–75%) yielded 0.50% oil. From that what was concluded that extraction of essential oil high from health leave, properly prepared and uninfected by micro bacteria.

4.1.1.2.2 Percentage yield of extraction from fresh lemongrass

Results obtained from steam distillation of essential oil from fresh lemongrass

Table 4.4 percentage essential oil yields of lemongrass for two factors

Factors					
Time(minute)	Particle size (mm)	Replicate 1 (grams)	Replicate 2 (grams)	Average (grams)	Extraction yield (%)
90	0.8-15	2.1	1.8	1.95	0.195
	15-25	1.6	1.4	1.5	0.15
	25-35	1.2	1.3	1.25	0.125
120	0.8-15	2.9	3.2	3.05	0.305
	15-25	2.4	2.6	2.5	0.25
	25-35	1.8	2	1.9	0.19
150	0.8-15	3.2	3.7	3.45	0.345
	15-25	3.1	2.7	2.9	0.29
	25-35	2.3	2.2	2.25	0.225
180	0.8-15	3.6	3.3	3.45	0.345
	15-25	2.6	3	2.8	0.28
	25-35	2.1	2.4	2.25	0.225
	25-35	2.1	2.4	2.25	0.225

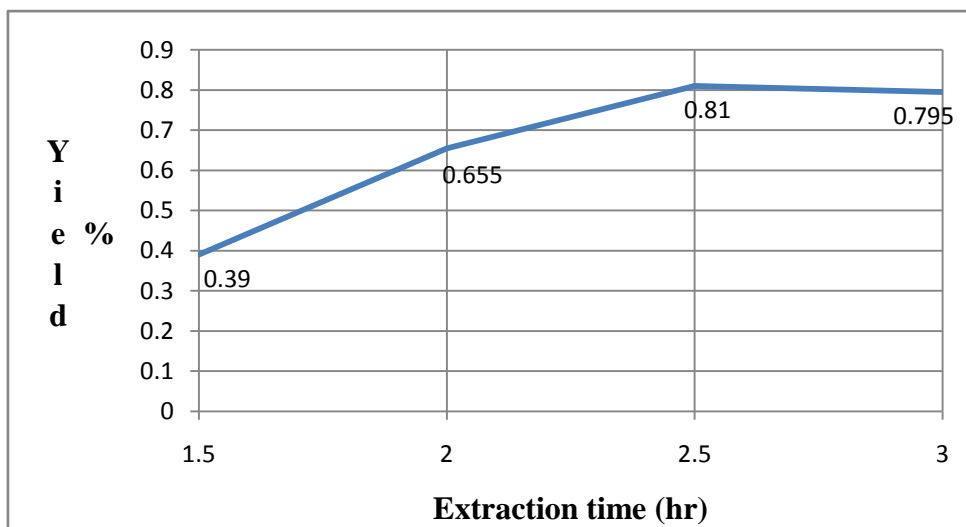
The maximum extraction of lemongrass essential oil was 0.345% for fresh weight. This result agrees with some works who reported that oil content of a normal cut should average 0.25–0.50%, but with good management and selected strains could be yielded up to 0.66–0.90% (Weiss, 1997; Maiti *et al.*, 2006). At particle size ranges from 8-15mm for the extraction time of 2.5 hours and the minimum yield obtained was 0.125 % at maximum Particle size and minimum extraction time.

4.1.1.3 Effect of process parameters in percentage essential oil yield of lemongrass

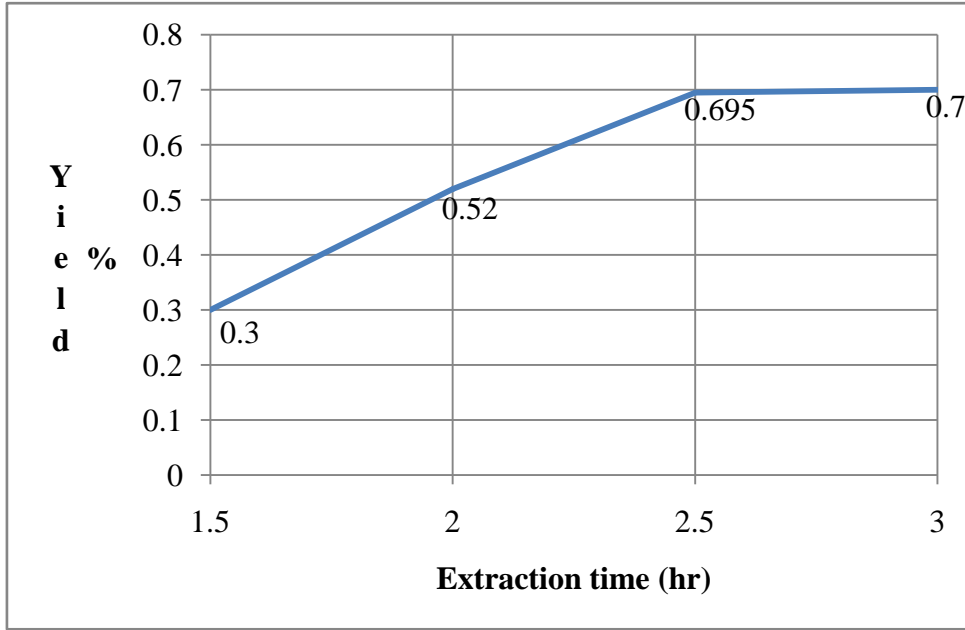
4.1.1.3.1 Effect of process parameters in percentage essential oil yield of dry lemongrass.

- ◆ Effect of extraction time on percent yield of essential oil.

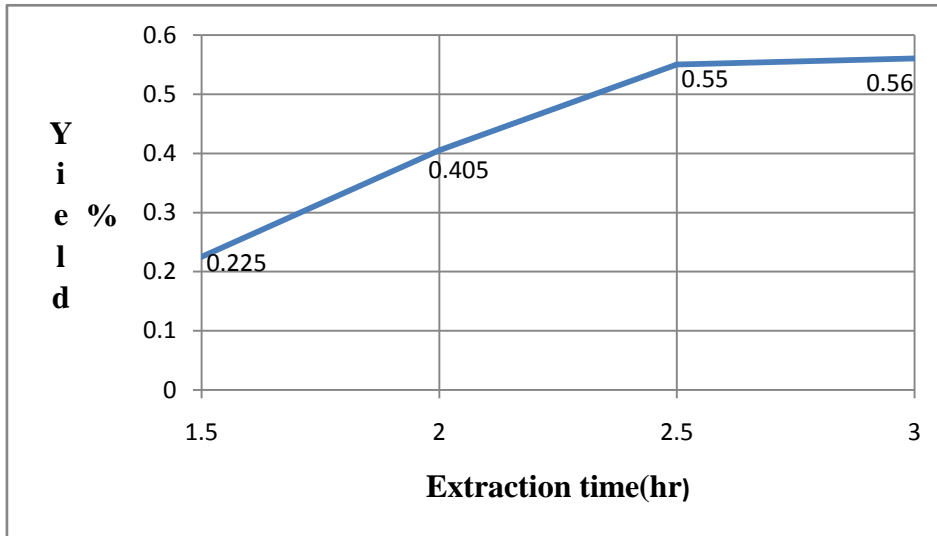
Effect of Extraction time Figure 4.1 shows the influence of extraction time on the extraction yields of Lemongrass (*Cymbopogon Citratus*) over the range 90-180 min under a fixed temperature and different raw material size. As being depicted in figure.4.1 most of the oil was being extracted within 90 to 150 minutes and the amount of yield obtained up to this period was 0.81%. However, further increase in extraction time resulted in no significant improvement in the extraction performance. In general, the rate of extraction was high at the beginning of the extraction but get slow gradually by time. These results confirmed the Fick's second law of diffusion which stated about the final equilibrium achieved by the solute concentrations in plant matrix and in the solvent after a certain time. This caused into no significant improvement in oil yield when prolonging the extraction time.



(a)



(b)



(c)

Figure 4.1: The effect of time on lemongrass oil yield at particle size (a) 8-15mm, (b) 15-25mm and (c) 25-35mm

◆ **Effect of particle size on percent yield of lemongrass of essential oil.**

Particle size plays a great role on the yield of lemongrass oil. Smaller the size gives high yield while samples with large particle size deliver low yield.

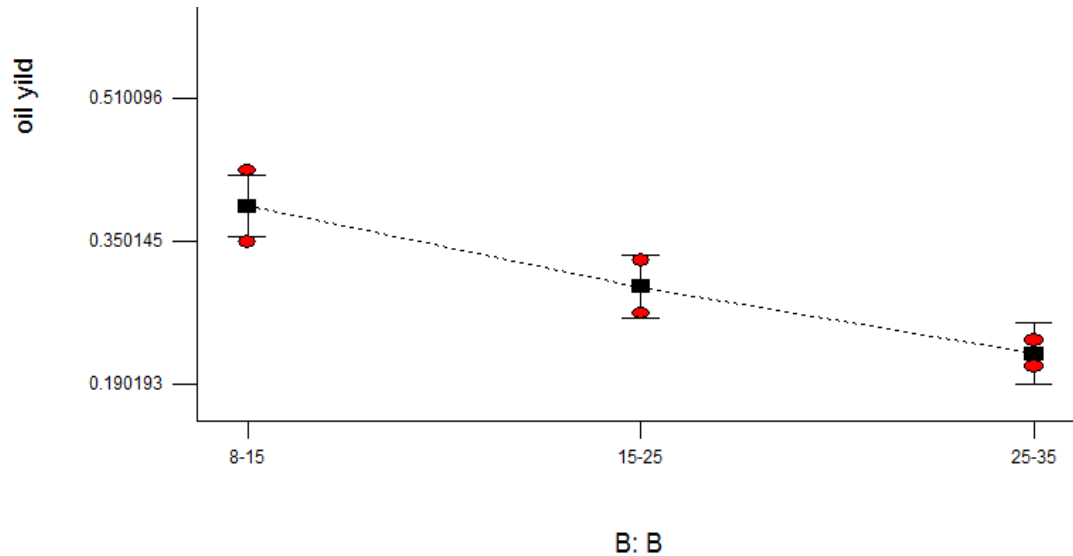


Figure. 4.2 effect of particle size on the percentage of essential oil yield.

From above figure 4.2 what we observed less essential oil is extracted from the larger particles (>25 mm) compared to the small size of the particles. The reason is that larger particles with smaller contact surface area have more resistant to steam entrance and carried out. Therefore, less amount of oil will be transferred from inside to the surrounding vapor in comparison with the smaller ones. Thus, an increase in particle size will decrease the oil yield. Nevertheless, we know that when the particle is too small (very fine chopped), 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. This May be due to the agglomeration of the fine particles which reduces the effective surface area available for the free flow of steam towards inside the solid particles (Spider sayyar *et al* 2009) reported the result of extraction of oil from jatropha seed for three different particle size range <0.5mm, 0.5-0.75mm and >0.75mm. The highest percentage of oil yield was obtained with the intermediate particle size (0.5-0.75) which indicate that decreasing the particle size below certain particle size doesn't increase the percentage of oil yield and may even decrease the yield.

◆ **Interaction effect between two factors.**

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

DESIGN-EXPERT Plot

oil yild

X = A: A

Y = B: B

◆ Design Points

■ B1 8-15

▲ B2 15-25

◆ B3 25-35

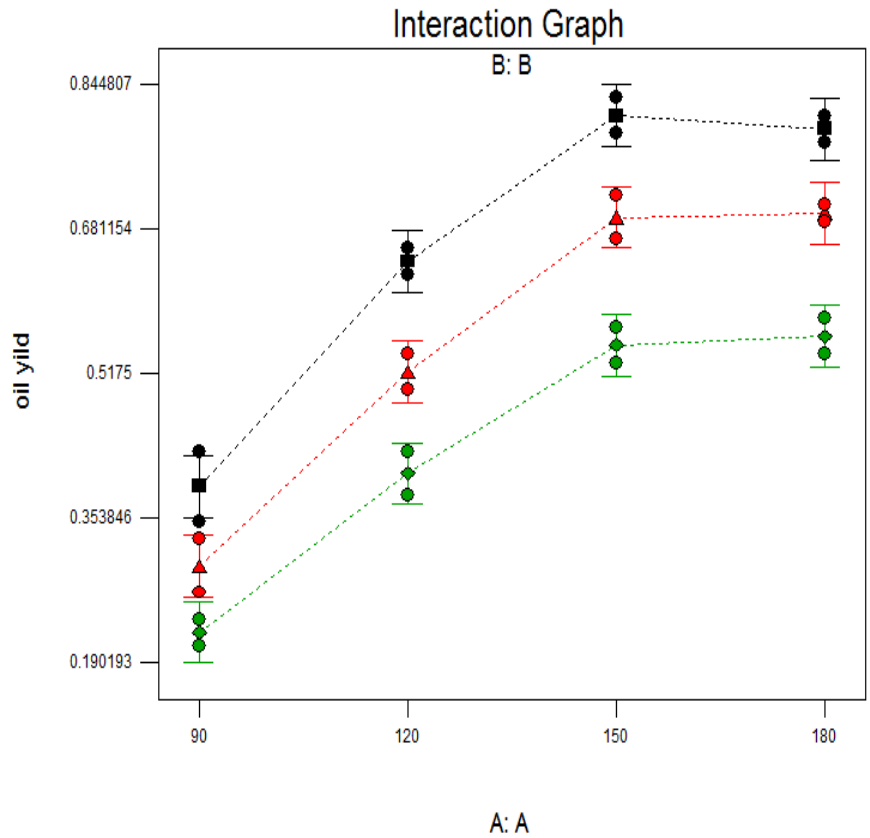


Figure. 4.3 interaction effect of particle size and extraction time on oil yield

Where, B1, B2 and B3 are codes for particle sizes ranges from 8-15mm, 15-25mm and 25-35mm respectively. A1, A2, A3 and A4 are codes for extraction time (hours) one and half, two, two and half and three hours, respectively. Design points are points on the graph which helps to develop mathematical model of the predicted response based on these points. Design -expert was applied to analyze results on the extraction process and a first order regression equation, with the interaction terms of the form:

◆ **Regression Model Equation**

The following table shows analysis of variance (ANOVA), attained from design expert soft ware which tell us the significance of different factors.

Table 4.5 Analysis of variance (ANOVA) for a response of percentage essential oil yield.

Source	Sum of square	Degree of freedom	Mean square	F value	P value Prob >F
Model	0.79	11	0.072	70.52	< 0.0001
A-Extraction time	0.58	3	0.19	190.40	< 0.0001
B-Particle size	0.20	2	0.1	99.29	< 0.0001
AB	6.025E-003	6	1.004E-003	0.98	0.4772
Pure Error	0.012	12	1.021E-003		
Cor Total	0.80	23			

F value (F model = 70.52) with low probability value (P < 0.0001) indicates the significance of the fitted model. 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. The P-value of AB (interaction factors) is 0.4772 >P-value thus, the interactions of size and time are not significant in the model terms.

Table 4.6 Design summary essential oil extracted from dry lemongrass

Design summary							
Study type	Factorial		Experiment :24				
Initial design	Full factorial		Blocks	No blocks			
Center point zero(0)							
Design model 2FI							
Response	name	units	obs	minimum	maximum	trans	model
Y	Oil yield	%	24	0.21	0.83	none	2FI
Factor	name	units	type	Low actual	High actual		
A	A		categorical	90	180	level	4
B	B		categorical	8-15	25-35	level	3

Table 4.7. Constraints and solution of essential oil yield for dry lemongrass (citrus)

Constraints						
Name	Goal	Lower limit	Upper limit	Lower weight	Upper weight	Importance
A	Is in range	90	180	1	1	3
B	Is in range	8-15	25-35	1	1	3
Oil yield	maximize	0.21	0.83	1	1	3
Solution						
number	A	B	Oil yield	Desirability		
1	<u>150</u>	<u>8-15</u>	<u>0.81</u>	<u>0.968</u>	<u>selected</u>	
2	180	8-15	0.795	0.944		
3	180	15-25	0.7	0.790		
4	150	15-25	0.695	0.782		
5	120	8-15	0.645	0.702		
6	180	25-35	0.56	0.565		
7	150	25-35	0.55	0.548		
8	120	15-25	0.52	0.500		
9	120	25-35	0.405	0.315		
10	90	8-15	0.39	0.290		
11	90	15-25	0.3	0.145		
12	90	25-35	0.225	0.024		

Consider ANOVA table (4.5) the model terms A and B were significant model terms whereas interaction model terms AB is not significant model terms. The final model equation in terms of coded factor was presented by equation 4.2

Final Equation in Terms of Coded Factors:

$$\text{Oil yield(\%)} = +0.55 - 0.24 \times A[1] - 0.026 \times A[2] + 0.14 \times A[3] + 0.11 \times B[1] + 4.167E-003 \times B[2] - 0.025 \times A[1]B[1] + 0.011 \times A[2]B[1] + 0.015 \times A[3]B[1] - 9.167E-003 \times A[1]B[2] - 7.500E-003 \times A[2]B[2] + 5.833E-003 \times A[3]B[2]. \quad (4.1)$$

was obtained growing the individual effect of time and particle sizes as parameter and their interactions in the entire extraction process was found to be ineffective. Therefore, the final equation in terms of coded factor without the interaction effect is given by a first order regression equation:

Final Equation in Terms of Coded Factors:

$$\text{Oil yild} = 0.55 - 0.24 \times A[1] - 0.026 \times A[2] + 0.14 \times A[3] + 0.11 \times B[1] + 4.167E-003 \times B[2] \quad (4.2)$$

Where, A[1]= the difference of time level-1 from the over all average.

