# WOLKITE UNIVERSITY

## COLLEGE OF ENGINEERING AND TECHNOLOGY

## DEPARTMENT OF CHEMICAL ENGINEERING

**FINAL YEAR THESIS ON EXTRACTION AND CHARACTERIZATION OF ESSENTIAL OIL FROM EUCALYPUS LEAVES FOR PHARMACEUTICAL PURPOSE.**

**A Thesis Submitted to Department of Chemical Engineering College of engineering and technology Wolkite University in Partial Fulfillment of the Requirements for the Degree of Bachelor of Science in Chemical Engineering (process engineering stream).**


**Prepared By**

**Wogderes Mekonnen…….. ENGR/911/09**

**Tibebu Abebawu……..ENGR/855/09**

**Yosef Adisu…….ENGR/1100/09**

**Ashenafi Nigatu……...ENGR/126/08**

**Mekonen Taye…….ENGR/588/08**


**WOLKITE ETHIOPIA**

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## DECLARATION

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Name of Advisor: Mr.Romedan Ahmed: date: _____ Signature: _____

| **Name of the student** | | **Sign. of each student** |
|---|---|---|
| Wogderes Mekonnen | date: _____ | _____ |
| Tibebu Abebaw | date: _____ | _____ |
| Yosef Adisu | date: _____ | _____ |
| Ashenafi Nigatu | date: _____ | _____ |
| Mekonnen Taye | date: _____ | _____ |

DATE OF SUBMISSION: AUGUST/23/2021G.C

WOLKITE, ETHIOPIA

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# LIST OF ACRONYMS

AFNOR: Association Francasis deNormalisation Organization

JACAF : Japan Association for International Collaboration of Agriculture and Forestry

EO : essential oil

EGEO: eucalyptus globule essential oil

M pas : Mille Pascal

℃:Degree Celsius

Km: kilo meter

Mm: mille meter

KOH: potassium hydroxide

Kg: Kilogram

ML: Mille Litter

## ABSTRACT

*Essential oils have gained a renewed interest in several areas in world. Though the raw materials to produce essential oil are highly available in our country, yet there is no enough essential oil production which can satisfy the demand throughout the country. The main objective of this project is to extract and characterize essential oil from eucalyptus leaves for pharmaceutical purpose by using Soxhlet extraction method. Raw materials were collected and prepared for the experiment through drying, milling and then the extraction of essential oil from eucalyptus leaves was carried out. The major factors considered were extrication time, particle size and solvent to solid ratio. The effect of time* $(1hr, 2hr, 3hr)$*.Particle size* $(0.5-1mm, 1-1.7mm, and\ 1.7-3.35mm)$ *and Solvent to solid ratio*$(6:1, 7:1\ and\ 8:1)$ *are evaluated. The product was characterized in terms of PH, viscosity, solubility, specific gravity, boiling temperature, evaporation residue, flash point, solubility, acid value, saponification value and iodine number. The maximum product yield was* $3.117\%$ *at the particle size* $(0.5-1mm)$*, extraction time* $180min$ *and solvent to solid ratio* $(7:1)$*. This shows that, increasing extraction time and decreasing particle size increases yield. Characterization value are: PH of* $6.31$*, viscosity of* $3.65$*mpas, flash poit of 50*℃ *, boiling point 174*℃*, evaporation residue 98.648%, soluble in alcohol, specific gravity of*$0.829$*, acid value of 19.635ml/g, saponification value of 14.00ml/g and iodine number 31.725ml/g. It can be concluded that it is possible to extract essential Oil from eucalyptus (Globules) leaves for pharmaceutical purpose using Soxhlet extraction method.*


*Key Words: Eucalyptus Globules, Soxhlet Extraction Method, Eucalyptus Essential Oil*

# 1. CHAPTER ONE

# Introduction

## background

Essential oils are volatile, natural base products, which are found in spices, aromatic and medicinal plants. The Extraction of essential oils is well known from old ages when pure essential oil and crude extract of essential oil bearing plants, herbs and grasses were in use for various medicinal and fragrances, flavors, preservatives and insect repellants purposes (Weiss, 1997; Panda, 2000). Essential oils contain highly volatile substances that are isolated by a physical method or process from plants of a single botanical species. The oils normally bear the name of the plant species from which they are derived. Essential oils are so termed as they are believed to represent the very essence of odor and flavor (Weiss, 1997). Essential oil plants and culinary herbs include a broad range of plant species that are used for their aromatic value as flavorings in foods and beverages and as fragrances in pharmaceutical and industrial products. Essential oils derive from aromatic plants of many genera distributed worldwide (panda, 2011).essential oils are used in the embalming process, in medicine and in purification rituals. There are also over 200 references to aromatics, incense and ointments in the old and new testaments. Research has confirmed centuries of practical use of essential oils, and we now know that the 'fragrant pharmacy' contains compounds with an extremely broad range of biochemical effects. There are about three hundred essential oils in general use today by professional practitioners. Continual bombardment of viral, bacterial, parasitic and fungal contamination occurs in our body. Essential oils are a great benefit to help protect our bodies and homes from this onslaught of pathogens. Immune system needs support and these essential oils can give the required endorsement (pandey and virendra, 2006-2007). The eucalyptus, a native genus from Ethiopia, belongs to myrtaceae family and comprises about 900 species (Weiss, 1997). More than 300 species of this genus contain volatile oils in their leaves. Fewer than 20, within these species, known for their high content of 1, 8-cineole (more than 70%), have been commercially used for the production of essential oils in pharmaceutical and cosmetic industries. Over the past few years, the interest in natural medicine has been increasing in industrialized societies particularly against microbial agents because of the ever growing problem of antibiotic resistance (panda, 2011) the use of EO as functional ingredients in drinks, foods, cosmetics and toiletries is gaining momentum, both for the growing consumers' interest in the ingredients coming

from natural products, and also because of the increasing concern with harmful synthetic chemical additives. Due to their bioactive chemical compounds, volatile oils are certainly promising in view of their use as efficient antimicrobial and therapeutic agents. With the rising significance in the use of EO in both food and pharmaceutical industries, a systematic assessment of the phytochemical extracts has become increasingly essential. Further, EO quality is determined by a complex of agro biological and technological factors, weather and geographical conditions of cultivation, duration of storage & the conditions thereof. There are some conventional ways of EO extraction to produce natural flavor products. The chemo type of the aromatic plant and the equipment used for EO extraction can establish the ratio of components in EO and also the quantity of undesirable impurities (pesticides). Many different types of methods like hydro distillation, microwave-assisted hydro-distillation, steam distillation, cold pressing, solvent extraction, co2 extraction and maceration are available for the extraction of essential oils. These all varieties of method are used in order to produce a good quality or say to obtain an oil of appreciable yield. But from the previous researches and paper reviews, it shows that different types of methods have their different influences on the quality and the yield of essential oils. It can also be seen that the extraction time may vary and the production cost too. Solvent extraction is one such traditional and widely used method for the extraction of essential oils. The solvent extraction method has shown the optimum result particularly for the eucalyptus essential oil. Eucalyptus is an important herbal and aromatic plant and its oil is one of the major essential oil used in the pharmaceutical industry. (www.irjet.net)

## 1.1 Statement of the problem

Eucalyptus trees are much of the constituents of the world's forestry and plantation. In Ethiopia, the increased demand for wood, particularly fuel wood, construction material and the government plan of reforestation have led to a rapid expansion of plantations of fast-growing species of eucalyptus globules, and more than 127,000 hectares of land have been planted in the last decades and usually harvested at the age of 5 - 7 years (kidanu et al., 2004; hunde et al., 2007; and dagne et al., 2000). The use of eucalyptus leaves is not widely known in our country except in some rural area they use only for generation of heat. On the other hand the huge amount of eucalyptus leaves can dispose during deforestation for the construction, furniture work, and for building purpose. This dispose can lead to decrease the soil fertility and Extraction of the eucalyptus oil is not also widely known in

most regions of the country. The raw materials to produce essential oil are highly available in our country, yet there is no enough eucalyptus essential oil production which can satisfy the demand throughout the country.so the project aims to extract essential oil from the disposal of eucalyptus leaves.

## 1.2 Objective

### 1.2.1 General objective

The general objective of this study was to extract and characterization of essential oil from eucalyptus leaves for pharmaceutical purpose.

### 1.2.2 Specific objectives

To investigate the effect of extraction time, particle size and solvent to solid ratio on the yield of extracted oil and to characterize eucalyptus oil.

## 1.3 Significance of the study

This study will contribute to the production and characterization of essential oil from eucalyptus leaves which can be used in pharmacy, cosmetic industries and us an additive in soap industry to improve the performance quality of the product. To establish local industries of essential oil production because of available raw material, land and cheap raw materials. To create jobs for those that will be engaged in planting/cultivating of the plant as well as establishing small scale extraction plants.

## 1.4 Scope of the project

The scope of this project is to produce and characterize essential oil from eucalyptus for pharmaceutical purpose. The study includes raw material collection, the experiment was done to investigated the effect of time, solvent to solid ratio and particle size, finally the product obtain were characterized. Analyzing the best particle size , solvent to solid ratio and extraction time on extraction of essential oil yield from eucalyptus leaves for pharmaceutical purpose. The study conducted at Wolkite University at main (gubireye) campus.

# 2. CHAPTER TWO

# Literature review

**Historical background**

it is estimated that there are 250,000 to 500,000 species of plants on earth. A relatively small percentage (1 to 10%) of these is used as foods by both humans and other animal species. It is possible that even more are used for medicinal purposes (moerman, d. E. 1996). Moerman (1996) reported that while 625 species of plants have been used by various native american groups as food, 2,564 have found use as drugs. According to his calculations, this leaves approximately 18,000 species of plants which were used for neither food nor drugs. Plant oils and extracts have been used for a wide variety of purposes for many thousands of years (jones, 1996). These purposes vary from the use of rosewood and cedar wood in perfumery, to flavoring drinks with lime, fennel or juniper berry oil, and the application of lemongrass oil for the preservation of stored food crops. In particular, the antimicrobial activity of plant oils and extracts has formed the basis of many applications, including raw and processed food preservation, pharmaceuticals, alternative medicine and natural therapies. Since ancient times, herbs and their essential oils have been known for their varying degrees of antimicrobial activity. More recently, medicinal plant extracts were developed and proposed for use in food as natural antimicrobials (kumar, 2010).