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

A[3]= the difference of time level-3 from the over all average.

B[1]= the difference of particle size level-1 from the over all average and

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

The coefficients of A(1),A(2) were negative but that of A(3),B(1) and B(2) are positive.

◆ Diagnostics

The following figure 4.4 shows the relation between the actual value of the experiment and the value predicted by the model equation developed by the Design Expert software.

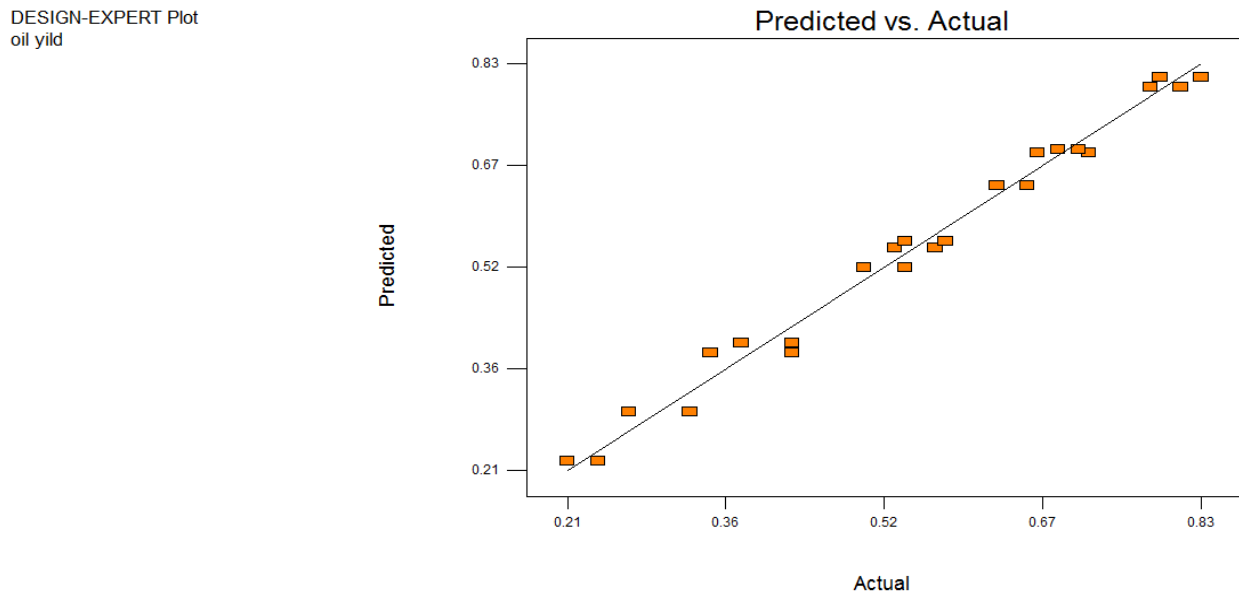


Figure 4.4: Predicted vs. actual value of yield for Steam distillation

The residue for equation (4.2) that will describe the difference between the actual values of the model and the predicted one was shown in the table below.

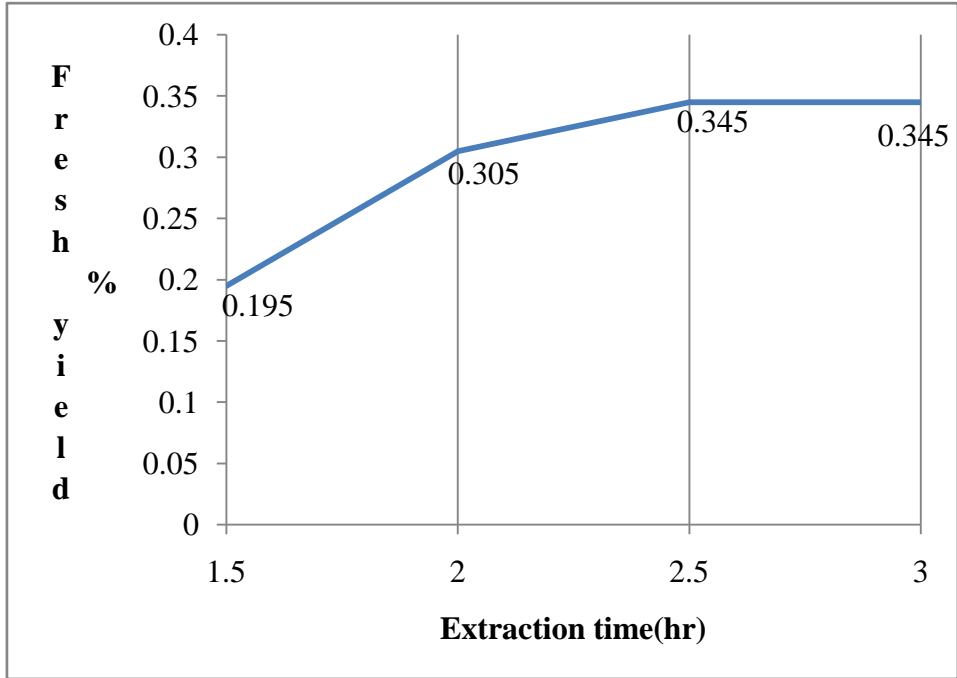
Table 4.8 Difference between the actual (experimental) value and predicted value

Standard Order	Actual Value	Predicted Value	Residual	Standard Order	Actual Value	Predicted Value	Residual
1	0.43	0.39	0.04	13	0.72	0.695	0.025
2	0.35	0.39	-0.04	14	0.67	0.695	-0.025
3	0.66	0.645	0.015	15	0.69	0.7	-0.01
4	0.63	0.645	-0.015	16	0.71	0.7	0.01
5	0.79	0.81	-0.02	17	0.21	0.225	-0.015
6	0.83	0.81	0.02	18	0.24	0.225	0.015
7	0.81	0.795	0.015	19	0.38	0.405	-0.025
8	0.78	0.795	-0.015	20	0.43	0.405	0.025
9	0.33	0.3	0.03	21	0.57	0.55	0.02
10	0.27	0.3	-0.03	22	0.53	0.55	-0.02
11	0.54	0.52	0.02	23	0.54	0.56	-0.02
12	0.5	0.52	-0.02	24	0.58	0.56	0.02

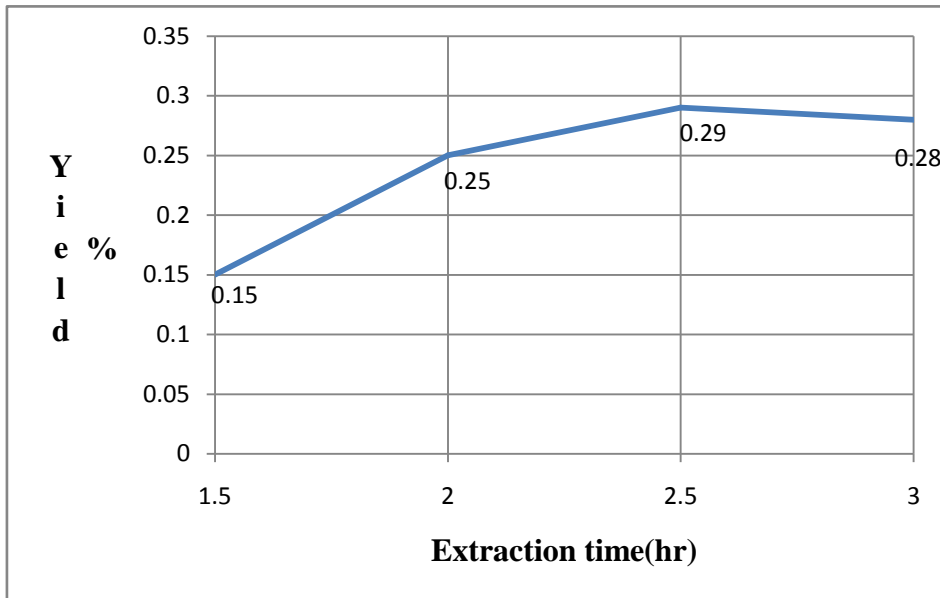
4.1.1.3.2 Effect of process parameters in percentage essential oil yield of fresh lemongrass

◆ Effect of extraction time on percent yield of oil

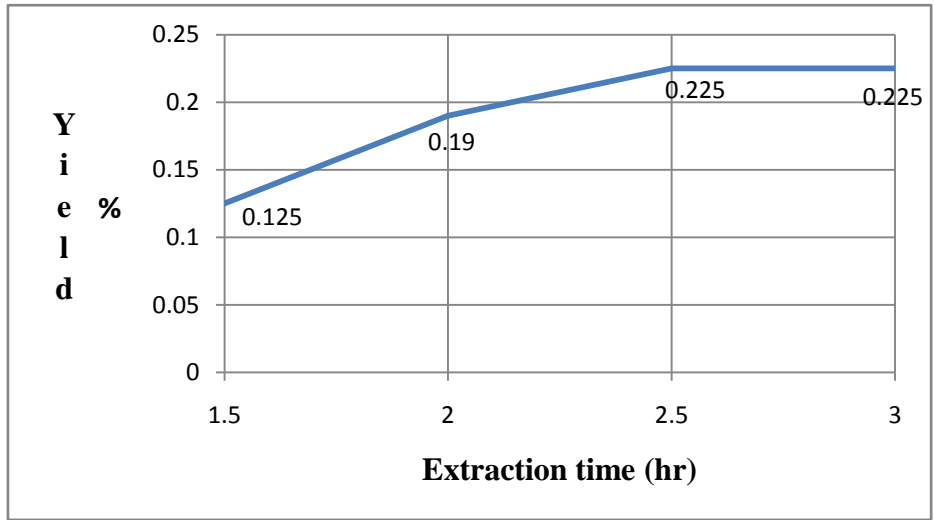
Percent yield of lemongrass essential oil can be affected by extraction time and particle size. Extraction time plays a great role on the percentage yield of lemongrass oil using steam distillation. Figures 4.5 (a), (b), and (c) show that as the contact time increases the oil yield also increases this continues till transfer of oil from the lemongrass leave when the maximum amount of extractable oil is obtained, the oil yield level remains invariable even by extending the reaction time. So that in the steam extraction the maximum oil yield could be finding at an extraction time of 2.5 hours and above. As shown graph of Figure 4.5, extracting the oil beyond two and half hours is wasting time. The extraction rate is fast at the beginning of the extraction but gets slow gradually. The reason is that when the chopped lemongrass is exposed to the steam, the free oil on the surface of leave is diffused into the vapor.



(d)



(e)



(f)

Figure 4.5: The effect of time on lemongrass oil yield at particle size (d) 8-15mm, (e) 15-25mm and (f) 25-35mm

◆ **Effect of particle size on percent yield of oil**

Particle size plays a great role on the yield of lemongrass oil. Smaller the size gives high yield while samples with large particle size deliver low yield. The principle is the same as dry lemongrass extraction as it was explained in the above.

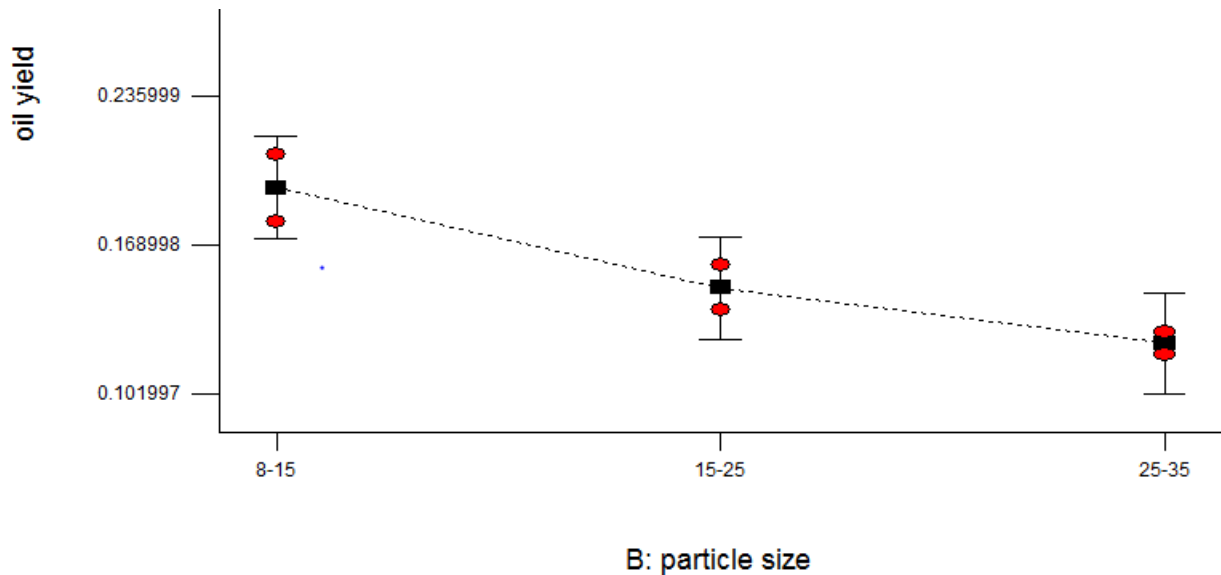


Figure 4.6: The effect of particle size on lemongrass oil yield

◆ **Regression model equation**

The following table shows analysis of variance (ANOVA), obtained from design expert soft

ware which tell us the significance of different factors.

Table 4.9 Analysis of variance (ANOVA) for a response of percentage oil yield

Source	Sum of square	Degree of freedom	Mean square	F value	P value Prob >F
Model	0.11	11	0.010	23.08	< 0.0001
A-Extraction time	0.066	3	0.022	49.40	< 0.0001
B-Particle size	0.045	2	0.023	50.66	< 0.0001
AB	1.958E-003	6	3.264E-004	0.73	0.6334
Pure Error	5.350E-003	12	4.458E-004		
Cor Total	0.12	23			

The Model F-value of 23.08 implies the model is significant. 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. The P-value of AB (interaction factors) is 0.6334 > P-value thus, the interactions of size and time are not significant in the model terms.