## 2.1 Essential oils

An essential oil is a concentrated, hydrophobic liquid containing volatile aroma compounds from plants. Essential oils are also known as volatile, ethereal oils or aetherolea, or simply as the "oil of" the plant from which they were extracted, such as oil of clove. Oil is "essential" in the sense that it carries a distinctive scent, or essence, of the plant (sewanu, 2012).Essential oils are frequently referred to as the “life force” of plants. These "essential" oils are extracted from flowers, leaves, stems, roots, seeds, bark, and fruit rinds. The amount of essential oils found in these plants can be anywhere from 0.01 percent to 10 percent of the total. These oils have potent antimicrobial factors, having wide range of therapeutic constituents. These oils are often used for their flavor and their therapeutic or odoriferous properties, in a wide selection of products such as foods, medicine, and

cosmetics. Only pure oils contain a full spectrum of compounds that cheap imitations simply cannot duplicate (asaad, 2014)

### 2.1.1 Sources of natural essential oils

essential oils are generally derived from one or more plant parts, such as flowers (e.g. Rose, jasmine, carnation, clove, mimosa, rosemary, lavander), leaves (e.g. Eucalyptus , mint, ocimum spp., lemongrass, jamrosa), leaves and stems (e.g. Geranium, patchouli, petitgrain, verbena, cinnamon), bark (e.g. Cinnamon, cassia, canella), wood (e.g. Cedar, sandal, pine), roots (e.g.Angelica, sassafras, vetiver, saussurea, valerian), seeds (e.g. Fennel, coriander, caraway, dill, nutmeg), fruits (bergamot, orange, lemon, juniper), rhizomes (e.g. Ginger, calamus, curcuma, orris) and gums or oleoresin exudations (e.g. Balsam of Peru, myroxylon balsamum, storax, myrrh, benzoin) (trieste, 2008).

## 2.2 Eucalyptus tree

### 2.2.1 Overview of eucalyptus in the world

in world large numbers of aromatic and medicated plants are available in most of the region. In which eucalyptus plant is most common among them. Eucalyptus is recognized today as a natural product which has much to offer in solving global agricultural, environmental and public health problems (chai Tanya et al., 2011). Natural properties of eucalyptus do not have any toxic reactions, so they are helpful in plant protection and management. All the parts of plant like seed, flowers, bark, and leaf can be used to produce high quality product (Pollack, 2010). Eucalyptus is an evergreen tall tree, native to Australia, effectively introduced worldwide, now extensively cultivated in many other countries including Portugal. In Portugal, the planting of eucalyptus globulus occupies about 20% of the forest area and is mainly used by the pulp industries, as source of cellulosic fiber, but some parts of the plant (principally leaves and bark) continue to be rejected by the paper industry (pombal s. Etal., 2014).

**Overview of eucalyptus in Ethiopia**

**History of afforestation**

The history of afforestation with eucalyptus species in Ethiopia goes back as early as the era of emperor menelik ii at the end of the 19th century. The imperial court until that time used to lead

a nomadic life, in which it simply repeated the cycle of exhaustion of wood vegetation around the court, followed by migration to a next place. The living style changed as a result of the afforestation with eucalyptus, and it enabled Addis Ababa to become the permanent capital city. When emperor menelik ii introduced eucalyptus, he tested more than ten varieties, and among them two varieties currently have been cultivated widely, eucalyptus globules (common name in Ethiopia, white eucalyptus) and e. Camaldulensis (red eucalyptus) (Jagger and Pender ., 2000).

**Local names**

Amharic (nech bahir zaf); creole patois (kaliptis); english (turpentine gas, tasmanian blue gum eucalypt, tasmanian blue gum, southern blue gum, fever tree,bluegum eucalyptus, blue gum); japanese (yukari-no-ki); spanish (eucalipto); swahili (mkaratusi); tigrigna (tsaeda-kelamitos); trade name (blue gum) (orwa et al., 2009).

**Botanic description**

eucalyptus globules ssp. Globules is a large to very large evergreen tree, 40-55 (max. 60) m tall, with straight, massive trunk 0.6-2 m in diameter; narrow, irregular crown of large branches and drooping aromatic foliage; crown of open-grown trees broadly rounded or irregular with branches nearly to the ground; bark smoothies, mottled grey, brown, and greenish or bluish, peeling in long strips, at base becoming grey, rough and shaggy, thick and finely furrowed; root system deep and spreading. Leaves alternate, drooping on flattened, yellowish leafstalks of 1.5-4 cm, narrowly lance shaped, 10-30 cm long, 2.5-5 cm wide, mostly curved or sickle shaped, long pointed at tip, short-pointed at base, not toothed on edges, hairless, thick, leathery, with fine, straight veins and vein inside margin, shiny, dark green on both surfaces, aromatic with an odor like that of camphor when crushed (orwa et al., 2009).Flowers 1 (rarely 2-3) at leaf base on very short, flattened stalk or none, more than 5 cm across the very numerous, spreading, white stamens about 12-15 mm long, with odour of camphor; buds top-shaped, 12-15 x 12-25 mm; base (hypanthium) 4 angled, very warty, whitish bloom, with 2 lids. Fruits or seed capsules single at leaf base, broadly top-shaped or rounded, 1.5-5 x 2- 2.5 cm, 4-angled, warty, with whitish, broad, thick, flat or convex disc and 3-5 slits; seeds many and irregularly elliptical, 2-3 mm long, dull black; many small, sterile seeds. The genus eucalyptus was described and named in 1788 by the French botanist l‟héritier. The flowers of the various eucalyptus species are protected by an operculum, hence the generic name, which comes from the Greek words „eu‟ (well), and „calypsos‟ (covered) therefore eucalyptus means well covered (ibid).

**Leaves constituents**

The herbal substance (dried leaves) contains 1-3.5% volatile oil (blaschek et al. 2007, wichtl, 2004). The oil contains as a major constituent 1, 8-cineole in an amount of 54-95% (who monographs, 2002; betts, 2000). The oil derived from fresh leaves consists of 45-75% 1, 8-cineole. Other authors stated a 1, 8-cineole content of 70-85% for the volatile oil (wichtl, 2004). Beside 1, 8-cineole, the oil contains monoterpenes such as cymene, α-pinen, β-pinen and small amounts of myrtenol, pinocarveol, aliphatic aldehyde, flavonoids such as rutin, hyperoside and quercitrin (blaschek et al., 2007). The concentration of α-terpineol was estimated to be 28% (who monographs, 2002). Takasaki et al., 1990 isolated 12 compounds with acylphloroglucinol-monoterpene or -sesquiterpene structures, euglobals from the leaves. The herbal substance also contains gallotannins and smaller amounts of procyanidines, triterpenoids (ursolic acid derivates) and flavonoids as well as phloroglucinol derivates such as euglobals and macrocarpals (wichtl, 2004). The leaves of eucalyptus globules have smaller amounts of tannins than many other eucalyptus species (duke, 1985). Tannin content can depend on the methods of drying leaves (cork & krockenberger, 1991). 1, 8-cineole is also known as eucalyptol. Some authors classified eucalyptol as the active ingredient in eucalyptus oil. Aside from medicinal use, 1, 8-cineole is used as a flavoring agent for lozenges, as a fragrance as well as in cosmetics (clare, 2010).

## 2.3 Eucalyptus oil

The essential oil extract from the eucalyptus leaves which contain compounds with an extremely broad range of biochemical effects as well as odor, flavor and functional properties. Antimicrobial, analgesic and anti-inflammatory properties of e. Citriodora, e. Globulus and e. Teretcorni have been reported from different parts of the world (ramezani et al., 2002; silva, 1997). Eucalyptus has a potential for eucalyptus oil because it mainly uses leaves which do not compete with existing usage. Considering that eucalyptus in Ethiopia (particularly, E. globules) is not currently suffering from diseases or insect pests, it has high potential for organic oil (Jaicaf, 2008).

Uses of Eucalyptus oil

### 2.3.1 Medicinal and Antiseptic characteristics

The cineole-based oil is used as component in pharmaceutical preparations to relieve the symptoms of influenza and colds, in products like cough sweets, lozenges, ointments and

inhalants. Eucalyptus oil has antibacterial effects on pathogenic bacteria in the respiratory tract (Salari et al., 2006). Inhaled eucalyptus oil vapor is a decongestant and treatment for bronchitis. Cineole controls airway mucus hyper secretion and asthma via anti-inflammatory cytokine inhibition (Juergens et al., 2003) (Juergens et al., 2004). Eucalyptus oil also stimulates immune system response by effects on the phagocytic ability of human monocyte derived macrophages (Serafino et al., 2008). Eucalyptus oil also has anti-inflammatory and analgesic qualities as a topically applied liniment ingredient (Göbel et al., 1994; Hong and Shellock, 1991). Eucalyptus oil is also used in personal hygiene products for antimicrobial properties in dental care and soaps. It can also be applied to wounds to prevent infection (Nagata et al., 2008).

### 2.3.2 Repellent and Bio pesticide

Cineole-based eucalyptus oil is used as an insect repellent and bio pesticide. In the U.S., eucalyptus oil was first registered in 1948 as an insecticide and miticide (Flower and Vegetable Oils).

**Flavoring**

Eucalyptus oil is used in flavoring. Cineole-based eucalyptus oil is used as flavoring at low levels (0.002%) in various products, including baked goods, confectionery, meat products and beverages. Eucalyptus oil has antimicrobial activity against a broad range of foodborne human pathogens and food spoilage microorganisms (Zhao and Agboola, 2007). Non-cineole peppermint gum, strawberry gum and lemon ironbark are also used as flavoring.