Table 4.10. Design summary of essential oil of fresh lemongrass (citratus)

Design summary							
Study type	Factorial		Experiment :24				
Initial design	Full factorial		Blocks	No blocks			
Center point zero(0)							
Design model 2FI							
Response	name	units	obs	minimum	maximum	trans	model
Y	Oil yield	%	24	0.12	0.37	none	2FI
Factor	name	units	type	Low actual	High actual		
A	Extraction time	minute	categorical	90	180	level	4
B	Particle size	mm	categorical	8-15	25-35	level	3

Table 4.11 Constraints and solution of essential oil yield for fresh lemongrass (citratu)

constraints						
Name	Goal	Lower limit	Upper limit	Lower weight	Upper weight	importance
Extraction time	Is in range	90	180	1	1	3
Particle size	Is in range	8-15	25-35	1	1	3
Oil yield	maximize	0.12	0.37	1	1	3
solution						
number	Extraction time	Particle size	Oil yield	desirability		
1	<u>150</u>	<u>8-15</u>	<u>0.345</u>	<u>0.900</u>	<u>selected</u>	
2	180	8-15	0.345	0.900		
3	120	8-15	0.305	0.740		
4	150	15-25	0.29	0.680		
5	180	15-25	0.28	0.640		
6	120	15-25	0.25	0.520		
7	150	25-35	0.225	0.420		
8	180	25-35	0.225	0.420		
9	90	8-15	0.195	0.300		
10	120	25-35	0.19	0.280		
11	90	15-25	0.15	0.120		
12	90	25-35	0.125	0.020		
12 Solution found						

◆ **Interaction effect between two factors.**

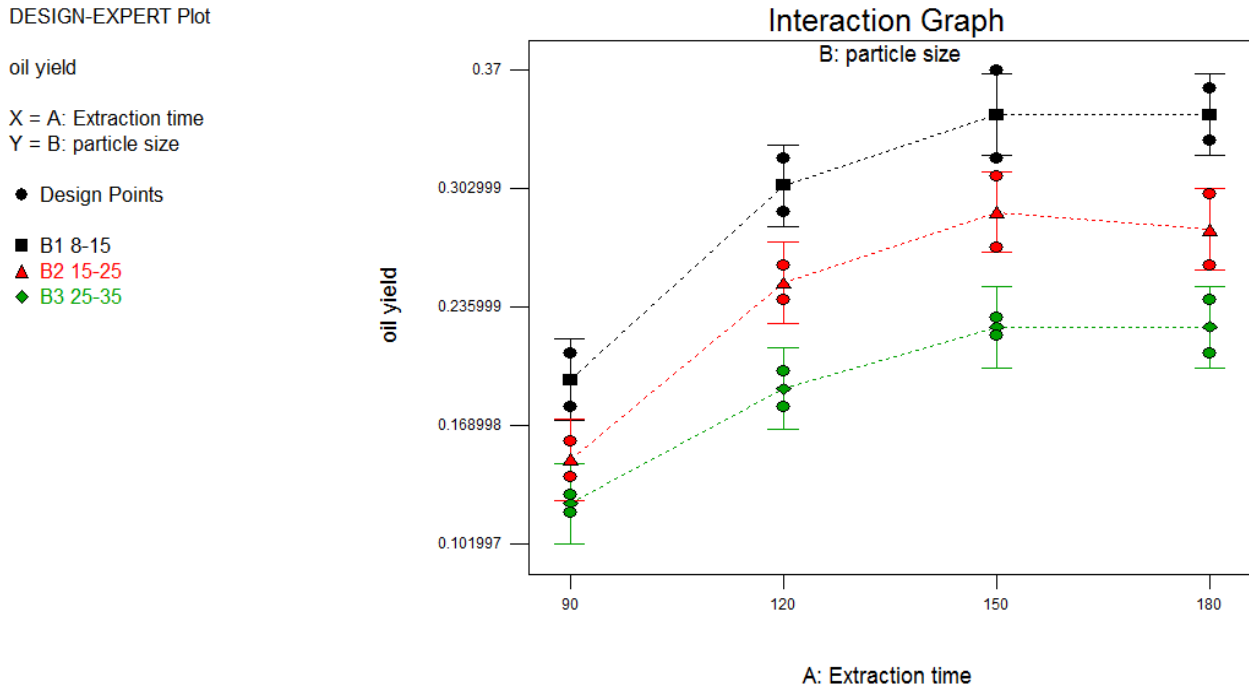


Figure 4.7 interaction effect of particle size and extraction time on oil yield

From design expert soft ware the output of interaction effect between particle size and extraction time.

Where: B1, B2 and B3 are codes for particle sizes ranges from 8-15mm, 15-25mm, and 25-35mm, respectively. A1, A2, A3 and A4 are codes for extraction time (hours) one and half, two, two and half and three hours, respectively. Design points are points on the graph which helps to develop mathematical model of the predicted response based on these points. Design -expert was applied to analyze results on the extraction process and a first order regression equation with the interaction terms of the form:

Final Equation in Terms of Coded Factors:

$$\text{Oil yield} = +0.24 - 0.087 * A[1] + 4.583E-003 * A[2] + 0.043 * A[3] + 0.054 * B[1] - 1.250E-003 * B[2] - 0.015 * A[1]B[1] + 2.917E-003 * A[2]B[1] + 4.583E-003 * A[3]B[1] - 5.417E-00 * A[1]B[2] + 2.917E-003 * A[2]B[2] + 4.583E-003 * A[3]B[2] \quad (4.3)$$

was obtained growing the individual effect of time and particle sizes as parameter and their interactions in the entire extraction process was found to be ineffective. Therefore, the final

equation in terms of coded factor without the interaction effect is given by a first order regression equation.

Final Equation in Terms of Coded Factors:

$$\text{Oilyield} = 0.24 - 0.087 * A[1] + 4.583E-003 * A[2] + 0.043 * A[3] + 0.054 * B[1] - 1.250E-003 * B_2 \quad [4.4]$$

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

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

A[3]= the difference of time level-3 from the over all average.

B[1]= the difference of particle size level-1 from the over all average. and

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

Diagnostics.

The following figure 4.8 shows the relation between the actual value of the experiment and the value predicted by the model equation developed by the Design Expert software.

DESIGN-EXPERT Plot
oil yield

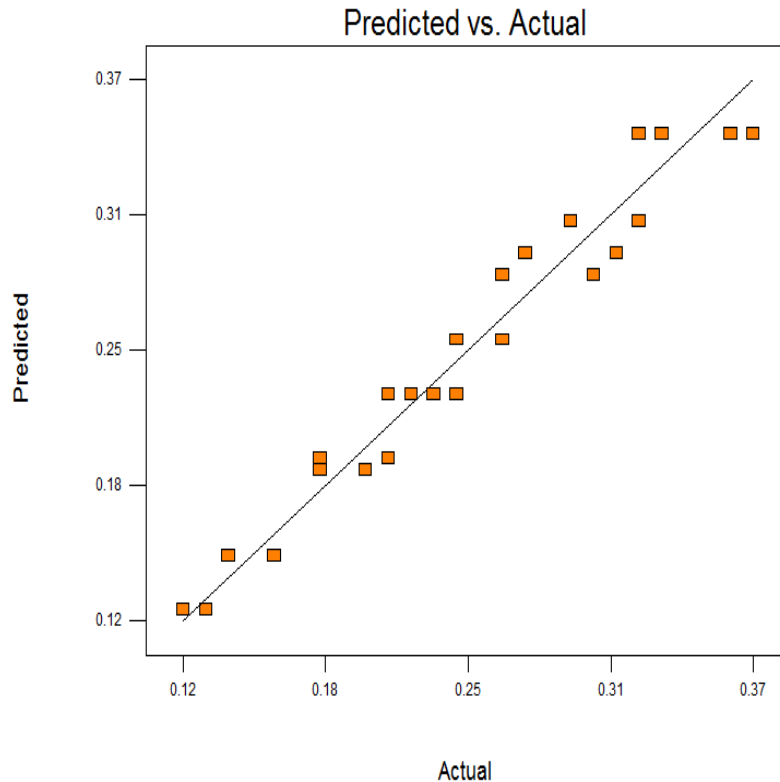


Figure 4.8: Predicted vs. actual value of yield for Steam distillation.

Table4.12: Difference between the actual (experimental) value and predicted value

Standard Order	Actual Value	Predicted Value	Residual	Standard Order	Actual Value	Predicted Value	Residual
1	0.21	0.195	0.015	13	0.27	0.29	-0.02
2	0.18	0.195	-0.015	14	0.31	0.29	0.02
3	0.29	0.305	-0.015	15	0.26	0.28	-0.02
4	0.32	0.305	0.015	16	0.3	0.28	0.02
5	0.32	0.345	-0.025	17	0.12	0.125	-0.005
6	0.37	0.345	0.025	18	0.13	0.125	0.005
7	0.33	0.345	-0.015	19	0.18	0.19	-0.01
8	0.36	0.345	0.015	20	0.2	0.19	0.01
9	0.14	0.15	-0.01	21	0.22	0.225	-0.005
10	0.16	0.15	0.01	22	0.23	0.225	0.005
11	0.24	0.25	-0.01	23	0.21	0.225	-0.015
12	0.26	0.25	0.01	24	0.24	0.225	0.015

4.1.2 Characterization of essential oil extracted from lemongrass

4.1.2.1 Determination of some physical properties of the lemongrass oil

4.1.2.1.1 Moisture and volatile matter of essential oil

The moisture and volatile matter of the oil determined by oven method.5gm of oil was taken and put in oven and the weight was recorded at 2hr and 4hr .The result obtained is summarized in the table below.

Table 4.13 moisture and volatility of essential oil

Time (hr)	0	2	4	dry	0	2	4	fresh
Weight(5gm)	5	4.9899	4.9899	0.2015%	5	4.991	4.99	0.2%

From equation (3.1), moisture and volatility of essential oil was determined for both case.Then $5-4.9899 = 0.0101\text{gm}$, $5-4.99 = 0.01$ for dry and fresh respectively.

W is weight in gm of oil taken for the test=5.0gm, Substituting the above values in the equation

$$\text{Moisture and volatile matter} = \frac{\text{Initial} - \text{final weight}}{\text{Initial weight}} = 0.2015\% \text{ for dry and for fresh} = 0.2\%$$

4.1.2.1.2 Kinematic viscosity dry and fresh lemongrass extracted oil

- Kinematic viscosity dry lemongrass extracted oil

Dynamic viscosity of oil which was read from vibro viscometer was 1.72mpa.s at temperature 20⁰c, substituting the dynamic viscosity oil =1.72mpa.s=1.72× 10⁻³ kg/m.s and density of oil =881.25 kg/m³

$$\text{Kinematic viscosity} = \frac{1.72 \times 10^{-3} \frac{kg}{m.s}}{881.25 \frac{kg}{m^3}} = 1.95173 \times 10^{-6} m^2/s, \text{ Therefore the kinematic viscosity of}$$

the lemongrass oil was 1.95173× 10⁻⁶m²/s

- Kinematic viscosity fresh lemongrass extracted oil

Dynamic viscosity of oil which was read from vibro viscometer was 1.1mpa.s at temperature 20⁰C, then Kinematic viscosity was calculated by substituting the dynamic viscosity oil =1.1mpa.s=1.1× 10⁻³ kg/m.s and density of oil =871.25 kg/m³ in equation (qa.4).

$$\text{Kinematic viscosity} = \frac{1.1 \times 10^{-3} \frac{kg}{m.s}}{871.25 \frac{kg}{m^3}} = 1.26255 \times 10^{-6} m^2/s, \text{ Therefore the kinematic viscosity of}$$

extracted oil of fresh lemongrass was 1.26255× 10⁻⁶m²/s

4.1.2.1.3 PH value of lemongrass oil

The PH value of oil was determined by PH electrode as measuring experiment

Table 4.14 The PH value of lemongrass oil was triplicated and the results obtained

Type of product	Run			Average
	1	2	3	
PH value of dry lemongrass extracted oil	3.98	4.200	4.21	4.13
PH value of fresh lemongrass extracted oil	4.24	4.25	4.21	4.23

Therefore the PH value of lemongrass oil was for dry and fresh were 4.13, 4.23 respectively. In the preparation of perfume the PH value is in the range of 3.5-6.5 (Mueller *et al*,2000).The obtained PH value of lemongrass oil is in the range of commercially recommended.

4.1.2.1.4 Specific gravity

Density bottle method was used to determine the specific gravity of oil as the detail experimental procedure were stated .From equation (3.5)

X_2 is weight in gm of density bottle with oil at $20^0\text{c} = 41.1051\text{gm}$

X_1 is weight in gm of density bottle at $20^0\text{c} = 18.7266\text{gm}$

X_3 is weight in gm of density bottle with water at $20^0\text{c} = 43.9690\text{gm}$

Substituting the above values in the equation (3.5)

Specific gravity = $\frac{X_2 - X_1}{X_3 - X_1} = \frac{41.1051 - 18.7266}{43.9690 - 18.7266} = 0.88125$, for dried lemongrass extracted oil

Where X_2 = Weight of pycnometer with sample, X_1 = Weight of pycnometer, X_3 = Weight of pycnometer with water

Density of lemongrass oil = Specific gravity \times density of water = $0.88125 \times 1 \frac{\text{gm}}{\text{ml}}$

Density of lemongrass oil = $0.88125 \frac{\text{gm}}{\text{ml}}$, for dried lemongrass extracted.

Density of lemongrass oil = $0.8715 \frac{\text{gm}}{\text{ml}}$, for fresh lemongrass extracted. The value of lemongrass oil for both around the recommended commercially.

4.1.2.1.5 Refractive index

The value refractive index of oil was determined in Addis Ababa University in physics department using spectroscope and Brewster's angle used to determine the refractive index.

The lower the refractive index is the higher the quality of essential oil (Anhwange *et al*, 2010).

4.1.2.1.6 Optical rotation

Optical rotation was determined in College of natural science of Addis Ababa University in physics department using prism spectrometer.

Table 4.15: Summery of measured value of physical property of extracted essential oil

Physical property	Value for dry lemongrass oil	Value for fresh lemongrass oil	Unit
Refractive Index At 25^0c	1.413	1.421	-
Optical rotation At 25^0c	0^0	0^0	Degree
Flash point	94	95	C^0
Density	0.88125	0.8715	g/ml

Table 4.15 continued

Boiling point at room temp	224	224	C ⁰
Odour	Strong like lemon	Like lemon	-
Colour	Light yellow	Light yellow	-
Specific gravity	0.88125	0.8715	-
Viscosity(dynamic)	1.7	1.1	mpa.s
PH	4.13	4.23	-

4.1.2.2 Determination of chemical properties of lemongrass essential oil

The quality of lemongrass essential oil could be determined based on the values of FT-IR, UV visible spectrometer, GC-MS, Saponification, aldehyde and acid value as a result of extraction temperature 94.5c⁰ at dry and fresh raw material conditions.

Different biochemical tests such as Saponification Value (SV), aldehyde value, acid Value (AV) and ester were determined as per standard methods and protocols prescribed by (Pearson, 1962; Thimmaiah, 2006).

4.1.2.2.1 Saponification value of the oil

The saponification value is the number that expresses in milligrams of the quantity of potassium hydroxide required to neutralize the free acids and to saponify the esters present in 1 g of the substance. The test method to determine saponification value of oil is standardized as in ASTM D1959-97 Standard Test Method for saponification value of oils and fatty Acids. Preparation of 0.5NKOH-Ethanol (Ethanolic potassium hydroxide): to prepare 0.5N of ethanol KOH solution, 14.027gm of KOH dissolved in 500ml of ethanol. Preparation of 0.5N of HCl: To prepare this solution, 43.7ml of HCl poured on 1000ml of distilled water. After gentle heated the oil with Ethanolic-KOH for 30min. Immediately five drops of phenolphthalein added and the amount of HCl require saponifying the oil and form a color change for steam extracts are 12, and 14ml for dry and fresh lemongrass respectively for the following corresponding temperatures 94.5. Thus, determination of saponification value found using equation (3.8) and the results obtained was shown in table 4.16:

Table4.16: Saponification value of lemongrass oil

Raw material condition	The volume of HCl for the Blank (ml)	The volume of HCl for The sample (ml)	Mass of sample (gm)	SV
Dry	25	12	2	168.3
Fresh	23	14	2	126.22

Hence, the saponification value of lemongrass essential oil for both conditions 168.3, 126.22mg KOH/gm for dry, fresh respectively. The value is in the range of recommended (28.06-547.17). The greater saponification values implies greater proportion of fatty acid of low molecular weight. The value obtained for both case favorable for perfume.

4.1.2.2.2 Acid value of the oil

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). Titration method was used to determine the acid value. 25ml of toluene and 25ml of ethanol was mixed in a 250ml beaker. The resulting mixture was added to 2g of oil in a 250ml conical flask and few drops of phenolphthalein were added to the mixture. The mixture was titrated with 0.1M KOH to the end point with consistent shaking for which a dark pink colour was observed and the volume 1.3 and 1.8 of 0.1M KOH (V_0) was noted for extracted oil from dry and fresh lemongrass.

Table 4.17 Acid value of essential oil lemongrass

Raw material condition	The volume of KOH for The sample (ml)	Mass of sample (gm)	Acid
Dry	1.3	2	18.23
Fresh	1.8	2	25.2

4.1.2.2.3 Determination of aldehyde content

Into a 150 cc cassia flasks, having a thin neck graduated in 0.1cc divisions, 75 cc of a freshly prepared, saturated, aqueous solution of 1 gm of sodium bisulfate was poured into graduated cylinder. Then 10 cc of the oil was added into the flask. Then the value of aldehyde content calculated by means of the following formula as it stated in equation (3.10)

Percentage of aldehyde = $10(10 - \text{No .of cc of unreacted oil})$

Where, Unreacted essential oil = 2.6, for both case the value the same.