### 2.3.3 Fragrance

Eucalyptus oil is also used as a fragrance component to impart a fresh and clean aroma in soaps, detergents, lotions and perfumes. It is known for its pungent, intoxicating scent (Kabuba, 2009).

**Industrial**

Research shows that cineole-based eucalyptus oil (5% of mixture) prevents the separation problem with ethanol and petrol fuel blends. Eucalyptus oil also has a respectable octane rating and can be used as a fuel in its own right. However, production costs are currently too high for the oil to be economically viable as a fuel. Phellandrene- and piperitone-based eucalyptus oils have been used in mining to separate sulfide minerals via flotation (Boland et al., 1991).

**Important Physical and Chemical properties of Essential Oils**

The chemical properties of essential oils depend on the natural factors such as type of species, the geographical origin and location of the plant, time of harvesting, plant parts from which the oils are extracted, etc. (Dey, 1996). Essential oils components and percentage are different from oil to oil even for the same botanic plant due to:

**Weather and planting time**

Most of herbs are planted but small amounts could also be wild grown or collected plants. By means of an example with spearmint, the oil percentage from a summer crop is double that from a winter crop. The oil percentage from a given summer could be different from a previous summer even from the same field. The component analysis of the oil could also be different from one season to another.

**Soil elements**

The B-phellanderene percentage increases in marjoram oil with the higher levels of molybdenum manganese, copper, calcium, zinc or iron in the soil.

**Irrigation**

The highest yield of plant material results from increasing the leaf area. For example, this will happen if a basil field is irrigated every 4 days. The essential oil is highest at medium levels of soil moisture.

**Time of harvest**

The peppermint oil yield increases as the herb approaches maturity in the full bloom stage.

**Physical properties**

**Specific gravity**

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

**Optical rotation**

Most essential oils when placed in a beam of polarized light possess the property of rotating the plane of polarization to the right (dextrorotatory), or to the left (laevorotatory).The degree of rotation and the direction are important indicators of purity.

**Refractive index**

When a ray of light passes from a less dense to a more dense medium, it is bent or "refracted" toward the normal. Refract meters offer a rapid and convenient method for the determination of this physical constant.

**Molecular refraction**

The index of refraction of a liquid varies with temperature and the wave length of the light. In order to compare the retroactivities of different liquids, the use of molecular refractivity (molecular refraction) is necessary.

**Solubility**

**Solubility in Alcohol**

Most essential oils are only slightly soluble in water and are miscible with absolute alcohol. The solubility of oil may change with age.

**Solubility in water**

Most of essential oils of commercial interest are steam volatile, reasonably stable to action of heat and practically insoluble in water and hence suitable for processing by steam distillation.

**Boiling range**

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

**Evaporation residue**

An important criterion of purity is the evaporation residue; i.e., the percentage of the oil which is not volatile at 100. It is important to study the odor of oil as it volatilizes during the heating.

**Flash point**

The flash point may prove useful in the valuation of an essential oil. The flash point has value as an indication of adulteration: additions of adulterants such as alcohol and low boiling mineral spirits will realty lower the flash point.

## 2.4 Chemical constituents of essential oils

Essential oil components are divided into terpenoids and non-terpenoids

i. **Non-terpenoids**: This group contains short-chain aliphatic substances, aromatic substances, nitrogenated substances, and substances with sulphur. They are less important than terpenoids in terms of uses and applications.

ii. **Terpenoids:** These are more important commercially and in terms of their properties. Terpenes derive from isoprene units (C5) bonded in a chain. Terpenes are a type of chemical substance found in essential oils, resins, and other aromatic plant substances, (pines, citrus fruits…). They are usually found in monoterpene oils (C15) and diterpenes (C20). They may be aliphatic, cyclic, or aromatic. According to their function group they can be:

Alcohols (menthol, bisabolol) and phenols (timol, carvacrol), Aldehydes (geranial, citral) and cetones (camphor, thuyone) , Esthers (bornile acetate, linalile acetate, methyl salicilate, anti-inflammatory compound similar to aspirin), Ethers (1.8 - cineol) and peroxides (ascaridol) ,Hydrocarbons (limonene, pinene α and β)

**a. Monoterpenic hydrocarbons**

These are the commonest compounds in essential oils, and precursors of the more complex oxidized terpenes. Their names end in –ene. Limonene, for example, is the precursor to the main components of mint essences (Mentha spp, Lamiaceae Family) such as carvone and menthol. Limonene is also found in citric plants and in dill (Anethum graveolens, Apiaceae family). Pinene α and β are also widely present in nature, especially in trementine essence of the Pinus genre (Pinaceae family).

**b. Alcohols**

Alcohols have the hydroxyl group (OH) bonded to a C10 skeleton. Their names end in –ol. They are ighly sought after for their aroma. Linalool, for example, has two forms. R-linalool is found in roses and lavender and is the main component of Mentha arvensis. S-linalool found in lavender oil at > 5% indicates adulteration. Linalool gives tea, thyme, and cardamom leaves their taste. Menthol, another compound found in this group, is responsible for the smell and taste of mint. Mint essence may contain up to 50% of this component. Geranial, from scented geraniums (Pelargonium spp), citronellal, from roses Rosa gallica), borneol from rosemary, and santalol from sandalwood (Santalum album, Santalaceae family).

**c. Aldehydes**

Aldehydes are highly reactive compounds. Their names end in –al. Many of them, such as those found in citrus fruits, match their respective alcohol. For example: geraniol – geranial, and citronelol – citronelal. They are found in abundance in citrus plants, and are responsible for their characteristic smell, particularly the isomers geranial (α citral) and neral (β citral) known as citral in combination. In addition to its characteristic aroma, citral has anti-viral, antimicrobiotic, and sedative properties. But many aldehydes, including citral, cause irritation to the skin and cannot

Be used externally. Another important group is the aromatic aldehydes, such as benzaldehyde, main ingredient of bitter almond oil and cause of their typical aroma.

**d. Phenols**

They are only found in a few species but are very powerful and irritating. The most important are timol and carvacrol, which are found in thyme (Thymus) and oregano (Origanus), both of the Labiatae family. Another important phenol is eugenol, which is found in many species, for example, clove essence. It is both a powerful bactericide and also anaesthetic, and is used in dentistry.

**e. Phenolic Ethers**

These are the main components of species such as celery and parsley (apiol), aniseed (anetol), basil (metilchavicol), and estragon (estragol). Safrol is a component which is used extensively in the perfume industry and is found in the bark of the sassafras tree (Sassafras albidum Lauraceae family).

**f. Ketones**

These are produced by the oxidization of alcohols and are fairly stable molecules. They end in –one. Carvone is found in Mentha spicata. Tuyone -first isolated in Tuya- (Thuja occidentalis Cupressaceae family) and pulegone are fairly toxic and should never be used during pregnancy. Tuyone is found in plants of the Artemisia genus (Artemisia absinthium with which absinthe and vermouth are made), and in salvia (Salvia officinalis). Pulegona was first isolated in Mentha pulegium.

**g. Ethers**

Ethers or monoterpenic oxides are reactive and unstable. One example is bisabolol oxide found in camomile (Matricaria chamomilla). Another common ether is 1.8 –cineol (also known as eucaliptol), which is the main component of eucalyptus oil. It is an expectorant and mucolitic, and the main component of cough medicines. The aroma of eucalyptus oil varies depending on 1.8 –cineol content: the oil with a high content (Eucalptus globulus) is used for medicinal purposes, whereas that with a lower content (Eucaliptus radiata) is used in aromatherapy.

**h. Esther's**

Most esthers are formed from a reaction of a terpenic alcohol with an acetic acid. Their aroma is characteristic of the oils in which they are found. Lavender oil, for example, contains linalool in its Esther, linalile acetate. The relative abundance of both these components is a sign of good

quality. Methyl salicilate, a derivate of salicylic acid and methanol, is an anti-inflammatory compound similar to aspirin and is found in a certain type of heather (Gaultheria procumbens Ericaceous family). It is used externally in liniments (Onyinyechi, 2012).

## 2.5 Methods of Extraction

The following are the methods of extraction of essential oil and their drawbacks.

**Maceration**

Maceration actually creates more of "infused oil" rather than an Essential Oil. Plant matter is soaked in vegetable oil, heated and strained which point it can be used for massage. This method is not desirable because it changes the composition of oil (Kumar, 2010).

**Cold Pressing:**

This method is used to extract the Essential Oils from citrus rinds such as orange, lemon, grapefruit and bergamot. This method involves the simple pressing of the rind at about 120 degrees F to extract the oil. The rinds are separated from the fruit, are ground or chopped and are then pressed. The result is a watery mixture of essential oil and liquid which will separate given time. Little alteration from the oil's original state occurs – these citrus oils retain their bright, fresh, uplifting aromas like that of smelling a wonderfully ripe fruit. The drawback of this method is, oils extracted using this method have a relatively short shelf life (Li Y. et al., 2014).

**Effleurage:**

This is one of the traditional ways of extracting oil from flowers. The process involves layering fat over the flower petals. After the fat has absorbed the essential oils, alcohol is used to separate and extract the oils from the fat. The alcohol is then evaporated and the Essential Oil is collected (Mukhtar et al., 2009).

### 2.5.1 Super Critical CO2 Extraction:

Supercritical CO2 extraction (SCO2) involves carbon dioxide heated to 87 degrees F and pumped through the plant material at around 8,000 psi, under these conditions; the carbon dioxide is likened to a 'dense fog' or vapor. With release of the pressure in either process, the carbon dioxide escapes in its gaseous form, leaving the Essential Oil behind. The usual method of extraction is through steam distillation. After extraction, the properties of a good quality essential

oil should be as close as possible to the "essence" of the original plant. The key to a 'good' essential oil is through low pressure and low temperature processing. High temperatures, rapid processing and the use of solvents alter the molecular structure, will destroy the therapeutic value and alter the fragrance (Singh et al., 2007).