Percentage of aldehyde = $10(10-2.6) = 74$, the value of aldehyde which was obtained in the range of the recommended (65-85%).

4.1.2.2.4 Ester value, content of esters and combined alcohols

The determination of the ester content is of great importance in the evaluation of many essential oils. The value of ester can be calculated as follow

Ester value = Saponification value – acid value

Ester value = $168.3-18.23 = 150.07$, for dry lemongrass extracted oil, Ester value = $126.22-25.2 = 101.02$, for fresh lemongrass extracted oil.

4.1.2.2.5 Determination of the functional groups present using FT-IR

The functional groups present in the essential oil was determined 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. In the FT-IR spectrum of *lemongrass leave* essential oil the absorption band or frequency as mentioned in figure 4.9 below, the main component of the *C. citrates* essential oil is citral. In the IR spectrum of *C. citratus*, the functional groups of citral was observed in the vibrations at 2968 cm^{-1} , a predominant asymmetric stretching of $-\text{CH}_3$ is observed corresponding to an alkyl saturated aliphatic group and at 2915 and 2857 cm^{-1} , symmetric and asymmetric stretching of $-\text{CH}_2$ are observed. The intense band observed at 1671 cm^{-1} is due to vibrations of $\text{C}=\text{C}$ (cis and trans), confirming the presence of conjugated double bonds ($\text{C} = \text{C}-\text{CHO}$) in citral which are common in acyclic monoterpenes. The peak at 1632 cm^{-1} indicates stretching of $\text{C}=\text{O}$ of the aldehyde group. At the 1442 cm^{-1} peak, bending of the $-\text{CH}_2$ group is observed. At 1377 cm^{-1} bending of $-\text{CH}_3$ group is observed. From 1194 to 1120 cm^{-1} , stretching of $-\text{C}-\text{O}$ and vibrations of the $-\text{CH}$ skeleton are observed. The IR spectrum of *C. citratus* essential oil is consistent with data obtained from gas chromatograph coupled to mass spectroscopy

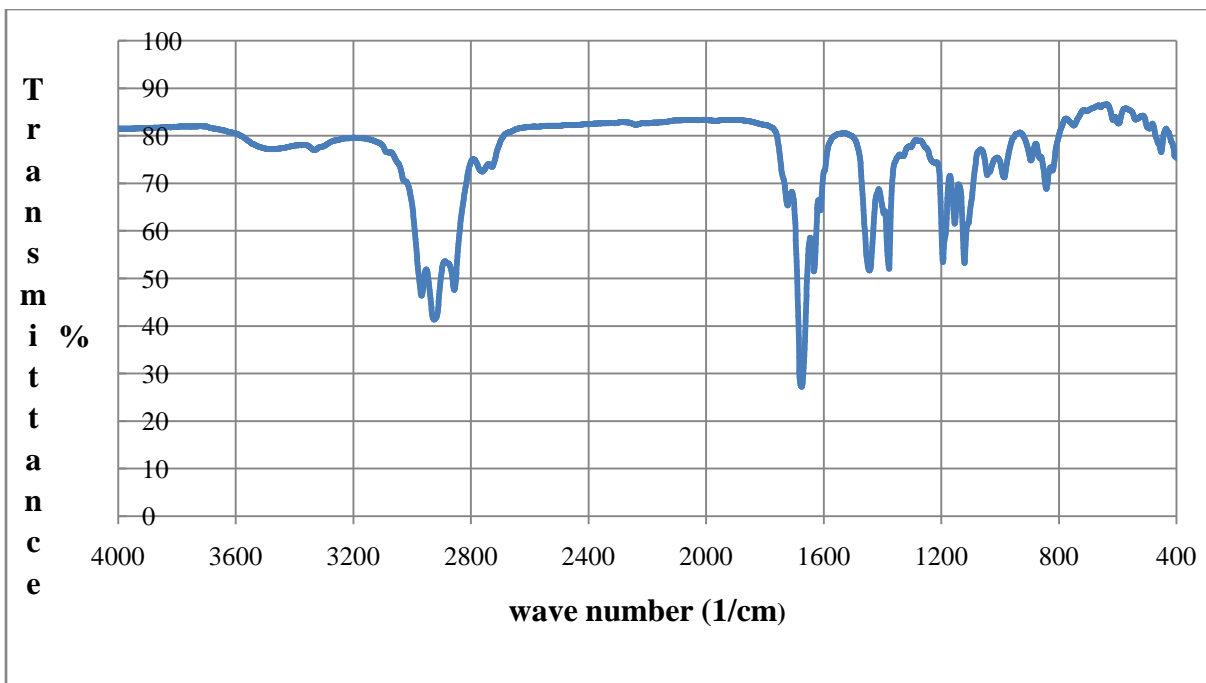


Figure 4.9 FT-IR profile analysis of essential oil of lemongrass

4.1.2.2.6 Gas Chromatography-Mass Spectroscopy

A total of twenty components, with different retention times, were eluted from the GC column as indicated by the chromatogram and were further analyzed with an electron impact MS voyager detector. Identification of constituents was done on the basis of their retention time and mass spectra library search. The mass spectrographs of the identified constituents are given in table 4.18 below. The relative amount of individual components was calculated based on GC peak areas. Comparison of the GC-MS spectrograph obtained with the instruments data bank together with NIST MS data demo version revealed that the essential oil of *C. citratus* contained a mixture of terpenes that eluted at different retention times depending on the boiling point of the eluted component. The GC chromatogram obtained revealed a high concentration of citral indicated by presence of two large peaks which eluted at 18.835 and 20.182 minutes with peak areas of 9870795776 and 3720576000 respectively (figure 4.10). The presence of these two peaks may be due to the two isomers of citral that is geranial and neral, which may be difficult to distinguish with GC-MS.

File :D:\MassHunter\GCMS\1\5977\MAR 09\03801038.D
Operator : Estif
Acquired : 11 Mar 2016 14:13 using AcqMethod Essential oil.M
Instrument : AAU
Sample Name: LEMMON.G
Misc Info :
Vial Number: 38

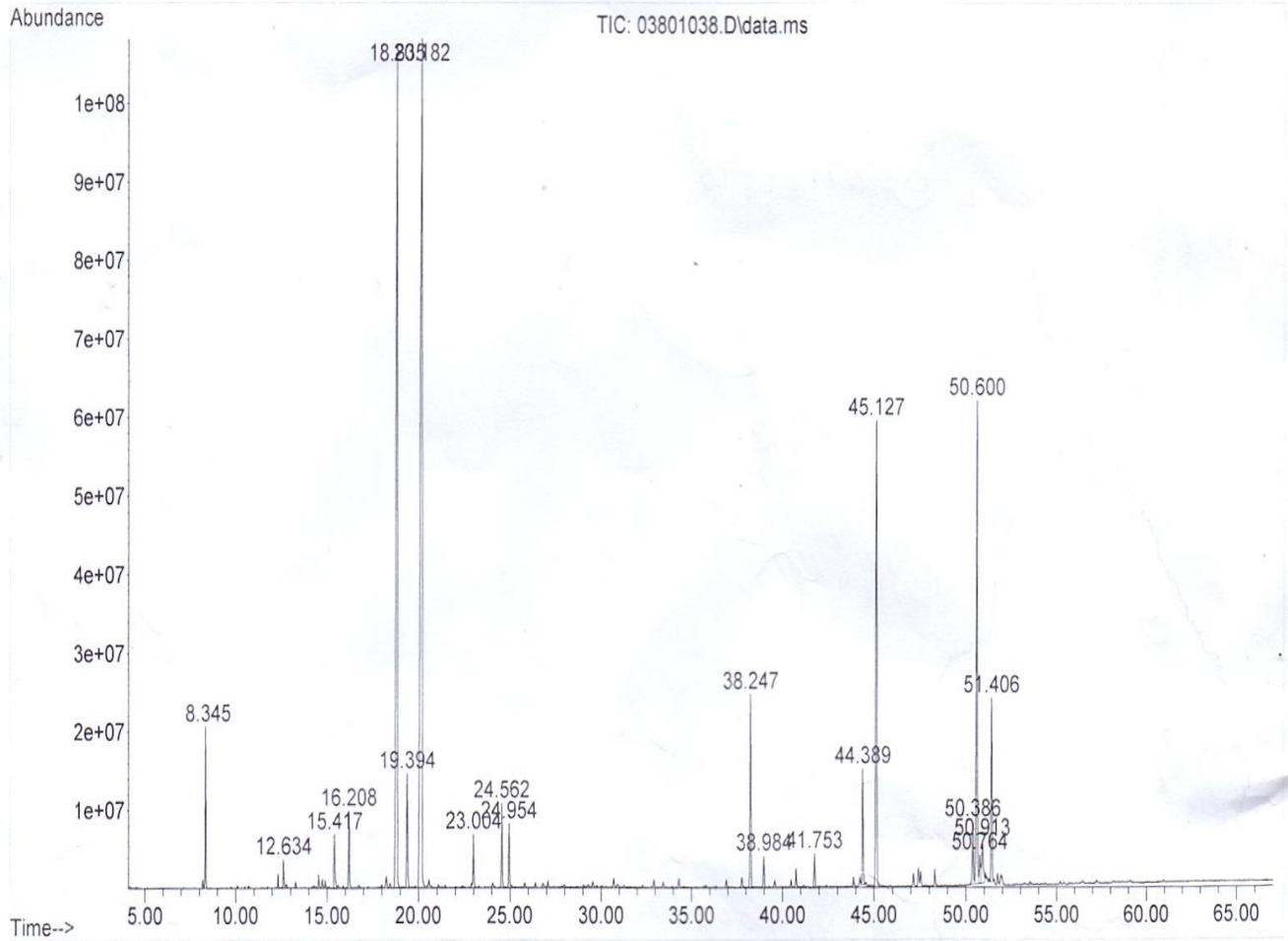


Figure.4.10 Result profile from GC-MS.

Table 4.18 Chemical composition of lemongrass (citrus) oil.

Compounds	Library Match Quality (%)	Retention time (min)	Area (%)
Beta-myrcene	96	8.345	2.24
Linalool	97	12.634	0.48
Isoneral	98	15.417	0.99
Isogeranial	98	16.208	1.4
Geraniol	95	19.394	2.4
Citral	95	20.182	30.43
Geranyl acetate	91	24.954	1.11
Methyl tetradecanoate	99	38.247	3.66
(6R,7R)-Bisabolone	98	38.984	0.6
Pentadecanoic acid, methyl ester	99	41.753	0.62
9-hexadecenoic acid, methyl ester(z)-	99	44.389	2.26
Hexadecanoic acid, methyl ester	98	45.127	10.28
9,12-octadecadienoic acid(z,z)-,methyl ester	99	50.386	1.36
Cis-13-octadecenoic acid, methyl ester	99	50.600	10.47
11-octadecenoic acid,methyl ester	99	50.764	0.71
Cis-13-octadecenoic acid,methyl ester	99	50.913	1.38
Methyl stearate	99	51.406	3.7

From the above it can be concluded that the main component of essential oil of lemongrass was citral that consisted aroma that used for perfume development. Both Gas chromatograph-mass spectroscopy and FT-IR indicate the main component of lemongrass essential oil which was citral. Here citral was the main component which found in the lemongrass oil as percentage of 30.43% .The percentage of citral can be increase by using fractional distillation to increase the aroma of it. But other components have great advantage for our body when we sprayed upon our flesh due to it has antiviral, anti fungal and anti-bacterial properties of it as literature cited.

4.2 Result of perfume proportion

Perfume oils usually contain tens to hundreds of ingredients and these are typically organized in a perfume for the specific role they play. Here only one ingredient used which is lemongrass essential oil instead of ten to hundreds ingredient what was used to develop perfume.

Table 4.19 perfume proportion from lemongrass essential oil

Type of perfume	Fragrance	Carrier solvent	Fixative	Diluent
Esprit de parfum	Lemongrass oil	Ethanol (96%)	Glycerin	D.water
Proportion of the perfume				
	20% lemongrass oil	75% ethanol	0.33% of glycerin	4.67%

These ingredients grouped into Primary scents (Heart): which consisted of one main ingredient such as lemongrass essential oil. Alternatively, multiple ingredients can be used together to create an abstract primary scent that does not bear a resemblance to a natural ingredient. For instance, jasmine and rose scents are commonly blended for abstract floral fragrances. But, here only lemongrass essential oil used to develop perfume. Blenders: are a large group of ingredients that smooth out the transitions of a perfume between different layers or bases. These blending ingredients are linalool which existed in the oil by itself. Fixatives: They used to support the primary scent by bolstering it in addition to elongate its aroma. Using the above proportion, perfume was developed based on the procedure which was stated in chapter three. After it was developed, sensor analysis carried out.

4.3 Result of the Sensory Evaluation of perfume

Sensory quality is the ultimate measure of product quality and success. Sensory analysis comprises a variety of powerful and sensitive tools to measure human responses to products (perfume). Appropriate application of these tests enables specific product and consumer insights and interpretation of volatile compound analyses to aroma perception. Results of sensory evaluation in terms of sensory attributes such as colour, aroma and overall acceptability are presented in table 4.20 below.

Table 4.20 Sensory analysis results of perfume in terms of aroma, color and acceptability

Each attributes	scored of each attributes based on ten panelist rating								
	1	2	3	4	5	6	7	8	9
color								2	8
acceptability							7	2	1
aroma							1	8	1
Each attributes	weight in percent for each attributes								
	1	2	3	4	5	6	7	8	9
color								20	80
acceptability							70	20	10
aroma							10	80	10

Hedonic scales: 1 = dislike extremely, 2 = dislike very much, 3 = dislike moderately, 4 = dislike slightly, 5 = neither like nor dislike, 6 = like slightly, 7 = like moderately, 8 = like very much, 9 = like extremely.

According to the sensory analysis results in Table 4.20 showed that, about 80% of the panelists preferred like very much of aroma sample of the perfume at concentration of 20% essential oil developed. The results of sensory evaluation showed that perfume can be developed from lemongrass essential oil.

5. Determination of techno-economy feasibility of perfume production

5.1 Material, energy balance and cost estimation

5.1.1 Material and energy balance

5.1.1.1 Material balance

Assumption: lemongrass collected on the harvesting period based on literature cited.

Note: All material balances had performed based on the experimental work in the laboratory.

Material balances are nothing more than the application of the conservation law for mass. According to Treybal (1981), matter is neither created nor destroyed.

$$\text{Accumulation} = \text{Output} + \text{Consumption} - \text{Input} - \text{Generation} \quad (5.1)$$

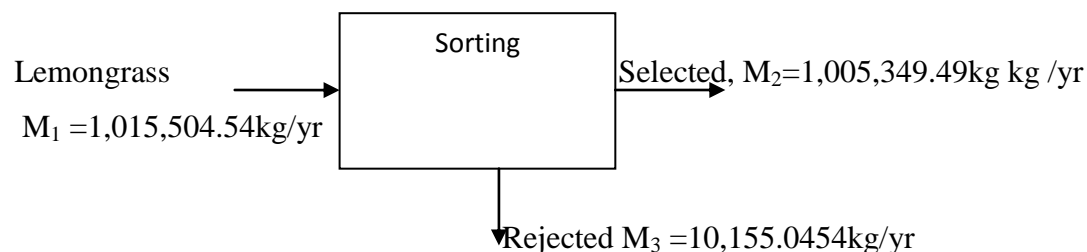
Since there is no reaction, the generation and consumption terms are zero, no accumulation

Total production feed for plant can be planted because we do have available climate condition and land for production of lemongrass but today the amount of lemongrass could not know how much we have. Even if it was not known but the people of Ethiopian have been using this herbal grass for anniversary ceremony to decorate the house and church.

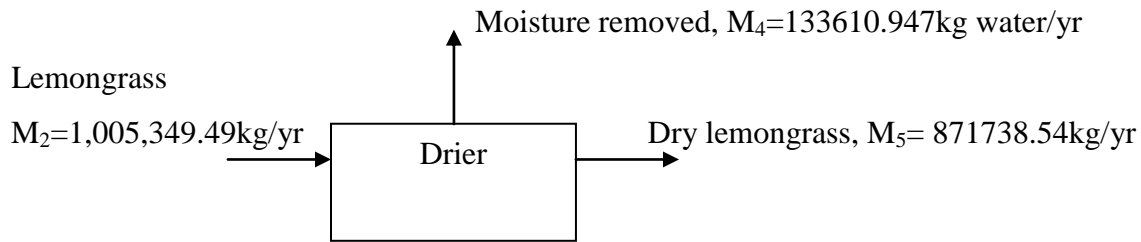
Only by 2015 Ethiopia imports 98,931 kg perfume from different countries to satisfy the demand of the customers (Ethiopian revenue and custom authority, 2015). So that perfume can be developed by substituted our indigenous raw material that used for perfume formulation. From this assume 40% for women. Assume if 35% will be substituted. As we know the concentration of fragrance perfume for women from 15-30%. Assume 20% concentrated. Then to produce 98,931kg of perfume, it needs 19,786.2 kg of fragrance required. Now the amount of land required to produce this essential oil 1,413.3 hectare needed. So let substitute 35% from the total import. The required essential oil needed 6990.47kg kg and the amount of land would be 285.3 hectare.

5.1.1.1.1 Material balance for dry lemongrass

❖ Sorting, assume 1% dirty material removed

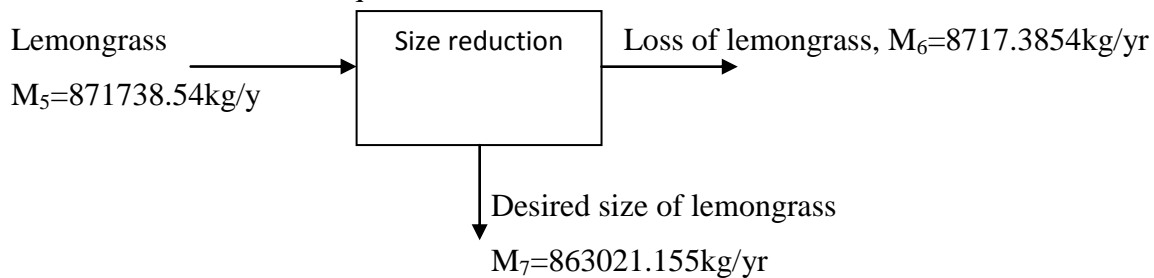


- ❖ Drying, from experiment 13.29% moisture removed from 1kg lemongrass



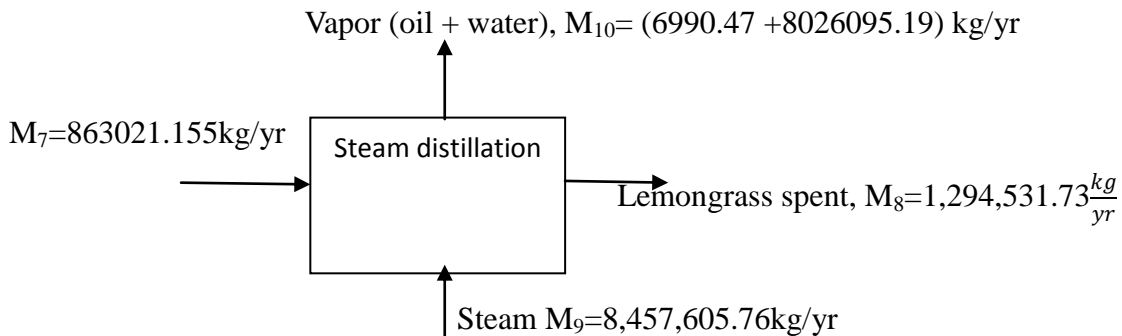
- ❖ Balance on size reduction Machine

Note: assuming there is 1% of lemongrass loss .Then, the amount of raw material left to the extraction vessel within required size.



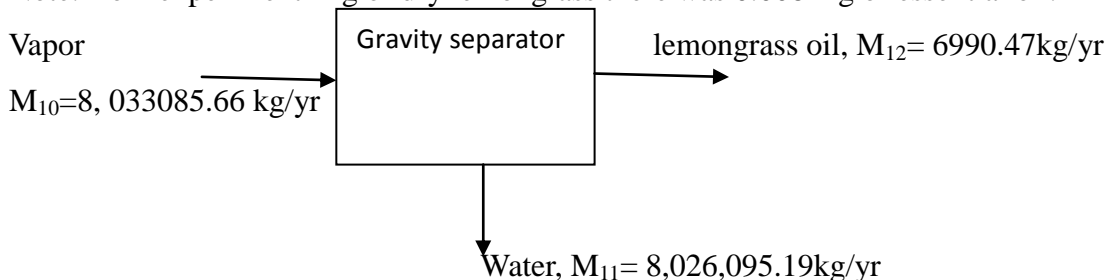
- ❖ Balance on still tank

From experiment to get 0.0081kg of essential oil, 9.3kg of water was distilled at 2.5hr after extraction the weight of spent lemongrass for 1kg was 1.42kg. Assume no loss of water from still tank.



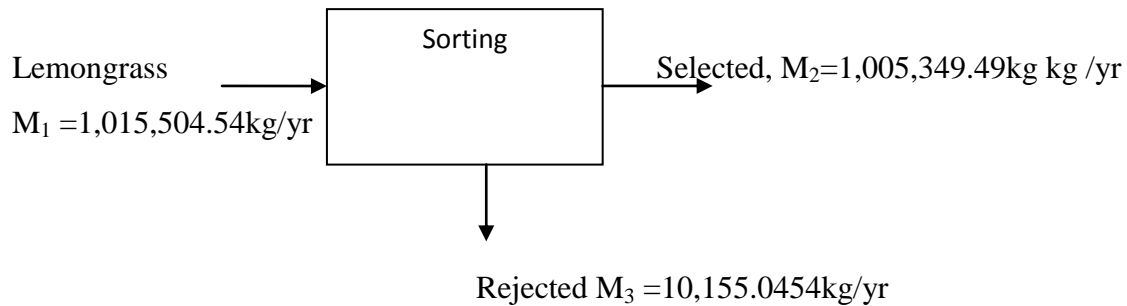
- ❖ Balance on gravity separator

Note: from experiment 1kg of dry lemongrass there was 0.0081kg of essential oil.



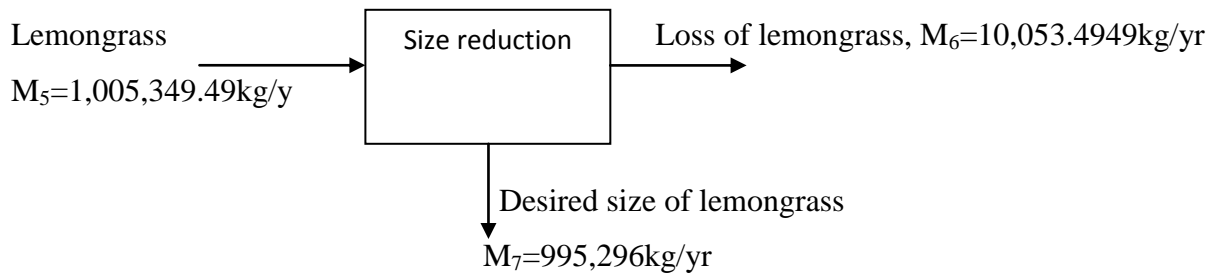
5.1.1.1.2 Material balance for fresh lemongrass

- ❖ Sorting, assume 1% dirty material removed



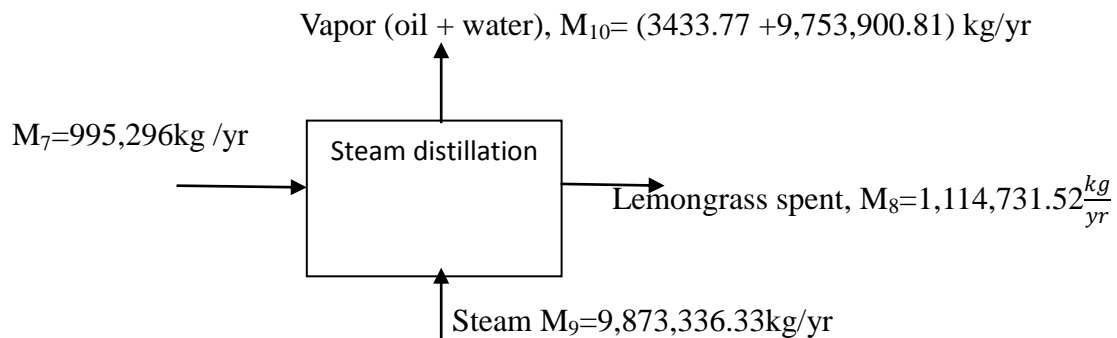
- ❖ Balance on size reduction Machine

Note: assuming there is 1% of lemongrass loss .Then, the amount of raw material left to the extraction vessel within required size.



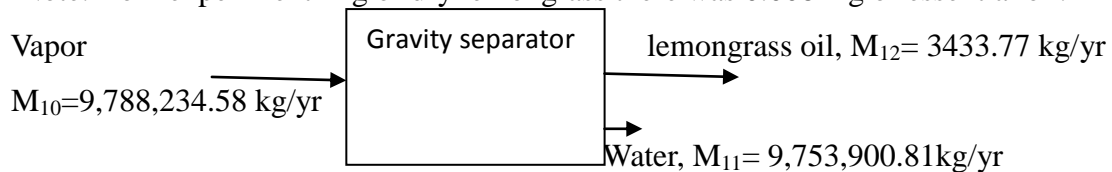
- ❖ Balance on still tank

From experiment to get 0.00345kg of essential oil, 9.8kg of water was distilled at 2.5hr
After extraction the weight of spent lemongrass for 1kg was 1.12kg. Assume no loss steam



- ❖ Balance on gravity separator

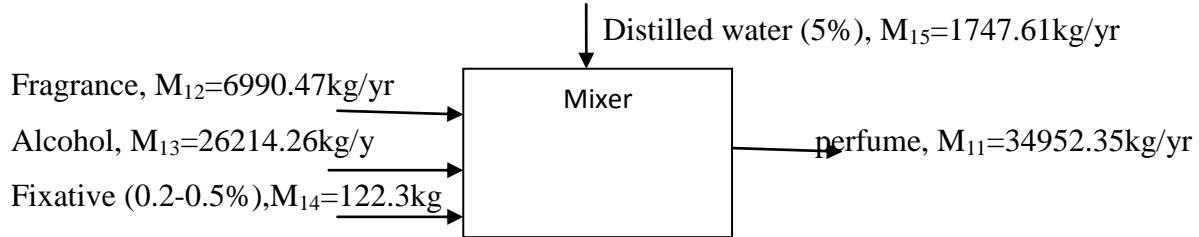
Note: from experiment 1kg of dry lemongrass there was 0.0081kg of essential oil.



5.1.1.1.3 Material balance in perfume formulation

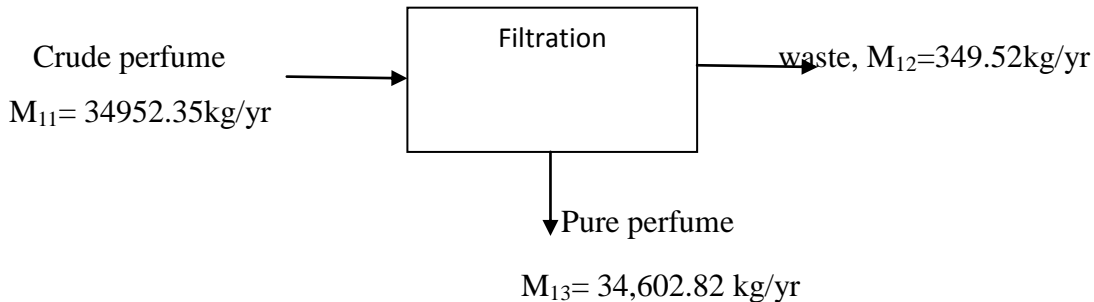
◆ Mixing machine

Note: let 20% concentrated of perfume and 75% of alcohol



◆ Filtration

Note: removal of undiluted ingredient 1%



5.1.1.2 Energy Balance on steam distillation for extraction of oil from lemongrass

5.1.1.2.1 Energy Balance on steam distillation for extraction of oil from dry lemongrass

This energy balance was specifically calculated only for the water in the System. It showed that heat was absorbed into the system when boiling water and later released when water turns to vapor. Equation 5.2 shows the formula to calculate heat absorbed by the system:

$$Q_A = mC_p\Delta T \quad (5.2)$$

Where, Q_A = heat absorbed by the system (kJ), m =mass of water (kg), C_p = heat capacity (kJ/kg.K), ΔT = temperature difference ($^{\circ}\text{C}$)

At the end of the experiment there was 9.3 liters of water distilled out with lemongrass oil, Initial temperature 25°C , and final temperature 94.5°C

$$Q_A = 9.3\text{kg} \times 4.184 \text{ (KJ/kg.K)} \times (367.5 - 298) \text{ K} = 2704.3284 \text{ KJ}$$

The heat released from the system, when water turns to vapor is illustrated in equation 5.3

$$Q_R = n \lambda \quad (5.3)$$

Where n =Number of moles of water (mol/g), $\lambda = 40.656 \times 10^3\text{J/mol}$

In this study, the experiment was carried out at atmospheric pressure (1atm). The heat capacity of steam at the constant pressure is $\frac{4.184\text{kJ}}{\text{kg.K}}$ (Perry and Green, 1997). The initial temperature of the system is 25°C (298K) and the final temperature is 94.5°C (367.5K).

$$Q_R = 9300\text{g} \times (1\text{mol} / 18\text{g}) \times 40.656\text{E3} (\text{J/mol}) = 21,005.6\text{KJ}$$

By using the data given, the total energy used by the system in this process, Q_s is as follows:

$$\begin{aligned} Q_S &= Q_A + Q_R \\ &= 2704.3284 \text{ KJ} + 21,005.6\text{KJ} = 23,709.9284\text{KJ} \end{aligned} \quad (5.4)$$

Where, Q_s = Energy used by the system in this process (kJ)

The total energy supplied into the system during the experiment, (kW.hr) is as follows:

$$\begin{aligned} Q_P &= \frac{1\text{KWH}}{3600\text{KJ}} \times Q_S \\ Q_P &= \frac{1\text{KWH}}{3600\text{KJ}} \times 23,709.9284\text{KJ} = 6.586\text{KWH} \end{aligned} \quad (5.5)$$

The total energy supplied by the heater, Q_H (KW.hr) can be calculated by multiplying the power supplied by the heater through the whole experiment, as in the equation below:

$$Q_H = 9000 \times \frac{J}{s} \times t \quad (5.6)$$

Where, t = Optimum operating time (hr). The optimum operating point was 2.5hr

$$Q_H = 9\text{KW} \times 2.5\text{hr} = 22.5\text{KWH}$$

The equipment efficiency (heating mantle) ϵ can be calculated as follows.

$$\epsilon = \frac{Q_P - Q_H}{Q_H} \times 100\% \quad (5.7)$$

$$\epsilon = \frac{|6.586\text{KWH} - 22.5\text{KWH}|}{22.5\text{KWH}} \times 100\% = 70.72\%$$

To find the cooling water required take the temperature difference 10°C . The flow rate cooling water 650ml/min.

$$Q_c = mC_p\Delta T \quad (5.8)$$

Where m = mass of cooling water, Q_c = heat of cooling,

C_p = heat capacity (KJ/kg.k)

ΔT = Temperature difference, $M = 0.65\text{l} \times /min \times \frac{1\text{min}}{60\text{sec}} = 10.83 \times 10^{-3}\text{kg/sec}$

$$Q_c = mC_p\Delta T = 10.83 \times 10^{-3} \times \frac{4189.1\text{J}}{\text{kg.K}} \times (315.15 - 293.15) = 1686.53166\text{J/sec}$$

Mass of cold water used as cooler = $1686.53166 \text{ J/sec} \times \frac{1}{4200 \times 10} = 0.04016 \text{ kg/sec} = 144.56 \text{ kg/hr}$

5.1.1.2.2 Energy Balance on steam distillation for extraction of oil from fresh lemongrass

This energy balance was specifically calculated only for the water in the System. It showed that heat was absorbed into the system when boiling water and later released when water turns to vapor. Equation 5.9 shows the formula to calculate heat absorbed by the system:

$$Q_A = m C_P \Delta T \quad (5.9)$$

Where, Q_A = heat absorbed by the system (kJ), m = mass of water (kg), C_p = heat capacity (kJ/kg.K), ΔT = temperature difference ($^{\circ}\text{C}$)