### 2.5.2 Microwave Extraction

Microwave energy is a superior alternative to several thermal applications owing to its efficient volumetric heat production. The volumetric heating or heating of the bulk as opposed to transferring heat from the surface, inwards, is more efficient, uniform and less prone to overkill or supererogation. Controllability is by far the greatest advantage of microwaves over conventional thermal technologies. In processing applications, the ability to instantaneously shut the heat source makes enormous difference to the product quality and hence the production economics. The raw material is heated directly by microwaves and this brings about quality consistency and minimizes the impact on the environment as opposed to using fossil fuels or less efficient, indirect electrical heating systems. Specifically in the essential oil extraction, microwave mediated processes are highly desirable due to their small equipment size (portability) and controllability through mild increments of heating. However, so far the microwave technology has found application in very few industrial bio-processing installations due to the lack of available data on microwave interaction with heterogeneous natural raw materials. The sensing and close control of microwave process is a challenging science and there seems to be insufficient literature in this regard (Kabuba, 2009).

### 2.5.3 Turbo Distillation Extraction:

Turbo distillation is suitable for hard-to-extract or coarse plant material, such as bark, roots, and seeds. In this process, the plants soak in water and steam is circulated through this plant and water mixture. Throughout the entire process, the same water is continually recycled through the plant material. This method allows faster extraction of essential oils from hard-to-extract plant materials (Kumar, 2010). As cited in Kumar (2010), Steam Distillation is a special type of distillation or a separation process for temperature sensitive materials like oils, resins, hydrocarbons, etc. which are insoluble in water and may decompose at their boiling point. The temperature of the steam must be high enough to vaporize the oil present, yet not so high that it destroys the plants or burns the essential oils. The experiment has been carried out for the extraction of oil from Eucalyptus which has high essential oil content. Such Eucalyptus essential oil, which have been

used as perfume and chemical raw materials for a long time, are now been studied as renewable sources of energy. Anitescu et al have studied that ripe fruits of Coriander sativum L. were extracted by steam distillation and by supercritical fluid extraction (SFE), using CO2 in a two-stage separation system. An inexpensive thermal expansion procedure for supercritical fluid delivery has been developed. The identification of components was performed by gas chromatography and mass spectrometry (GC±MS). The percentage composition of the 40 identified compounds was compared with the composition of commercial coriander oil extracted by hydro distillation. Roy Teranishi et al have studied that system combines steam distillation and liquid-liquid extraction to recover volatiles from fats and oils. Oil is pumped in at the top of a spinning-band distillation column, in which the oil is heated to100 and spread to a thin film. As the oil film drops down to the pot, steam, which is introduced at the bottom, travels upward to strip the volatiles from the oil. The steam distillate is extracted in liquid-liquid extractor incorporated in the system, and the extracted water is recycled as steam. Stripped oil in the pot serves as a liquid seal to force steam up the column. The level of the oil in the pot is maintained automatically by an overflow system. Many liters of oil can be pumped through this system to be stripped of volatiles by steam. The volatiles can be isolated easily from the small amount of solvent recycled in the liquid-liquid extractor. Referring to the above literature review, it was found that Steam Distillation method is an appropriate and economical method for extraction of Essential Oil.

### 2.5.4 Steam distillation Method:

Steam distillation is a special type of distillation or a separation process for temperature sensitive materials like oils, resins, hydrocarbons, etc. which are insoluble in water and may decompose at their boiling point. The fundamental nature of steam distillation is that it enables a compound or mixture of compounds to be distilled at a temperature substantially below that of the boiling point(s) of the individual constituent(s). Essential oils contain substances with boiling points up to 200 or higher temperatures. In the presence of steam or boiling water, however, these substances are volatilized at a temperature close to 100 at atmospheric pressure. Fresh, or sometimes dried, botanical material is placed in the plant chamber of the still and the steam is allows to pass through the herb material under pressure which softens the cells and allows the Essential Oil to escape in vapor form. The temperature of the steam must be high enough to vaporize the oil present, yet not so high that it destroys the plants or burns the Essential Oils. Besides the steam tiny droplets

of Essential Oil evaporates and travel through a tube into the still's condensation chamber. Here Essential Oil vapors condense with the steam. The essential oil forms a film on the surface of the water. To separate the Essential Oil from the water, the film is then decanted or skimmed off the top. The remaining water, a byproduct of Distillation is called floral water, distillate, or hydrosol. It retains many of the therapeutic properties of the plant, making it valuable in skin care for facial mists and toners (A solution containing chemicals that can change the color of a photographic print). In certain situations, floral water may be preferable to be pure essential oil, such as when treating a sensitive individual or a child, or when a more diluted treatment is required. Rose hydrosol, for example, is commonly used for its mild antiseptic and soothing properties, as well as its pleasing floral aroma (Kumar, 2010). A number of factors determine the final quality of a steam distilled essential oil. Apart from the plant material, most important are time, temperature and pressure, and the quality of the distillation equipment. Essential oils are very complex products. Each is made up of many, sometimes hundreds, of distinct molecules which come together to form the oil's aroma and therapeutic properties. Some of these molecules are fairly delicate structures which can be altered or destroyed by adverse environmental conditions. So, much like a fine meal is more flavorful when made with patience, most oils benefit from a long, slow 'cooking' process (http://www.bellevuemassagetherapy.com/methods-of-extracting-essential-oils.html). It is possible that longer distillation times may give more complete oil. It is also possible however, that longer distillation time may lead to the accumulation of more artifacts than normal. This may have a curious effect of appearing to improving the odor, as sometimes when materials that have a larger number of components are sniffed, the perception is often of slightly increased sophistication, added fullness and character, and possibly, and extra pleasantness (Kumar,2010).

**Advantages of using Steam Distillation:**

The advantage of Steam Distillation is that it is a relatively cheap process to operate at a basic level and the properties of oils produced by this method are not altered. As steam reduces the boiling point of a particular component of the oil, it never decomposes in this method. This method apart from being economical is also relatively faster than other method (Kumar, 2010).

**Methods for Distillation**

Three are three types of distillation for isolating essential oils from plant materials:

A. Water distillation

B. Water and steam distillation

C. Direct steam distillation

**A. Water Distillation**

In this method, the material is completely immersed in water, which is boiled by applying heat by direct fire, steam jacket, closed steam jacket, closed steam coil or open steam coil. The main characteristic of this process is that there is direct contact between boiling water and plant material. When the still is heated by direct fire, adequate precautions are necessary to prevent the charge from overheating. When a steam jacket or closed steam coil is used, there is less danger of overheating; with open steam coils this danger is avoided. But with open steam, care must be taken to prevent accumulation of condensed water within the still. Therefore, the still should be well insulated. The plant material in the still must be agitated as the water boils, otherwise agglomerations of dense material will settle on the bottom and become thermally degraded. Certain plant materials like cinnamon bark, which are rich in mucilage, must be powdered so that the charge can readily disperse in the water; as the temperature of the water increases, the mucilage will be leached from the ground cinnamon. This greatly increases the viscosity of the water-charge mixture, thereby allowing it to char. Consequently, before any field distillation is done, a small-scale water distillation in glassware should be performed to observe whether any changes take place during the distillation process. From this laboratory trial, the yield of oil from a known weight of the plant material can be determined. The laboratory apparatus recommended for trial distillations is the Clevenger system. During water distillation, all parts of the plant charge must be kept in motion by boiling water; this is possible when the distillation material is charged loosely and remains loose in the boiling water. For this reason only, water distillation possesses one distinct advantage, i.e. that it permits processing of finely powdered material or plant parts that, by contact with live steam, would otherwise form lumps through which the steam cannot penetrate. Other practical advantages of water distillation are that the stills are inexpensive, easy to construct and suitable for field operation. These are still widely used with portable equipment in many countries. The main disadvantage of water distillation is that complete extraction is not possible. Besides, certain esters are partly hydrolyzed and sensitive substances like aldehydes tend to polymerize. Water distillation requires a greater number of stills, more space and more fuel. It demands considerable experience and familiarity with the method. The high-boiling and somewhat water soluble oil constituents cannot be completely vaporized or they require large quantities of

steam. Thus, the process becomes uneconomical. For these reasons, water distillation is used only in cases in which the plant material by its very nature cannot be processed by water and steam distillation or by direct steam distillation.

**Disadvantages of Water Distillation**

Oil components like esters are sensitive to hydrolysis while others like acyclic mono terpene hydrocarbons and aldehydes are susceptible to polymerization (since the pH of water is often reduced during distillation, hydrolytic reactions are facilitated). Oxygenated components such as phenols have a tendency to dissolve in the still water, so their complete removal by distillation is not possible. As water distillation tends to be a small operation (operated by one or two persons), it takes a long time to accumulate much oil, so good quality oil is often mixed with bad quality oil. The distillation process is treated as an art by local distillers, who rarely try to optimize both oil yield and quality. Water distillation is a slower process than either water and steam distillation or direct steam distillation.

**B. 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.

**Advantages of Water and Steam Distillation over Water Distillation**

• Higher oil yield.

• Components of the volatile oil are less susceptible to hydrolysis and polymerization (the control of wetness on the bottom of the still affects hydrolysis, whereas the thermal conductivity of the still walls affects polymerization).

• If refluxing is controlled, then the loss of polar compounds is minimized.

• Oil quality produced by steam and water distillation is more reproducible.

• Steam and water distillation is faster than water distillation, so it is more energy efficient. Many oils are currently produced by steam and water distillation, for example lemongrass is produced in Bhutan with a rural steam and water distillation system.

**Disadvantages of Water and Steam Distillation**

• Due to the low pressure of rising steam, oils of high-boiling range require a greater quantity of steam for vaporization - hence longer hours of distillation.

• The plant material becomes wet, which slows down distillation as the steam has to vaporize the water to allow it to connse further up the still.

• To avoid that the lower plant material resting on the grid becomes waterlogged, a baffle is used to prevent the water from boiling too vigorously and coming in direct contact with the plant material.

**C. Direct Steam Distillation**

As the name suggests, direct steam distillation is the process of distilling plant material with steam generated outside the still in a satellite steam generator generally referred to as a boiler. As in water and steam distillation, the plant material is supported on a perforated grid above the steam inlet. A real advantage of satellite steam generation is that the amount of steam can be readily controlled. Because steam is generated in a satellite boiler, the plant material is heated no higher than 100 and, consequently, it should not undergo thermal degradation. Steam distillation is the most widely accepted process for the production of essential oils on large scale. Throughout the flavor and fragrance supply business, it is a standard practice. An obvious drawback to steam distillation is the much higher capital expenditure needed to build such a facility. In some situations, such as the large-scale production of low-cost oils (e.g. rosemary, Chinese cedar wood, lemongrass, lit sea cubeb, spike lavender, eucalyptus, citronella, corn mint), the world market prices of the oils are barely high enough to justify their production by steam distillation without amortizing the capital expenditure required to build the facility over a period of 10 years or more.

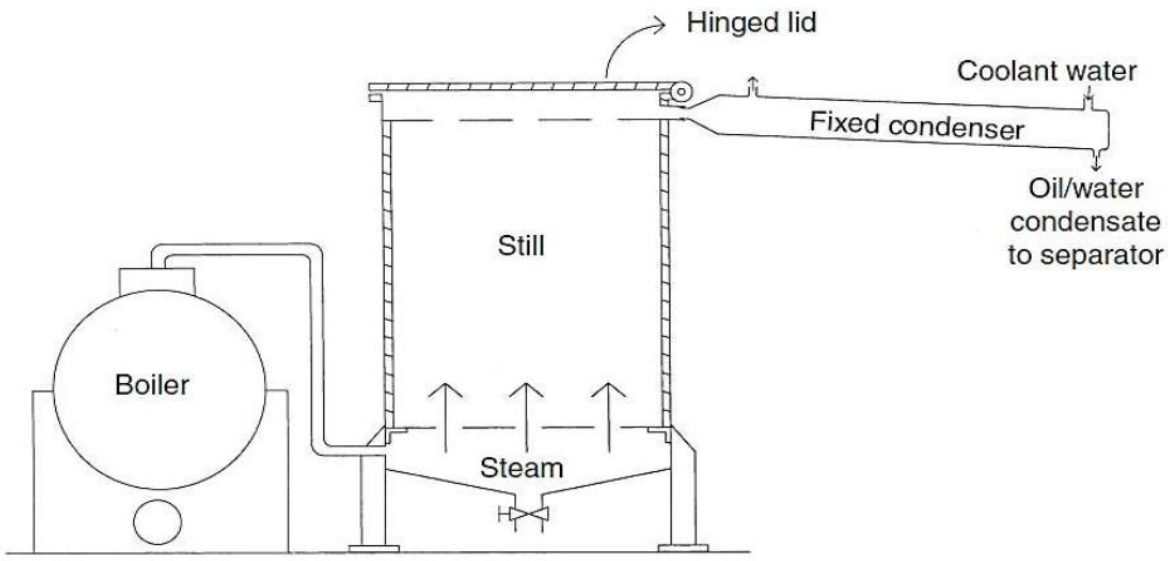



Figure 1 Steam distillation process with a separate boiler for extraction of Eucalyptus globules Essential oil (Coppen J. J, 2003)

**Components of the extraction plant are:**

**Boiler**

Generate steam by boiling water which can facilitate the extraction process by entering to the extraction chamber. For this experimental work the boiler was operates at atmospheric pressure and constant temperature.

**Extraction chamber**

This served primary as a container and as a vessel in which the steam contacts the plant material and vaporize its oils. The plant material was pack in to the extraction chamber so that distillation commence. Proper charging was very important otherwise the steam channels through the plant material and low yield results. The first load was contact to the set-up and establishes the procedure determines processing parameters.

**Condenser**

A coil flow condenser was used to convert all the steam and the accompanying oil vapors from the top of the extraction chamber in to liquid. Water was feed to the overhead reservoir and this permitted the water to trickle over the entire length of the condenser tubes. It was noted that the condenser tubes all sloped down ward slightly, to ensure proper drainage of the condenser oil and steam. The cooling medium used in this device was cooling water drawn from a running tap.

**Separator**

Essential oil extracts and water condensate were known to have different densities and also from an immiscible two liquid phases mixture at low room temperature conditions. The separation of essential oils from the condensation hence utilizes the density and immiscibility advantage for the two be isolated each other. This phenomena was the oil extract float on the water layer due to being dense than water. In separation the water from the oil, the water layer is carefully run out from the bottom of decanter by opening the tap until its meniscus was just at the calibration mark. The contents that remained inside the decanter are the oil layer and the water between the tap bridge and the bottom of the calibration mark.

**Advantages of Direct Steam Distillation**

• Amount of steam can be readily controlled. No thermal decomposition of oil constituents.

• Most widely accepted process for large-scale oil production, superior to the other two processes.

**Disadvantage of Direct Steam Distillation**

• Much higher capital expenditure needed to establish this activity than for the other two processes.

## 2.6 Solvent Extraction

Solvent extraction is adapted in producing essential oils generated by some flowers (Rose, Violetta and Geranium), gums, Eucalyptus Leaves and resins. The raw material is placed in a glass, vessel and soaked with a suitable solvent (petroleum, hexane, ether or benzene). After the extraction, the solids are separated from the liquid mixture. The latter is heated so that the more volatile essential oils can be evaporated to be subsequently condensed. Alternatively, if the solvent is more volatile, such as ethanol, it could then be vaporized leaving behind the essential oils (Ndou, 1986). As solvent extraction uses very little heat, it is found to be advantageous in producing essential oils with whole fragrances that would otherwise be destroyed or altered during steam distillation. Therefore, this extraction technique can be used to extract essential oils from very delicate plants to produce higher amounts of essential oils at lower costs (Ndou, 1986). However, some disadvantages associated with the solvent extraction technique. Solvent residues often contaminate the product causing side effects which make the use of essential oil undesirable for skin applications but could still be fine for fragrances or perfumes (Ndou, 1986).

### 2.6.1 Solvent Extraction of Eucalyptus Leaves

**Selection of Solvent**

The most important factor for the success of the extraction process is the quality of the solvent employed. The ideal solvent should possess the following properties (Ndou, 1986).

- It should completely and quickly dissolve all the materials being extracted.
- The solvent must not dissolve in water.
- The solvent must be chemically inert.
- The solvent should have a uniform boiling point when evaporated and does not leave any residue.
- The solvent should be low priced.

But the ideal solvent, which fulfills these entire requirements, does not exist. Considering every feature, highly purified petroleum ether appears to be the most suitable. There are different solvents used for the extraction of eucalyptus leaves; benzene, hexane, diethyl ether and petroleum ether are some of the solvents. Benzene has a high boiling point (80.1$^{o}$C) resulting high amount of benzene in the last product. Besides, benzene is highly flammable. Therefore, benzene is not preferable solvent for the extraction of eucalyptus essential oil. Diethyl ether on the other hand has a low boiling point resulting in a lot of solvent lose and is highly flammable as well as costly solvent; hence diethyl ether is not preferable solvent option for the extraction of back cumin oil. Petroleum ether is the best solvent, unfortunately it is very costly and is not found easily as a result it will not be an option as solvent for the extraction of black cumin. Hexane which has a moderate boiling point (69$^{o}$C) is relatively cheap and safe compared to diethyl ether and benzene; hence hexane is a preferred solvent for the extraction of eucalyptus oils.

### 2.6.2 Soxhlet apparatus

Soxhlet extraction is one of the most common laboratory procedures for solid liquid extraction, it has been standard technique for over a century. And at present, it is still the main reference to which the performance of other extraction methods is compared. Soxhlet extraction has some attractive advantages. Firstly the sample is repeatedly brought in to contact with fresh solvent, which facilitates displacement of the transfer equilibrium. Secondly, no filtration is required after the extraction step and the sample throughput can be increased by simultaneous extraction in parallel since the basic equipment is inexpensive. Soxhlet extraction is selective because interfering compounds (which could hinder good separation of interested analyses during extraction) are also extracted. Although the hardware required for a Soxhlet extraction is relatively simple, extreme care

must be taken to prevent contaminants in the extract and to minimize losses during sample transfer and solvent exchange.(9) Normally a solid material containing some of the desired compounds was placed inside a cellulose or ceramic thimble, which was loaded into the main chamber of the Soxhlet extractor. The extractor was connected to a flask containing the solvent and a condenser was placed on top of the extractor. The solvent was heated to reflux, the vapor passed through a bypass arm to reach the condenser which it condensed and dripped down into the thimble housing the solid material. The extractor containing the solid material slowly filled with warm solvent and some of the desired compounds would then dissolve into the warm solvent. Once the solvent level in the extractor reached the top of siphon arm, the solvent and the extract were siphoned back into the lower flask. The completed extraction produced a high volume; dilute solution which usually needed to be concentrated by a rotary evaporator. The non-soluble portion of the extracted solid remained in the thimble and was usually discarded.

# 3. CHAPTER THREE

## 3.1 MATERIALS AND METHODS

**STUDY AREA**

The experimental work was done in the laboratory of wolkite University college of engineering and Technology, department of Chemical Engineering.

### 3.1.1 Material and Equipment

The materials and equipment needed for the experiment work were plastic bag, eucalyptus leaves, oven, rapid grinder, sieve, electronic balance, soxhlet extractor, flask, black bottle, filter, heating mantle, thermometer, pH meter, viscometer, Pycnometer, spectrometer, ,simple distillation set up.