At the end of the experiment there was 9.8 liters of water distilled out with lemongrass oil, Initial temperature 25°C , and final temperature 94.5°C

$$Q_A = 9.8 \text{ kg} \times 4.184 \text{ (kJ/kg.K)} \times (367.5 - 298) \text{ K} = 2762.486 \text{ KJ}$$

The heat released from the system, when water turns to vapor is illustrated in equation 5.10

$$Q_R = n \lambda \quad (5.10)$$

Where n = Number of moles of water (mol/g), $\lambda = 40.656 \times 10^3 \text{ J/mol}$ (I, 2010)

In this study, the experiment was carried out at atmospheric pressure (1atm). The heat capacity of steam at the constant pressure is $\frac{4.184 \text{ kJ}}{\text{kg.K}}$ (Perry and Green, 1997). The initial temperature of the system is 25°C (298K) and the final temperature is 94.5°C (367.5K).

$$Q_R = 9800 \text{ g} \times (1 \text{ mol} / 18 \text{ g}) \times 40.656 \text{ E3 (J/mol)} = 22,134.9333 \text{ KJ}$$

By using the data given, the total energy used by the system in this process, Q_s is as follows:

$$Q_S = Q_A + Q_R \quad (5.11)$$

$$= 2762.486 \text{ KJ} + 22,134.9333 \text{ KJ} = 24897.4193 \text{ KJ}$$

Where, Q_s = Energy used by the system in this process (kJ)

The total energy supplied into the system during the experiment, (kW.hr) is as follows:

$$Q_P = \frac{1 \text{ KWH}}{3600 \text{ KJ}} \times Q_S \quad (5.12)$$

$$Q_P = \frac{1 \text{ KWH}}{3600 \text{ KJ}} \times 24897.4193 \text{ KJ} = 6.9159 \text{ KWH}$$

The total energy supplied by the heater, Q_H (KW.hr) can be calculated by multiplying the power supplied by the heater through the whole experiment, as in the equation below:

$$Q_H = 9000 \times \frac{1}{S} \times t \quad (5.13)$$

Where, t = Optimum operating time (hr). The optimum operating point was 2.5hr

$$Q_H = 9\text{KW} \times 2.5\text{hr} = 22.5\text{KWH}$$

The equipment efficiency (heating mantle) ϵ can be calculated as follows.

$$\epsilon = \frac{Q_P - Q_H}{Q_H} \times 100\% \quad (5.14)$$

$$\epsilon = \frac{|6.9159\text{KWH} - 22.5\text{KWH}|}{22.5\text{KWH}} \times 100\% = 69.26\%$$

To find the cooling water required take the temperature difference 10°C . The flow rate cooling water 650ml/min.

$$Q_c = mC_p\Delta T \quad (5.15)$$

Where, m=mass of cooling water,

Q_c = heat of cooling, C_p = heat capacity (KJ/kg.k)

ΔT = Temperature difference

$$M = 0.65\text{l} \times /min \times \frac{1min}{60sec} = 10.83 \times 10^{-3}\text{kg/sec}$$

$$Q_c = mC_p\Delta T = 10.83 \times 10^{-3} \times \frac{4189.1\text{J}}{\text{Kg.K}} \times (315.15 - 293.15) = 1686.53166\text{J/sec}$$

$$\text{Mass of cold water used as cooler} = 1686.53166\text{J/sec} \times \frac{1}{4200 \times 10} = 0.04016\text{kg/sec} = 144.56\text{kg/hr}$$

Sizing of major equipment

Sizing and Equipment Listing For the Production of Perfume

The engineer developing a process design must be the responsibility of preparing specific cation for individual places of equipment and design data must be developed. Safety factor: is a factor to represent the amount of our design that would be used to account for the changes in the operating performance with time. As a general, safety factor = (10-20) % of capacity of the equipment.

◆ Mixing tank

$$V_m = \frac{\text{mass of input ingredients} + \text{mass of input ingredients} \times \text{safety factor}}{\text{density of input ingredient}} \quad (5.16)$$

$$= \frac{(6990.47 + 26214.24 + 122.3 + 1747.61 + 0.15(35074.62)) \frac{\text{kg}}{\text{yr}}}{\text{average density}} = \frac{134 \frac{\text{kg}}{\text{day}}}{982.81 \frac{\text{kg}}{\text{m}^3}}$$

$$= \frac{0.136\text{m}^3}{\text{day}}, \text{ Residence time two day}$$

$$V_m = \frac{0.136m^3}{day} \times 2days = 0.272m^3. \text{ Assume mixer height } 0.8m, \text{ the diameter will be calculated}$$

$$\text{as follow} = 0.272m^3 = \frac{\pi d^2}{4} \times h, d^2 = 0.433, d = 0.658m$$

◆ **Still tank**

$$V_s = \frac{\text{mass of lemongrass} + \text{mass of lemongrass} \times \text{safety factor}}{\text{density of lemongrass}}$$

$$= \frac{(863021.155kg/yr + 863021.155kg/yr \times 0.15)}{0.88125 \frac{g}{ml}} = \frac{992474.328kg/yr}{881.25kg/m^3}$$

$$= \frac{3308.24776kg/day}{881.25kg/m^3} = 3.754m^3/day, \text{ residence time } 4hr$$

$$V_s = 3.754m^3 / 16hr \times 4hr = 0.9385m^3$$

Assume the length of still tank can be estimated 1m. Then the diameter will be calculated as follow.

$$\text{Volume} = \frac{\pi d^2}{4} \times \text{length} = \frac{\pi d^2}{4} \times 1, V_s = 0.9385 = \frac{\pi d^2}{4} \times 1 \Rightarrow d^2 = 1.1949m, d = 1.09m$$

◆ **Storage of ethanol tank**

$$V_e = \frac{\text{Mass of ethanol} + \text{Mass of ethanol} \times 0.15}{\text{density of ethanol}} = \frac{87.38 + 0.15 \times 87.38 \frac{kg}{day}}{789 \frac{kg}{m^3}} = \frac{100.487 \frac{kg}{day}}{789 \frac{kg}{m^3}}$$

$$= \frac{0.1273m^3}{day}, \text{ seven day residence time (day)}, V_e = \frac{0.1273}{day} \times 7days = 0.8911m^3$$

◆ **Storage of water tank**

From total required per year 9,153,241.38 lit/yr

$$V_w = \frac{\text{Mass of water} + \text{Mass of water} \times 0.15}{\text{density of water}} = \frac{(25060.209 + 25060.209 \times 0.15) \frac{kg}{day}}{1000 \frac{kg}{m^3}}$$

$$= \frac{28819.240 \frac{kg}{day}}{1000 \frac{kg}{m^3}} = \frac{28.81924m^3}{day}, \text{ One day residence time}$$

$$V_w = \frac{28.8192m^3}{day} \times day = 28.8192m^3. L=4.4m, d=2.9m$$

5.1.2 Economic analysis of perfume formulation from lemongrass additive

5.1.2.1 Building, equipment and manpower requirements

◆ Plant parameters

Capacity per year = 34,602.82kg/yr

Working days/year = 300 day

Number of shifts /day = 2

Land area/ covered, =1000m²

◆ Machinery and equipment

Purchased cost for some basic plant equipment's are estimated from www.mhhe.com/engcs/chemical/peters/data/, and [www. Matche/equipcost/index.com](http://www.Matche/equipcost/index.com).

Table 5.1 Specification and purchased equipment cost.

Purchased equipment	Capacity	Qty	Material	Total cost (birr)
Belt conveyer	L=7m	3	Carbon steel	382,807.5
Rotary Cutter	2kg/s	1	-	267,653.5
Mixing tank	0.38m ³	1	Stainless steel	119,626
Distillation(tray)	D=1.09m	1	Carbon steel	137,858
Condesor	0.07m ³ /s	1	Carbon steel	179095
Filter gravity	1m ²	1	Carbon steel	255,850
Boiler	-	1	Stainless steel	355395
Ethanol Storage	0.89m ³	1	Carbon steel	70,950
Homogenizer	1m ³	1	304 Stainless steel	569,664
Filter (1PCS)	Db=0.5m	1	Carbon steel	320,350
Filler/bottling machine	1.05bt/min	1	-	430,000
Pump	0.009m ³ /s	1	Centrifuge Cast steel	119,045.5
pump	0.004m ³ /s	2	Diaphram	150,672
Water storage tank (H)	L=4.4m		Carbon steel	330,734.5
Total				3,689,701
Contingencies on Equipment 10%				368,970.1
Transportation cost = 10% of equipment cost				368,970.1
Grand total				4,427,641.1

◆ **Manpower requirement**

Table 5.2 manpower requirement & cost

Sr.No	Manpower	Req. No	Monthly Salary (Birr)	Annual Salary (Birr)
1	General manager	1	5000	60,000
2	Secretary	2	2000	48,000
3	Accountant	2	2500	60,000
4	Production and Technical Head	1	3000	36,000
5	Mechanic	2	2000	48,000
6	Electrical	2	2000	48,000
7	Store keeper	2	1800	43,200
8	Quality control	2	2500	60,000
9	Operators	6	2000	144,000
10	Ass. Operators	3	1800	64,800
11	Guards	2	1500	36,000
Total				648,000

5.1.2.2 Cost estimation

◆ **Cost of raw materials**

Table 5.3 Cost of raw materials

Particulars	Unit price	Quantity per annum	Total cost(birr)
Lemon grass	5birr/kg	1,015,504.54kg/yr	5,077,522.7
Ethanol	17.95birr/lit	26,214.26lit/yr	470,545.967
Water	0.005Bir/lit	6,407,268.97lit/yr	3,497.2
Castor oil or glycerin	24/lit	122.3lt/yr	2,935.2
Total raw material cost			5,554,501.07

◆ **Fixed capital cost estimation**

Table 5.4 Estimation of Fixed Capital Investment

	Component	Factor	Cost (birr)
1.Total direct cost	Purchased equipment cost (PEC)	-	4,427,641.1
	Installation	0.25 PEC	1,106,910.275
	Instrumentation	0.1PEC	442,764.11
	Electrical installed	0.1PEC	442,764.11
	Piping installed	0.2PEC	885,528.22
	Building including service	0.1PEC	442,764.11
	Yard improvement	0.4PEC	1,771,056.44
	Land	-	-
	Total direct cost (DC)		
2.Total indirect cost	Engineering and supervision	0.06 DC	571,165.702
	Construction expense and contractors fee	0.05DC	475971.418
	Total indirect cost (IC)		1,047,137.12
3. Fixed capital investment (FCI)	FCI = DC + IC		10,566,565.485
4.Working capital	0.15TCI		1,864,688.03
5.Total capital investment (TCI)	WC + FCI		12,431,253.512

$$FCI = DC + IC, FCI = 10,566,565.485 \text{ birr}$$

$$TCI = FCI + WC, \text{ since working capital cost} = (10-20) \% \text{ of total capital investment}$$

$$TCI = FCI + 0.15TCI, TCI = 10,566,565.485 + 0.15TCI$$

$$TCI = \frac{10,566,565.485}{0.85} = 12,431,253.512 \text{ birr}$$

◆ **Estimation of total product cost**

$$\text{Total production cost}(x) = \text{Manufacturing cost} + \text{General expense}$$

Manufacturing cost = Direct production cost + Fixed charges + Plant overhead cost.

A. Fixed Charges (FC)

1. Depreciation = 10% of equipment cost + 2.5% of building = $442,764.11 + 442,764.11 \times 0.025$
= 453,833.212 birr,

2. Local taxes = 2.5% of FCI = $0.025 \times 10,566,565.485 = 264,164.14$ birr

3. Insurance = 0.7% of FCI = $0.007 \times 10,566,565.485 = 73,965.9583$ birr

Thus, Fixed Charges = $453,833.212 \text{ birr} + 264,164.14 \text{ birr} + 73,965.9583 \text{ birr} = 791,963.31 \text{ birr}$

B. Direct Production Cost: (about 60% of total product cost) Let, the total product cost be X

- Raw materials raw material cost = 5,554,501.07 birr
- Operating labor (OL): Operating labor cost = 648,000
- Direct Supervisory and Clerical Labor (DS & CL): (10-25% of OL) Consider the cost for Direct supervisory and clerical labor = 14% of OL
Labor cost = $0.14 \times 648,000 \text{ birr} = 90,720 \text{ birr}$
- Utilities: (10-20% of total product cost) Consider the cost of Utilities = 15% of total product cost
Utilities cost = $0.15 * X$
- Maintenance and repairs (M & R): (2-10% of fixed capital investment) Consider the Maintenance and repair cost = 5% of fixed capital investment
Maintenance and repair cost = $0.05 \times 10,566,565.485 = 528,328.27 \text{ birr}$
- Laboratory Charges: (10-20% of OL) Consider the Laboratory charges = 15% of OL
Laboratory charges = $0.15 \times 648,000 \text{ birr} = 97,200 \text{ birr}$

Direct Production Cost (DPC), $X_b = 5,554,501.07 \text{ birr} + 648,000 \text{ birr} + 90,720 \text{ birr} + 0.15 * X$
 $X_b = 0.15 * X + 6,918,749.34 \text{ birr}$, $X_b = 13,939,427.818 \times 0.15 \text{ birr} + 6,918,749.34 = 9,009,663.51$

C. Plant overhead Costs :

(50-70% of Operating labor, supervision, and maintenance or 5-15% of total product cost) includes for the following: general plant upkeep and overhead, payroll overhead, packaging, medical services, safety and protection, restaurants, recreation, salvage, laboratories, and storage facilities. Plant overhead cost 10% of total product cost, Plant overhead cost = $0.1 * X$

Manufacture cost = Direct production cost + Fixed charges + Plant overhead costs.

Manufacture cost = $0.15 * X + 6,874,721.99 \text{ birr} + 791,963.31 \text{ birr} + 0.1 * X$

D. General expenses

- Administration cost (4% of TPC) = $0.04 * X$
- Distribution and selling cost (11% of TPC) = $0.11 * X$
- Research and development cost (5% of TPC) = $0.05 * X$

$$\text{General expense} = 0.04 * X + 0.11 * X + 0.05 * X$$

Total production cost(x) = Manufacturing cost + General expense

$$X = 0.15 * X + 6,874,721.99 \text{ birr} + 791,963.31 \text{ birr} + 0.1 * X + 0.04 * X + 0.11 * X + 0.05 * X$$

$$X = 0.45X + 7,666,685.3 \text{ birr}, X - 0.45X = 7,666,685.3 \text{ birr}$$

$$\text{TPC}(X) = \frac{7,666,685.3 \text{ birr}}{0.55} = 13,939,427.818 \text{ birr}$$

◆ Gross earning/ income

Total income from product = unit selling price × Quantity of product manufactured

$$\text{Annual income} = 40 \text{ birr}/60 \text{ ml bottle} \times 35,208.046 \text{ lit/yr} \times 1000 \text{ ml/lit} = 23,472,030.8 \text{ br}$$

Total income from solid cake = unit selling price × Quantity of by product...

$$\text{Annually earning of solid cake} = 2.00 \text{ birr/kg} * (431,510.575 \text{ kg/yr}) = 863,021.15 \text{ birr}$$

$$\text{Total Income} = \text{Income of product} + \text{earning of solid cake} = 23,472,030.8 \text{ br} + 863,021.15 \text{ birr}$$

$$\text{Total income} = 24,335,052 \text{ birr}$$

Gross income = Total Income – Total Product Cost

$$= 24,335,052 \text{ birr} - 13,939,427.818 \text{ birr} = 10,395,624.182 \text{ birr}$$

Let the tax rate be 35% (income tax of Ethiopia)

$$\text{Taxes} = 0.35 * 10,395,624.182 \text{ birr} = 3,638,468.46 \text{ birr}$$

$$\text{Net profit} = \text{gross income} - \text{tax} = 10,395,624.182 \text{ birr} - 3,638,468.46 \text{ birr} = 6,757,155.718 \text{ birr}$$

◆ Rate of return

Minimum acceptable rate of return (Mar) Minimum acceptable rate of return (mar) for new capacity with established corporate with low levels of risk = 12%

$$\text{ROI} = \frac{\text{Net profit}}{\text{Total capital investment}} \times 100\% = \frac{6,757,155.718 \text{ br}}{12,431,253.512} \times 100\% = 54.35\%$$

Since $\text{ROI} \geq \text{Mar}$, $59.98 \geq 12\%$ the project is feasible

$$\text{Payback period} = \frac{\text{FIC}}{\text{NP} + \text{Depre}} = \frac{10,566,565.485}{6,757,155.718 \text{ br} + 453,833.212 \text{ br}} = \frac{10,566,565.485}{7,210,988.928} = 1.5 \text{ yrs.}$$

◆ **Break even analysis**

Breakeven point is the point when total annual production cost equals total annual sales. That is the point where profit equals zero. The breakeven point is determined from the relation:

$$BEP = \frac{TFC}{Sup - V_{cup}} = \frac{TPC - DPC}{Sup - V_{cup}}$$

Where BEP = Break-even point (units of production), V_{cup} = variable costs per unit of production, Sup = selling price per unit of production, TPC = total production cost, DPC = direct production cost.

$$V_{cup} = \frac{\text{Direct production cost}}{\text{Amount of perfume produced}} = \frac{9,009,663.51\text{birr/yr}}{35,208,046\text{ml/yr}} = 0.256\text{birr/ml}$$

$$Sup = 0.667\text{birr/ml}$$

$$BEP = \frac{(13,939,427.818\text{birr} - 9,009,663.51\text{birr})/\text{yr}}{(0.667 - 0.256)\text{birr/ml}} = \frac{4,929,764.31}{0.411} = 11,994,560.36 \text{ ml/yr}$$

$$BEP (\%) = \frac{11,994,560.36 \text{ ml/yr}}{35,208,046\text{ml/yr}} \times 100\% = 34.1\%$$

Perfume development from essential oil using steam distillation extraction from lemongrass is profitable as it is clearly observed from the above cost estimation. The rate of return investment 54.35% implies the plant returns 54.35% of its total capital investment in fifteen month. The payback period tells us the plant return its total investment cost in around fifteen month. Totally the income statement and the other indicators of profitability show that the project is viable. The project can be implemented after detailed feasibility study has been done.

6. Conclusion and Recommendations

6.1 Conclusion.

This work was intended to study the influence of different factors (Extraction time and Particle sizes,) on the quality and quantity of extraction of essential oil from dry and wet raw material of lemongrass that used for perfume development. Variability of these operating conditions is the pre-dominant factors for the quantity of the oil. There are different methods of essential oil extraction from lemongrass. In this thesis, steam distillation extraction was used due to availability of the equipment.

The percentage essential oil yield in dried samples is higher as compared to fresh samples. The reason for that Peisino *et al.* 2005 explained as essential oil extracted from the wet plants present fewer components than dry sample due to water molecules solvate the components.

Characterization of essential oil was determined to see its quality and the main component of monoterpenes hydrocarbons which is citral as indicated by the strong intense vibration frequency in the FT-IR and in the GC-MS spectrograph. The citral was the main component of essential oil which has aroma that used to develop perfume. The maximum oil yield obtained was 0.81% for dry and 0.345% for wet lemon grass at the particle size of 8-15mm and the extraction time of 2.5 hours, the observed quantitative difference in the quantity of the oil was due to particle size and extraction time variability. Thus, determination of the appropriate size of the particles and optimal time needs to have a consideration to get the maximum amount of the required product for development of perfume. Finally perfume was developed from essential oil of tej-sar and sensor analysis was conducted to see its quality of aroma and the result was acceptable.

Based on the rough economic analysis of steam distillation extraction and development of perfume using lemongrass essential oil is profitable since the rate of return on investment was 54.35% %, this show us the project returns its 54.35%% of the initial investment in one and fifth month.

6.2 Recommendation

- ◆ Here in the experimental work, temperature and pressure factors have not been studied. This is due to uncontrollability of equipment of steam distillation that used for extraction of essential oil.
- ◆ Sedimentation analysis should be carried out on the yield obtained so that the approximate weight of the oil and water can be determined.
- ◆ It also recommends that formulation of perfume from lemongrass and other aromatic plant should have to be carried out to see the intensity and quality of it.
- ◆ A further study can also be conducted in focusing on the quality of the lemongrass essential oil obtained at each operating conditions using GC-MS. This will provide alternative to manufacturers on the minimum conditions required to get a specific quality of essential oil with minimum capital for perfume application.
- ◆ Here also recommend that the project to be implemented after detail feasibility studies.

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APPENDIX

Appendix A

Results from UV-Visible, GC-MS and design expert software

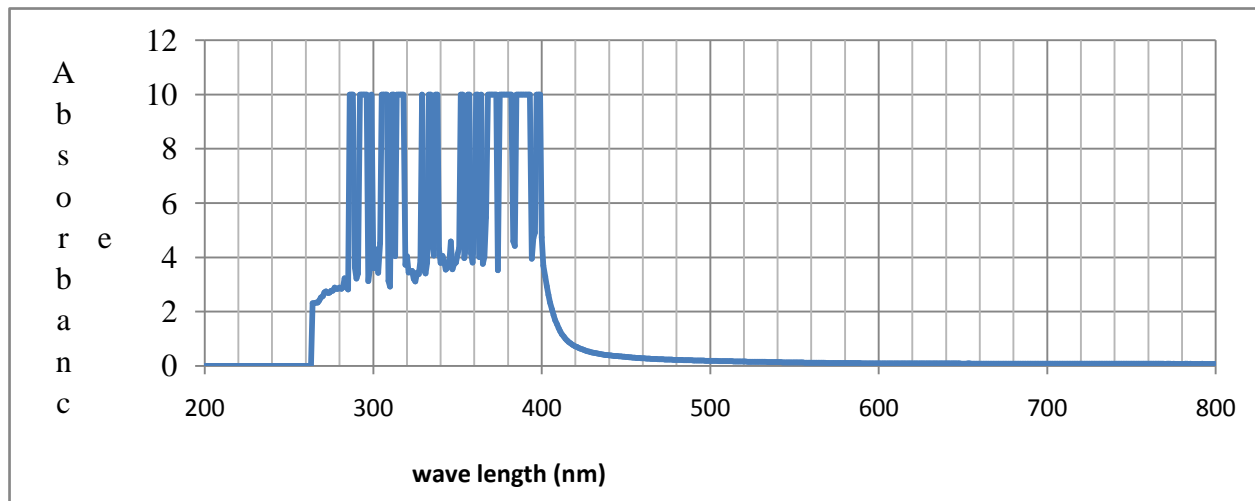


Fig.A1. UV-visible spectroscopy result of extracted essential oil from fresh lemongrass (citrus)

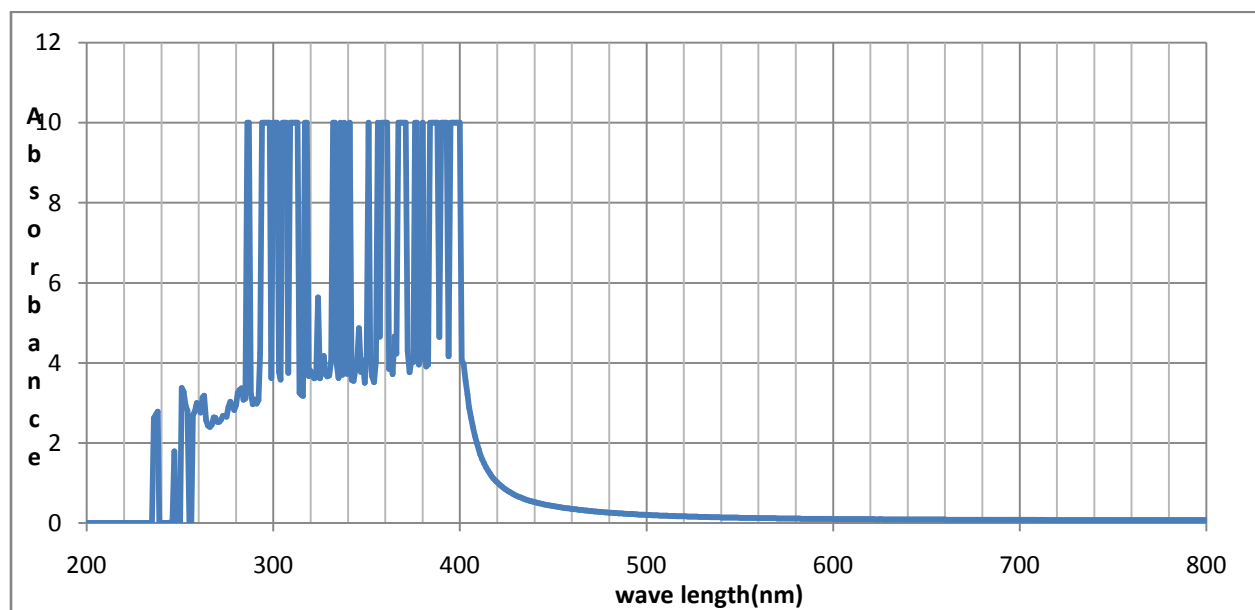


Fig.A2. UV-visible spectroscopy result of extracted essential oil from dry lemongrass (citrus).

From this figure many component observed and it is complex to analysis using UV-visible spectroscopy. From 200-300nm many components were observed in dry lemongrass extracted due to existence of component. Most components were extracted from dry lemongrass rather than fresh because of no solvation happened during extraction.

Table.A1.Library search report of GC-MS for essential oil of dry lemongrass.

Library Search Report

Data Path : D:\MassHunter\GCMS\1\5977\MAR 09\
 Data File : 03801038.D
 Acq On : 11 Mar 2016 14:13
 Operator : Estif
 Sample : LEMMON.G
 Misc :
 ALS Vial : 38 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	8.345	2.24	D:\MassHunter\Library\NIST14.L .beta.-Myrcene .beta.-Myrcene Bicyclo[3.1.0]hex-2-ene, 4-methyl- 1-(1-methylethyl)-	16066 16061 16276	000123-35-3 000123-35-3 028634-89-1	96 91 87
2	12.634	0.48	D:\MassHunter\Library\NIST14.L Linalool Linalool Linalool	27447 27451 27453	000078-70-6 000078-70-6 000078-70-6	97 90 86
3	15.417	0.99	D:\MassHunter\Library\NIST14.L Isoneral Isogeranial 3,6-Octadienal, 3,7-dimethyl-	25795 25818 25922	1000414-18-0 1000414-18-1 055722-59-3	98 94 93
4	16.208	1.40	D:\MassHunter\Library\NIST14.L Isogeranial 3,6-Octadienal, 3,7-dimethyl- Isoneral	25818 25922 25795	1000414-18-1 055722-59-3 1000414-18-0	98 95 64
5	18.835	23.27	D:\MassHunter\Library\NIST14.L 2,6-Octadienal, 3,7-dimethyl-, (Z) 3-Decyne 1-(1-Butyny)cyclopentanol	25967 17251 17973	000106-26-3 002384-85-2 1000342-86-5	62 38 35
6	19.394	2.40	D:\MassHunter\Library\NIST14.L Geraniol 2,6-Octadien-1-ol, 3,7-dimethyl-, (Z)- 4-Hexen-1-ol, 5-methyl-2-(1-methyl ethenyl)-, (R)-	27446 27682 27765	000106-24-1 000106-25-2 000498-16-8	95 91 83
7	20.182	30.43	D:\MassHunter\Library\NIST14.L Citral 2,6-Octadienal, 3,7-dimethyl-, (E) Citral	25782 25959 25781	005392-40-5 000141-27-5 005392-40-5	95 74 64
8	23.004	1.04	D:\MassHunter\Library\NIST14.L 2,7-Dimethyl-2,7-octanediol Methanamine, N-[3-methyl-2-butenyl idene] 1H-Imidazole-4-ethanamine, N-methyl-	42787 3044 10982	019781-07-8 1000196-86-4 000673-50-7	38 35 35
9	24.562	1.57	D:\MassHunter\Library\NIST14.L 2,4-Dimethyl-1-hepten-4-ol trans-(2-Ethylcyclopentyl)methanol Cyclohexene,3-propyl-	20529 12905 10848	019549-94-1 036258-08-9 003983-06-0	35 27 16
10	24.954	1.11	D:\MassHunter\Library\NIST14.L			

Essential oil.M Tue Mar 22 12:35:13 2016

1

Library Search Report

Data Path : D:\MassHunter\GCMS\1\5977\MAR 09\
Data File : 03801038.D
Acq On : 11 Mar 2016 14:13
Operator : Estif
Sample : LEMMON.G
Misc :
ALS Vial : 38 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
			Geranyl acetate	61517	000105-87-3	91
			Butanoic acid, 3,7-dimethyl-2,6-oc tadienyl ester, (E)-	87637	000106-29-6	91
			2,6-Octadien-1-ol, 3,7-dimethyl-, acetate	61587	016409-44-2	91
11	38.247	3.66	D:\MassHunter\Library\NIST14.L Methyl tetradecanoate	104286	000124-10-7	99
			Methyl tetradecanoate	104287	000124-10-7	97
			Methyl tetradecanoate	104288	000124-10-7	96
12	38.984	0.60	D:\MassHunter\Library\NIST14.L (6R,7R)-Bisabolone	83529	072441-71-5	98
			(S)-3-Methyl-6-((R)-6-methylhept-5 -en-2-yl)cyclohex-2-enone	83613	072441-70-4	95
			3-Methyl-6-(6-methylhept-5-en-2-yl) cyclohex-2-enone	83600	066964-98-5	64
13	41.753	0.62	D:\MassHunter\Library\NIST14.L Pentadecanoic acid, methyl ester	117468	007132-64-1	99
			Pentadecanoic acid, methyl ester	117471	007132-64-1	98
			Pentadecanoic acid, methyl ester	117470	007132-64-1	97
14	44.389	2.26	D:\MassHunter\Library\NIST14.L 9-Hexadecenoic acid, methyl ester, (Z)-	128700	001120-25-8	99
			9-Hexadecenoic acid, methyl ester, (Z)-	128693	001120-25-8	99
			7-Hexadecenoic acid, methyl ester, (Z)-	128697	056875-67-3	99
15	45.127	10.28	D:\MassHunter\Library\NIST14.L Hexadecanoic acid, methyl ester	130813	000112-39-0	98
			Hexadecanoic acid, methyl ester	130822	000112-39-0	97
			Hexadecanoic acid, methyl ester	130820	000112-39-0	96
16	50.386	1.36	D:\MassHunter\Library\NIST14.L 9,12-Octadecadienoic acid (Z,Z)-, methyl ester	153891	000112-63-0	99
			9,12-Octadecadienoic acid, methyl ester	153873	002462-85-3	99
			9,12-Octadecadienoic acid (Z,Z)-, methyl ester	153889	000112-63-0	99
17	50.600	10.47	D:\MassHunter\Library\NIST14.L cis-13-Octadecenoic acid, methyl e ster	155747	1000333-58-3	99
			7-Octadecenoic acid, methyl ester	155720	057396-98-2	99
			9-Octadecenoic acid, methyl ester,	155758	001937-62-8	99

Library Search Report

Data Path : D:\MassHunter\GCMS\1\5977\MAR 09\
 Data File : 03801038.D
 Acq On : 11 Mar 2016 14:13
 Operator : Estif
 Sample : LEMMON.G
 Misc :
 ALS Vial : 38 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
18	50.764	0.71	D:\MassHunter\Library\NIST14.L			
			11-Octadecenoic acid, methyl ester	155737	052380-33-3	99
			cis-13-Octadecenoic acid, methyl ester	155747	1000333-58-3	99
			9-Octadecenoic acid, methyl ester, (E)-	155758	001937-62-8	99
19	50.913	1.38	D:\MassHunter\Library\NIST14.L			
			cis-13-Octadecenoic acid, methyl ester	155747	1000333-58-3	99
			9-Octadecenoic acid, methyl ester, (E)-	155758	001937-62-8	99
			11-Octadecenoic acid, methyl ester	155737	052380-33-3	99
20	51.406	3.70	D:\MassHunter\Library\NIST14.L			
			Methyl stearate	157879	000112-61-8	99
			Methyl stearate	157883	000112-61-8	98
			Methyl stearate	157882	000112-61-8	98

APPENDIX B

Steam distillation Extracted Data Analysis by design expert

Table .B1. Analysis of experimental (actual) data of essential oil from dry lemongrass (citratrus)

Std	Run	Block	Factor 1 A:A	Factor 2 B:B	Response 1 oil yild %
24	1	Block 1	180	25-35	0.58
18	2	Block 1	90	25-35	0.24
19	3	Block 1	120	25-35	0.38
8	4	Block 1	180	8-15	0.78
13	5	Block 1	150	15-25	0.72
10	6	Block 1	90	15-25	0.27
3	7	Block 1	120	8-15	0.66
16	8	Block 1	180	15-25	0.71
9	9	Block 1	90	15-25	0.33
20	10	Block 1	120	25-35	0.43
6	11	Block 1	150	8-15	0.83
5	12	Block 1	150	8-15	0.79
23	13	Block 1	180	25-35	0.54
4	14	Block 1	120	8-15	0.63
17	15	Block 1	90	25-35	0.21
14	16	Block 1	150	15-25	0.67
11	17	Block 1	120	15-25	0.54
7	18	Block 1	180	8-15	0.81
21	19	Block 1	150	25-35	0.57
22	20	Block 1	150	25-35	0.53
1	21	Block 1	90	8-15	0.43
12	22	Block 1	120	15-25	0.5
15	23	Block 1	180	15-25	0.69
2	24	Block 1	90	8-15	0.35

Table B2. Values for reasonable agreements

Std. Dev	0.03195	R-Squared	0.984765
Mean	0.549583	Adj R-Squared	0.970801
C.V	5.813583	Pred R-Squared	0.939062
PRESS	0.049	Adeq Precision	25.89366

The "Pred R-Squared" of 0.939062 is in reasonable agreement with the "Adj R-Squared" of 0.970801."Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. Our ratio of 25.89366 indicates an adequate signal. This model can be used to navigate the design space.