### 3.1.2 Chemical and reagent

Chemicals needed for extraction and characterization of eucalyptus essential oil were; potassium hydroxide (85%), ethanol (97%), oxalic acid (99.5%), sodium hydroxide (99.8%), phenolphthalein, chloroform (99.8%), and buffer solution were used for saponification value determination, for solvent of eucalyptus oil, as titrated of unreacted (KOH), for titration in acid value determination, indicator, for solvent of eucalyptus oil, and for standardized pH electrode respectively, iodine bromide solution, potassium iodide , n-hexane, Anhydrous sodium sulfate, and
sodium thiosulphate.

### 3.1.3 Experimental design

In this project, three factors which are solvent to solid ratio, time and particle size with three levels were considered. The number of experiments = (level) $^{factor}$ *K, where K =replication constant. Total number of experiments=$3^3$*1=27. The particle sizes based on wolkite university food engineering laboratory sieves analysis.

Table 1 Experimental Design

| | | Solvent to solid ratio(ml/gram) | | |
|---|---|---|---|---|
| Time(hour) | Particle size (mm) | 6:1 | 7:1 | 8:1 |
| 1 | 0.5-1 | | | |

| | | | | |
|---|---|---|---|---|
| | 1-1.7 | | | |
| | 1.7-3.35 | | | |
| 2 | 0.5-1 | | | |
| | 1-1.7 | | | |
| | 1.7-3.35 | | | |
| 3 | 0.5-1 | | | |
| | 1-1.7 | | | |
| | 1.7-3.35 | | | |

## 3.2 Method

### Block flow diagram

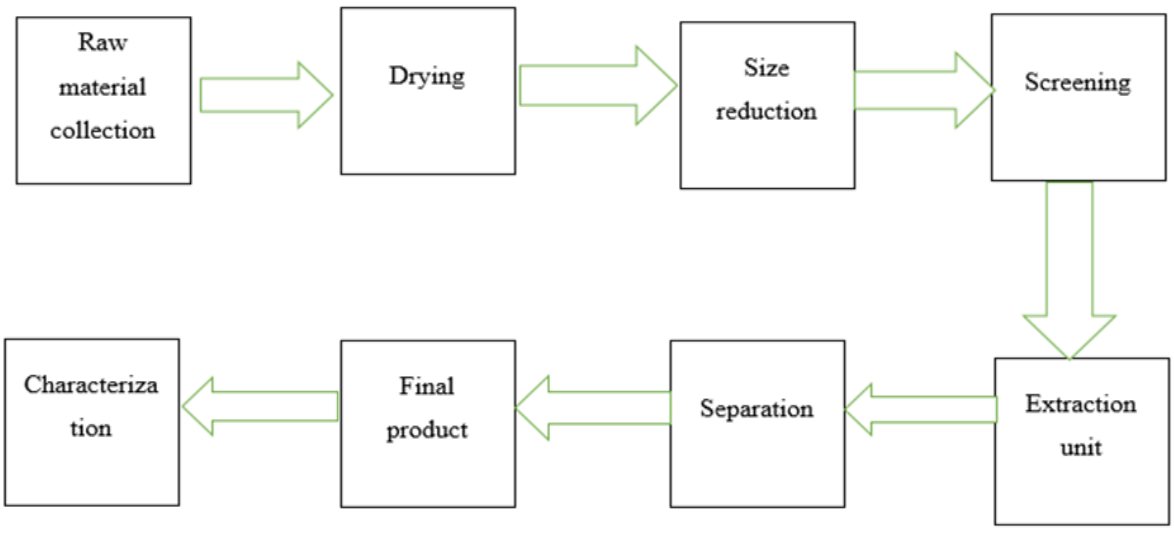



Figure 2 block flow diagram of EGEO extraction process

### 3.2.1 Raw Material preparation

Eucalyptus leaves were collect from emdibir around Mariam church. The family of eucalyptus was Myrtaceae and the botanical name eucalyptus E. globulus. The impurities were removed. The eucalyptus leaves were collected as fresh leaves then partial sun drying was used to dry the leaves.

**Size reduction and Sieve analysis of the leaves**

The moisture was removed by partial sun drying. The dried Eucalyptus leaves were crushed in rapid grinder. The sample was sieved using set of sieves sizes arranged in descending order size of 3.35-2.5mm,2.5-1.4mm,1.4mm-0.5mm to obtain particular sizes of 0.5 -1 mm, 1- 1.7 mm, and 1.7 - 3.35 mm. This was aimed to investigate the effect of particles size on yield. Particular size range was selected because literature reveled that to have a higher yield of oil particle size should be less than 5mm and higher than 0.2mm (Henry, 1983).

**Essential Oil Extraction Experimental procedure**

Experimental work was conducted using Soxhlet extractions methods. In solvent extraction process, hexane was used as a solvent. Eucalyptus leaves sample were placed in the thimble and was inserted in the center of the extractor. The distiller in Soxhlet unit was heated to at the temperature 70℃. The extraction was done by varying the extraction time.one, two and three hours was used for the experiment. The experiment was repeated by placing the same 40 grams of sample into the thimble with solvent to solid ratio (6:1, 7:1 and 8:1) and the particle size range of (3.35-1.7mm, 1.7-1mm and 1-0.5mm). The weight of oil extracted was determined for each run time, particle size and solvent to solid ratio. At the end of the extraction, the resulting mixture (essential oil and hexane) containing the oil was heated to recover solvent from the oil. Separation of oil from hexane was carried out using simple distillation and water bath. The resulting extracts, obtained under different operating conditions were separated by evaporating the solvents using simple distillation in which the setup was established in the laboratory under specific temperature of boiling points of the solvent of hexane.

**Sample Analysis**

**Moisture Content Determination**

70 g, 86g, and 92g of the eucalyptus leaves was weighed and dried in an oven at 105°C and the weight was measured every 3 hrs. The procedure was done repeatedly until a constant weight was obtained. The percentage moisture in the leaves was calculated using the following formula:

$$\mathbf{moisture}\% = \frac{\mathbf{w1-w2}}{\mathbf{w1}} * \mathbf{100}$$ Equation (3.1.) Where w1 is the weight of the sample before drying and w2 is the weight after drying.

### 3.2.2 Determination of the yield of eucalyptus oil

The extraction of eucalyptus essential oil was conducted by using Soxhlet extraction method with different extraction time, particle size and solvent to solid ratio. For this project work, eucalyptus leaves were used at three different extraction time intervals; such as 60min, 120 min and 180min with different particle size ((mm) 3.35:1.7, 1.7:1, and 1:0.5) and solvent to solid ratio(ml/g) 6:1, 7:1, and 8:1with one replication to get maximum yield. The percentage yield oil was calculated using the formula below.

$$\%\ \textbf{age yeild of oil} = \frac{\textbf{weight of oil}}{\textbf{weight of eucalyptus}} * \mathbf{100} \qquad \textbf{Equation (3. 2)}$$

## 3.3 Characterization of eucalyptus oil

### 3.3.1 Physicochemical properties of eucalyptus oil

Physical characterization is determining the physical properties of the extracted oil. Some of these properties are specific gravity, refractive index, flash point, evaporation residue etc... Chemical properties of eucalyptus oil are such as acid value, saponification value, and iodine number. Physicochemical properties were used to determine the quality of EGEO extracted. All the parameters are determined according to the method of European Pharmacopeia (European Pharmacopoeia Commission, 2001).

### 3.3.2 Determination of pH Value

3g of the eucalyptus oil was poured into a clean dry 25ml beaker next 13ml of hot distilled water was added in to the beaker and stirred slowly. It was then cooled in a cold-water bath to 25°C. The pH electrode was standardized with buffer solution then the electrode was immersed into the sample finally the pH value was read and record.

### 3.3.3 Specific gravity determination

A tub of known weight (W) filled was first with essential oil and then with water and the respectively weight $w_1$ and $w_2$ was determined. Then, the specific gravity was calculated using the following formula**:**

$$\textbf{specific gravity} = \frac{\mathbf{w1-w}}{\mathbf{w2-w}} \qquad \textbf{Equation ( 3.3)}$$

### 3.3.4 Determination of viscosity of the eucalyptus oil

35m1 of eucalyptus oil was poured into a test tube and a viscometer was used to measure the viscosity at a temperature of 19℃.

### 3.3.5 Determination of boiling temperature of the eucalyptus oil

25 ml of eucalyptus oil was poured in to Borosilicate glass and a thermometer was inserted and placed on a heating mantle or heater, it was observed that the oil in the Borosilicate started circulating leading to boiling of oil and read temperature on thermometer then recorded.

### 3.3.6 Evaporation residue of eucalyptus oil

37 grams of eucalyptus oil was poured into Borosilicate glass, put it on heater then thermometer was inserted into the Borosilicate glass until it reads temperature of 100℃ to determine volatile matter.

$$\% \textbf{ age of the oil} = \frac{w2}{w1} * 100\% \quad \text{Equation(3.4)}$$

Where: $W_1$ is weight before evaporation and $W_2$ is weight after evaporation

### 3.3.7 Flash point of eucalyptus oil

The flash point of eucalyptus oil was measure by adding 25 milliliter of sample in to Borosilicate glass and then put it on heater next thermometer was immersed in to the glass containing the sample to read the minimum temperature of first flame. Finally the temperature was read and then recorded.

### 3.3.8 Solubility of eucalyptus oil

Solubility of eucalyptus oil has been seen by adding 2 grams of sample into 10 milliliter of alcohol and water.