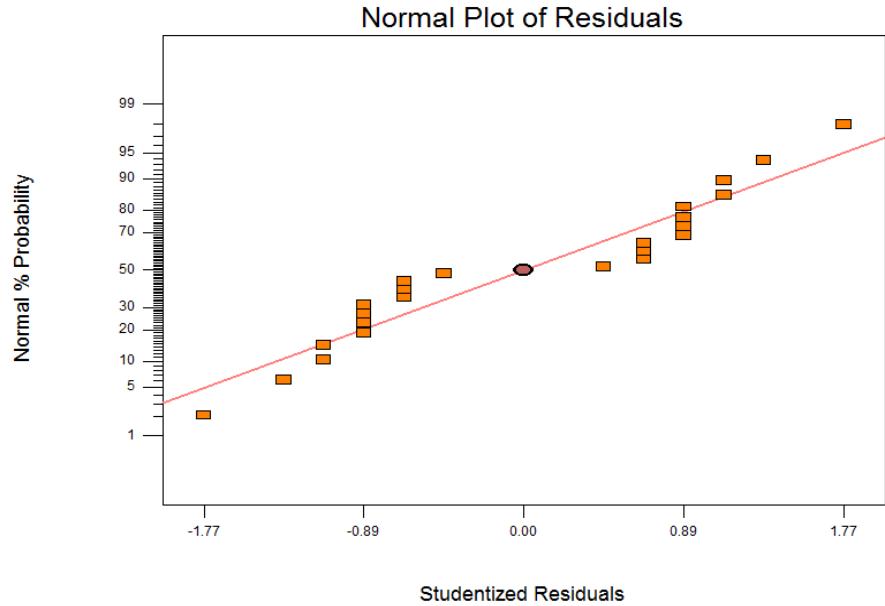


Fig.B1. Graph of Normal plots of residual **by design expert**

Table B3. Analysis of experimental (actual) data of essential oil from fresh lemongrass

Std	Run	Block	Factor 1 A:Extraction time minute	Factor 2 B:particle size mm	Response 1 oil yield %
14	1	Block 1	150	15-25	0.31
22	2	Block 1	150	25-35	0.23
23	3	Block 1	180	25-35	0.21
10	4	Block 1	90	15-25	0.16
1	5	Block 1	90	8-15	0.21
11	6	Block 1	120	15-25	0.24
8	7	Block 1	180	8-15	0.36
17	8	Block 1	90	25-35	0.12
19	9	Block 1	120	25-35	0.18
18	10	Block 1	90	25-35	0.13
2	11	Block 1	90	8-15	0.18
13	12	Block 1	150	15-25	0.27
12	13	Block 1	120	15-25	0.26
15	14	Block 1	180	15-25	0.26
5	15	Block 1	150	8-15	0.32
3	16	Block 1	120	8-15	0.29
21	17	Block 1	150	25-35	0.22
20	18	Block 1	120	25-35	0.2
9	19	Block 1	90	15-25	0.14
4	20	Block 1	120	8-15	0.32
16	21	Block 1	180	15-25	0.3
7	22	Block 1	180	8-15	0.33
6	23	Block 1	150	8-15	0.37
24	24	Block 1	180	25-35	0.24

Table B4. Values for reasonable agreements

Std. Dev	0.021115	R-Squared	0.954876
Mean	0.24375	Adj R-Squared	0.913513
C.V	8.662468	Pred R-Squared	0.819504
PRESS	0.0214	Adeq Precision	14.73504

The high F value ($F_{\text{model}} = 23.08$) with low probability value ($P < 0.0001$) indicates the significance of the fitted model. The low value of the coefficient of variation ($CV = 8.66\%$) indicates that results of the fitted model are reliable. The quality of the model fit was evaluated by the coefficient of determination (R^2), this value being calculated to be 0.95 for the response, indicating that the developed model equation successfully captured the correlation between the process parameters to the yield of bottom product. The adjusted coefficient of determination ($R^2_{\text{Adj.}}$) value reconstructs the expression with all the significant terms included. The value of the adjusted coefficient of determination ($R^2_{\text{Adj.}} = 0.91$) is also very high, supporting the significance of the model. As the fitted model provides a good approximation to the experimental condition, the model was employed to find the values of the process variables for optimum yield of lemongrass oil.

DESIGN-EXPERT Plot
oil yield

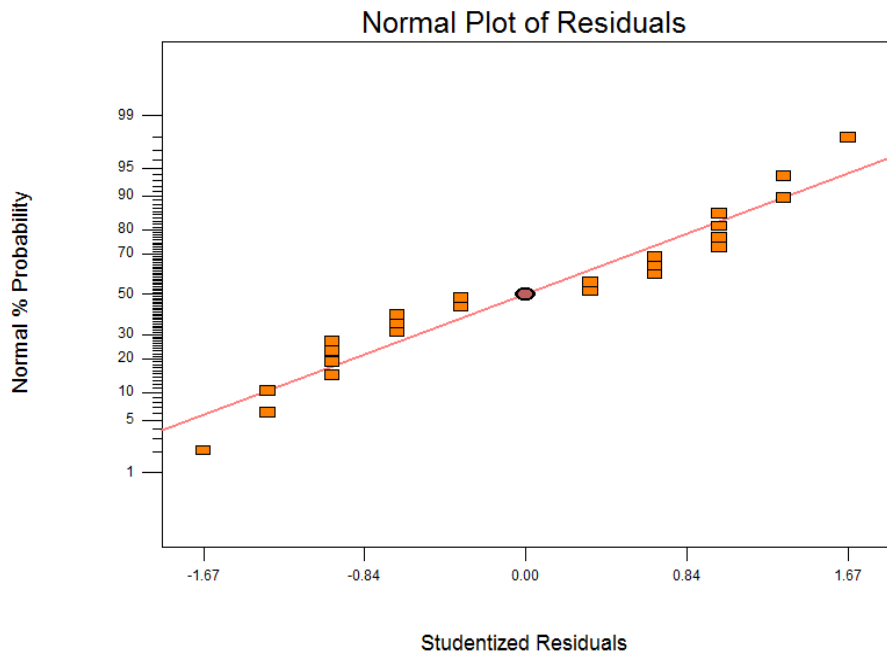


Fig.B2. Graph of Normal plots of Residua

APPENDIX C

The design calculations of the condenser fully discussed below. The heat capacity of the system will be calculated.

◆ Heat capacity:

$$H = \dot{m} C \quad (C_1)$$

where, H = heat capacity (W/K) of the oil at 20°C , C = specific heat capacity (KJ/Kg.K) of the oil at 20°C , \dot{m} = mass flow rate (Kg/s)

◆ For water:

$$H_w = \dot{m}_w C_w \quad (C_2)$$

Where, $\dot{m}_w = 0.04\text{Kg/s}$ is calculated, C_w = specific heat capacity of water at 20°C is 4225J/Kg.K

$$H_w = 169\text{W/K}$$

◆ For Lemon grass oil;

$H_{lg} = \dot{m}_{lg} C_{lg}$, C_{lg} = specific heat capacity of water at 20°C is 4200J/Kg.K

$\dot{m}_{lg} = 0.022\text{Kg/s}$ is assumed

$$H_{lg} = 92.4\text{W/K}$$

◆ Heat transfer (Q)

$$Q_{\max} = (Th_{in} - Th_{out}) \quad (C_3)$$

Where, Th_{in} = inlet temperature of lemon grass oil (94.5°C), Th_{out} = outlet temperature of lemon grass oil (20°C)

$$Q_{\max} = 6883.8\text{W}$$

To obtain the temperature of the water at the outlet, after the cooling effect; we have:

$$Q = (T_{w_{out}} - T_{w_{in}})$$

$$T_{w_{out}} = \frac{Q_{\max}}{H_w} + T_{w_{in}}, T_{w_{out}} = 40.73^{\circ}\text{C}$$

In summary, the operating temperature of oil and water are:

Oil inlet temperature = 94.50°C

Outlet temperature of oil = 20°C

Water inlet temperature = 25°C

Outlet temperature of water = 40.75°C

◆ For the tube dimensions

Heat supplied (Q) by steam to the condenser

$$Q = \dot{m}_{lg} L + \dot{m}_{lg} C_{lg} \Delta T \quad (C_4)$$

Where, L = latent heat of vaporization, 40706.136KJ/Kg, ΔT = temperature change ($^{\circ}\text{C}$)

$$Q = 902.41877\text{KJ/s}$$

Assumptions:

- ◆ Both oil and water flows are fully developed.
- ◆ Properties of the water and oil are constant.
- ◆ Steady operating conditions exist.
- ◆ There is turbulent flow in the tubes

Thus, for turbulent flow, Reynolds number is given as

$5000 < \text{Re} < 10,000$, take $\text{Re} = 5500$, $\mu_{lg} = 1.72\text{E-}3 \text{ Nsm-}2$, μ_{lg} = viscosity of lemon grass oil at 20°C , $\dot{m}_{lg} = 0.022\text{Kg/s}$

From
$$\text{Re} = \frac{4\dot{m}}{\pi\mu D} \tag{C_5}$$

$D = 0.33755\text{m}$ OR $D = 337.55\text{mm}$

Hence, diameter of tube can be obtained within the range 330mm to 340 mm depending on availability in the market.

Now, to obtain the length of the tube in which the condensation will be completed.

Using Prandtl's Number, Pr

$$\text{Pr} = \frac{C_p \times \mu_w}{k} \tag{C_6}$$

Where, K = thermal conductivity of water 0.58W/mK

C_p = specific heat capacity of water at constant pressure, 4200J/Kg.K

μ_w = viscosity of water, 0.798E-3Nsm-2

$\text{Pr} = 5.78$

◆ **Nusselt Number, Nu is given as**

$$\text{Nu} = 0.023\text{Re}^{0.8}\text{Pr}^{0.4} \tag{C_7}$$

$\text{Nu} = 45.58$

$$\text{Nu} = \frac{hD}{K} \tag{C_8}$$

Where, h = heat transfer coefficient, W/m²K

K = thermal conductivity of copper material at 25⁰C; 401W/mK

◆ **From which h can be derived**

$h = 54,147.77\text{W/m}^2\text{K}$

That is, the amount derived above will be the quantity of heat transferred across the tube surface to the surrounding.

Rate of heat transferred through the condenser is:

$$Q = h.A_t.\Delta T_{lm} \tag{C_9}$$

$$\text{Where, } \Delta T_{lm} = \frac{\Delta T_A - \Delta T_B}{\ln\left(\frac{\Delta T_A}{\Delta T_B}\right)} = \frac{74.5 - 22.75}{\ln\left(\frac{74.5}{22.75}\right)} = \frac{51.75}{\ln(3.27)} = 43.8 \text{ } ^\circ\text{C}$$

A_t = area of tube

ΔT_{lm} = logarithmic mean temperature difference between the two fluids = 43.8 °C

Then, $A_t = 0.38\text{m}^2$

But, $A_t = \pi D L_t$ from this L_t can be calculated $L_t = 0.3585\text{m}$

◆ **Design Calculations for Pump Discharge Rate**

To determine the discharge rate, pressure of water at a maximum head of 1.5m and the pipe diameter to suit a 2.5hp pump.

$$\text{Power, } P_w = \frac{\text{force} \times \text{distance}}{\text{time}}$$

$$P_w = \text{pressure} \times \text{volumetric rate} \tag{C_{10}}$$

But, volumetric rate, $\dot{v} = AU$, U = velocity of flow, m/s

Also, $\dot{m} = \rho \dot{v}$, ρ = density of water, Kg/m^3

Heat is supplied at the rate of 902.41877KJ/s to the condenser. The mass flow rate of water through the condenser is given as:

$$Q = \dot{m}_w C_{pw} . \Delta T_2 \tag{C_{11}}$$

Where, C_{pw} = specific heat capacity of water at 25°C = 4128.97J/Kg.K

$\dot{m}_w = 13.87\text{Kg/s}$, $\dot{v} = 0.01387\text{m}^3/\text{s}$

◆ **To obtain diameter of pipe**

$$\dot{v} = AU \tag{C_{12}}$$

$$\text{Then, } \dot{m} = \frac{\rho A U}{U}$$

$$P = \rho U^2 \tag{C_{13}}$$

$U = 10.066\text{m/s}$

Hence, $A = \frac{\dot{v}}{U} = \frac{4}{\pi D^2}$, $D = 0.0331\text{m}$

Appendix D:
Laboratory equipments and samples photo.



D₁.Lemongrass (*Cymbopogon citratus*)



Essential Oil

D₂.Gravity oil separator



D₃. Vibro-viscometer



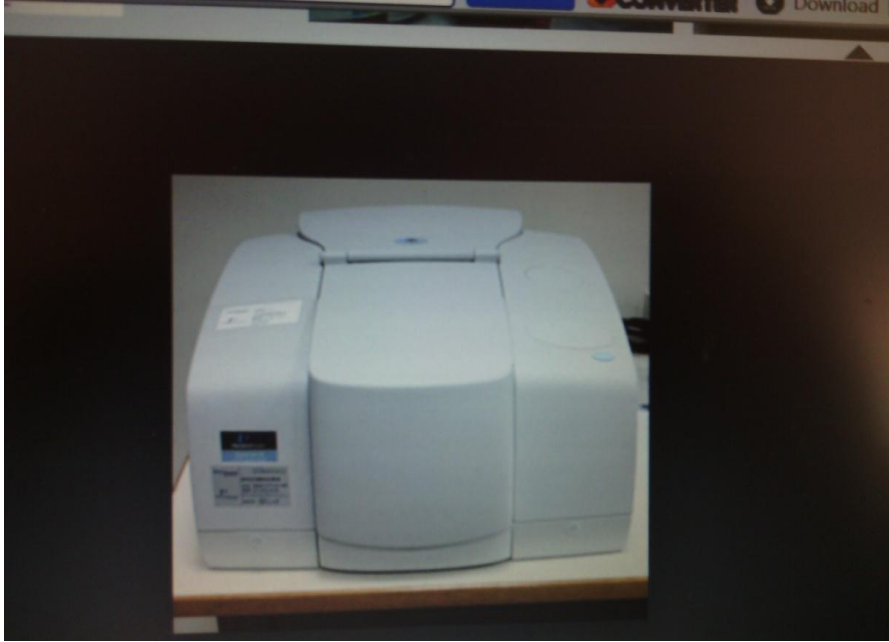
D₄. Determination of optical rotation by prism spectrometer



D₅.Determination of refractive index by prism spectrometer



D₆.Uv-visible spectroscopy



D₇.FT-IR



D₈.GC-MS



D₉.Perfume sample

Signed declaration

I, the undersigned, declare that this thesis is my original work, has not been presented for a degree in any other University and that all sources of materials used for the thesis have been duly acknowledged.

Name: _____

Signature: _____

Place Addis Ababa, Ethiopia

Date of submission: _____

This thesis has been submitted for examination with our approved as University Advisors.

Name: _____

Signature: _____