### 3.3.9 Acid value determinations

Acid value is the mass of potassium hydroxide (KOH) in g that is requisite to neutralize one g of chemical substance. The acid number is a measure of the amount of carboxylic acid groups in a chemical compound. The acid number is used to quantify the amount of acid present, in EGEO sample. (1g) of eucalyptus oil was accurately weighted and dissolved in 10 ml of 95 % ethanol and 2-3 drops of phenolphthalein indicator was added. The free acid was then titrated with standard 0.1 Normality of aqueous sodium hydroxide solutions by adding the alkali drop-wise at a uniform

rate of about 30 drops per minute. The content of the flask was continuously agitated. The primary manifestation of the red coloration that did not fade within 10 seconds was considered the end point. Afterward, the acid value is determined using the following equations (Boukhatem *et al*., 2014):

$$\textbf{acid value} = \frac{\textbf{5.61}*(\textbf{nomber of ml of 0.1 N NaOH})}{\textbf{weigh of sample in gram}} \qquad \text{Equation(3.5)}$$

Where N is normality

### 3.3.10 Saponification value determination

Saponification value represents the number of grams of potassium hydroxide or sodium hydroxide required to saponify 1 g of EGEO under the condition specified. Saponification value was calculated by standard procedure. EGEO (1g) was accurately weighed and dissolved in 10 ml of ethanol and then 10 ml of 2.5 Normality KOH solutions was added. This procedure was performed together with blank experiment which was also performed omitting the oil. The mixture was refluxed for two hours then cooled. The unreacted KOH was titrated with standard 0.5 Normality of oxalic acid by adding 2-3 drops of phenolphthalein indicator until became colorless. After that, the saponification value was determined using the following equation:

$$\textbf{saponification value} = \frac{\textbf{56}(\textbf{v1}-\textbf{v2})}{\textbf{2}*\textbf{w}} \qquad \text{Equation (3. 6)}$$

Where W is the weight of oil, V1 is the volume of 0.5 Normality of oxalic acid for blank; V2 is the volume of 0.5 Normality of oxalic acid for sample (Boukhatem *et al*., 2014).

### 3.3.11 Iodine number determination

EGEO (0.1g) was dissolved in 10 ml of chloroform. Then 25 ml of iodobromide solution was added and allowed to stand for 30 minutes in dark. Again 30 ml of 1 N potassium iodide and 100 ml of distilled water were added and the liberated iodine was titrated with 0.1 normality solution of sodium thiosulphate with constant shaking. When iodine color became quite pale, 1 ml of 1 % starch solution was added and the titration was continued until the blue color was discharged. A blank test was also carried out parallel under identical condition. The iodine number was determined using the formula: $\textbf{Iodine number} = \textbf{1.269} * \frac{(\textbf{v 1}-\textbf{v2})}{\textbf{w}}$ **Equation (3. 7)**

Where: W is the weight of sample, v1 is the number of ml of thiosulphate consumed by the blank; and V2 is the number of ml of thiosulphate consumed by the test sample.

# 4. CHAPTER FOUR

## 4.1 Results and discussion

### 4.1.1 Determination of Moisture Contents

The fresh leaves was collected on, July 20 after drying by taking 70g, 86g and 92g the moisture content of the sample was obtained in the following table.

Table 2 Moisture content determination of the dry eucalyptus leaves

| | Time for drying (hour) | | | | | | | Moisture content (%) |
|---|---|---|---|---|---|---|---|---|
| Sample weight (g) | 0 | 2 | 4 | 6 | 8 | 10 | 12 | |
| | 70 | 69.59 | 69.56 | 69.54 | 69.49 | 69.47 | 69.47 | 0.757 |
| | 86 | 85.58 | 85.16 | 85.15 | 85.12 | 85.11 | 85.11 | 1.034 |
| | 92 | 91.43 | 90.86 | 90.85 | 90.81 | 90.80 | 90.80 | 1.304 |

### 4.1.2 Determination of the yield of eucalyptus oil

The extraction of eucalyptus essential oil was conducted by using Soxhlet extraction method with different extraction time, particle size and solvent to solid ratio. For this project work, eucalyptus leaves were used at three different extraction time intervals; such as 60min, 120 min and 180min with different particle size ((mm) 3.35:1.7, 1.7:1, and 1:0.5) and solvent to solid ratio(ml/g) 6:1, 7:1, and 8:1. . The percentage yield of oil was calculated by the equation (3.2) in the methodology and the result is show in the table below**:**

Table 3 Yield of extracted essential oil by percentage

| | | Solvent to solid ratio(ml/gm) | | |
|---|---|---|---|---|
| Time(min) | Particle size(mm) | 6:1 | 7:1 | 8:1 |
| 60 | 2.525 (3.35-1.7) | 0.821 | 1.952 | 1.761 |
| | 1.35 (1.7-1) | 1.217 | 2.313 | 2.210 |

| | | | | |
|---|---|---|---|---|
| | 0.75 (1-0.5) | 1.923 | 2.531 | 2.335 |
| 120 | 2.525(3.35-1.7) | 1.871 | 2.725 | 2.535 |
| | 1.35 (1.7-1) | 2.005 | 3.001 | 2.855 |
| | 0.75 (1-0.5) | 2.153 | 3.125 | 3.11 |
| 180 | 2.525 (3.35-1.7) | 1.699 | 2.710 | 2.553 |
| | 1.35 (1.7-1) | 2.010 | 2.910 | 2.70 |
| | 0.75 (1-0.5) | 2.154 | 3.117 | 3.10 |

Table 4 the weight of essential oil in gram. The 40-gram eucalyptus leaves are used as sample.

| | | Solvent to solid ratio(ml/gm) | | |
|---|---|---|---|---|
| Time(min) | Particle size(mm) | 6:1 | 7:1 | 8:1 |
| 60 | 2.525 (3.35-1.7) | 0.25 | 0.586 | 0.585 |
| | 1.35 (1.7-1) | 0.364 | 0.694 | 0.695 |
| | 0.75 (1-0.5) | 0.58 | 0.759 | 0.760 |
| 120 | 2.525(3.35-1.7) | 0.561 | 0.82 | 0.821 |
| | 1.35 (1.7-1) | 0.602 | 0.900 | 0.901 |
| | 0.75 (1-0.5) | 0.646 | 0.940 | 0.94 |
| 180 | 2.525 (3.35-1.7) | 0.570 | 0.82 | 0.82 |
| | 1.35 (1.7-1) | 0.603 | 0.902 | 0.903 |
| | 0.75 (1-0.5) | 0.650 | 0.938 | 0.936 |

## 4.2 Effect of Process Parameters on Percentage Oil Yield

### 4.2.1 Effect of Particle Size and Solvent to Solid Ratio on Percentage Oil Yield

Figure 4.1 shows the effect of particle sizes and solvent-solid ratio on yield of eucalyptus of essential oil. There is an increase in the oil yield as the particle size decreased and thus an increase in the particle size of eucalyptus leaves results in a drop in oil yield. The reason is that larger particles have smaller surface area of contact and larger distance to solvent entrance and oil diffusion in comparison to smaller particle using n-hexane solvent (Ndou, 1986).The maximum oil yield was 2.95% at solvent to solid ratio was 7:1 and the particle size is 1-0.5 mm or average particle size is 0.75mm. This implies that, the percentage oil yield increases with an increase in solvent to solid ratio (Yesenofski, 2005).

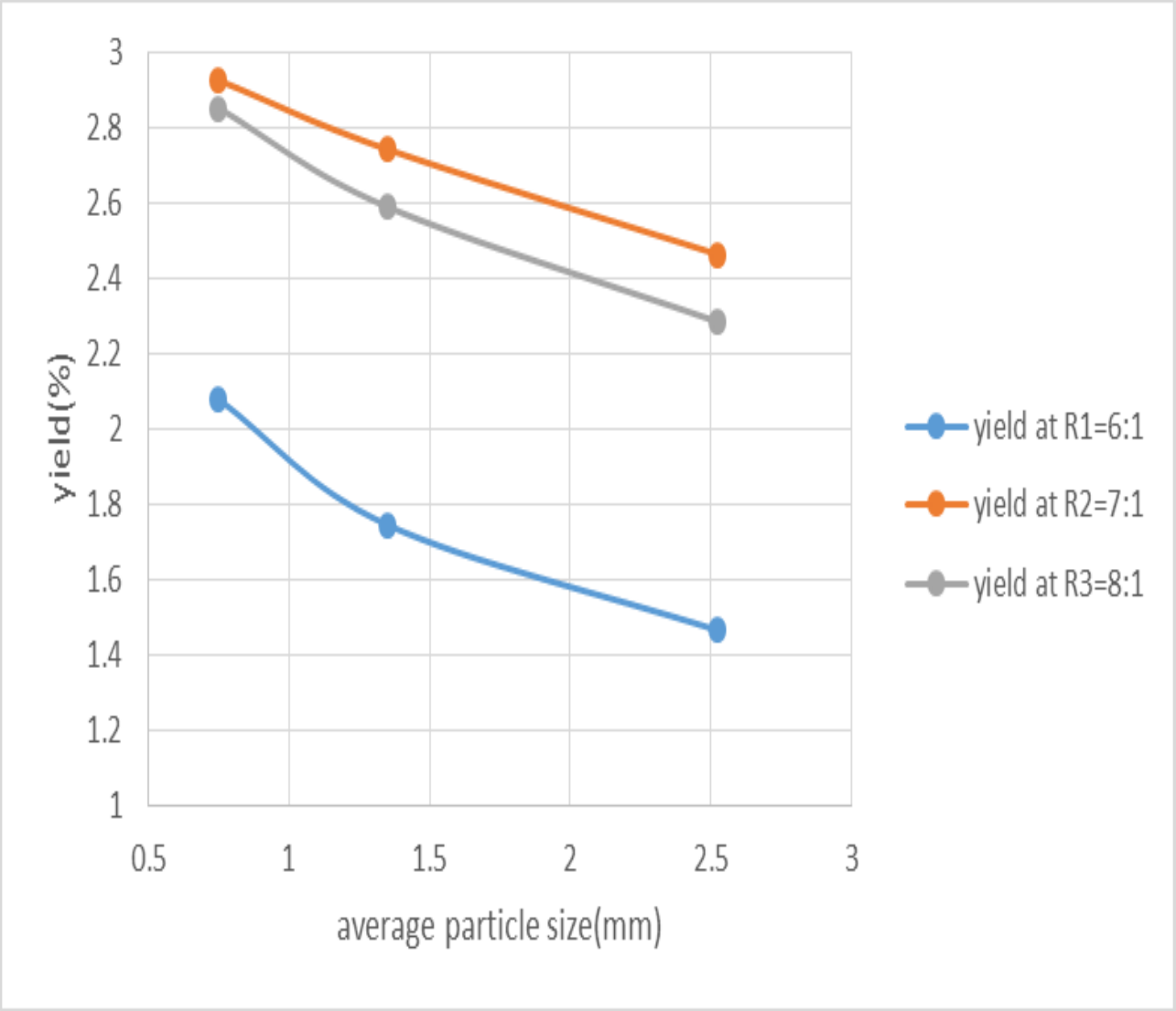


Figure 3 the effect of particle size and solvent to solid ratio on essential oil yield

### 4.2.2 Effect of Extraction Time and solvent to solid ratio on Percentage Oil Yield

The effect of time and solvent-solid ratios on the extraction yield of the essential oil is illustrated in Figure 4.2. As shown, as time increases the percentage yield increases up to 180 minutes, the maximum percentage yield of oil was obtained at 180 minute and 7:1, but as time increased further from 180 minutes the yield slightly decreases and it continues constantly (Yesenofski, 2005).

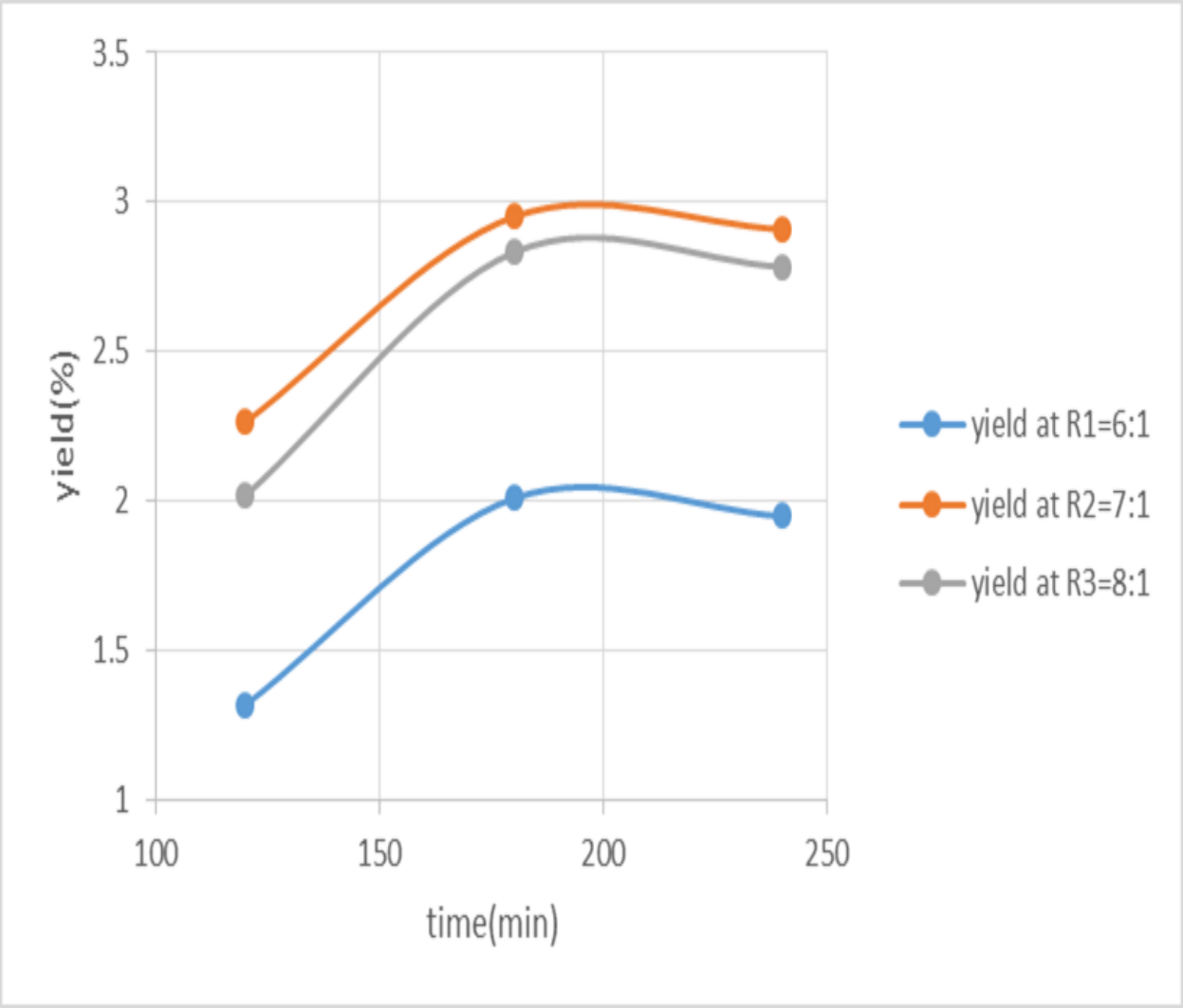



Figure 4 Effect of time and solvent to solid ratio on oil yield

### 4.1.3 Effect of Extraction Time and Particle Size Oil Yield

As shown in figure 4.3, the oil yield decreases slightly linearly with an increase in particle size. And also, the yield increased as the extraction time increases (Jaicaf, 2008).

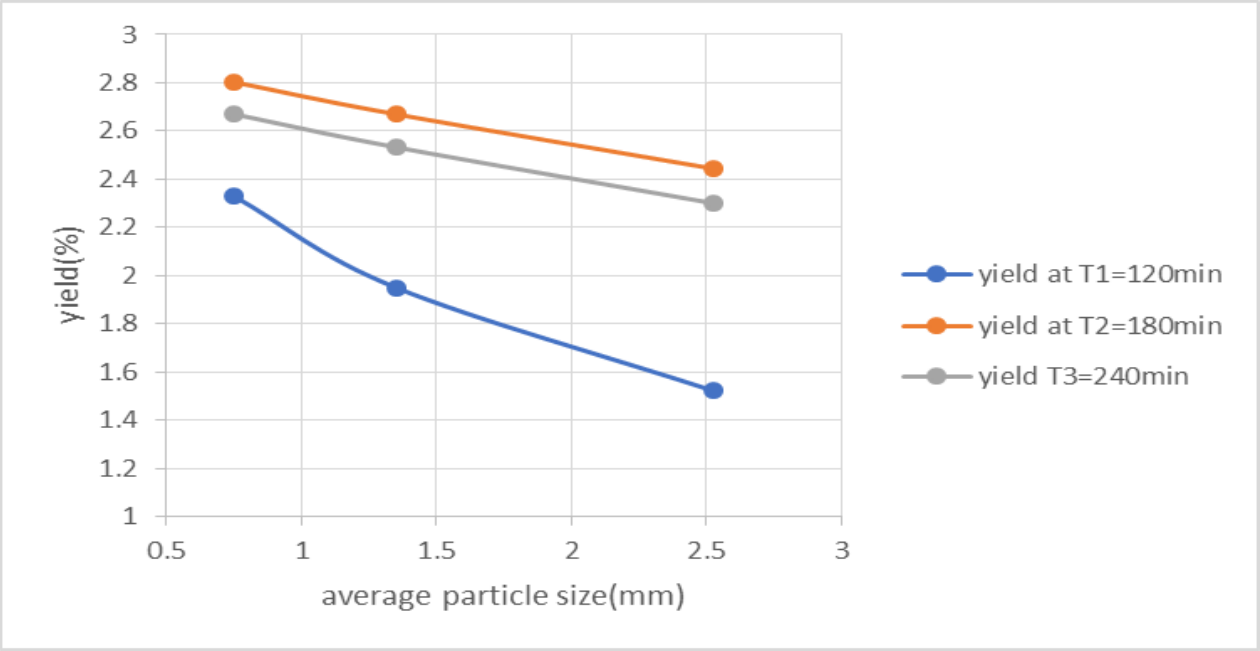



Figure 5 Effect of particle size and extraction time on the yield of oil

## 4.3 Characterization of Essential Oil

### 4.3.1 PH value

The pH of eucalyptus globule's essential oil was measured by pH meter in thermal and mass transfer laboratory of Chemical Engineering Department. Its value was recorded as 6.31.

### 4.3.2 Specific Gravity

Weight of tube (Pycnometer) (W) at 25 = 23.4 gram, Weight of with eucalyptus oil (W1) at 25 = 45.212 gram,Weight of Pycnometer with eucalyptus water (W2) at 25 = 48.512 gram

$$\text{Specific gravity} = \frac{W1-W}{W2-W1}$$

$$\text{Specific gravity} = \frac{45.21-23.4}{48.512-23.4} = 0.829$$

### 4.3.3 Viscosity

Viscosity of oil was measured by viscometer at 19℃ in food engineering laboratory. The value was 3.65 mili Pascal second (mpas).

### 4.3.4 Solubility of essential oil

The eucalyptus oil was soluble in alcohol and insoluble in water.

### 4.3.5 Evaporation residue of eucalyptus (globules) oil

The percentage of eucalyptus oil which does not evaporate at 100℃ was 98.65%. Mass of eucalyptus oil that evaporates at a temperature of 100℃ is 1.35%.

% age of oil not volatile at 100°C $=\frac{W2}{W1} * 100\% = \frac{36.5}{37} * 100\%$= 98.65%

### 4.3.6 Flash point of eucalyptus (globules) oil

Flash point of eucalyptus oil was measured as 50℃.

### 4.3.7 Boiling point of eucalyptus (globules) oil

The boiling point of eucalyptus oil was measure by the procedure described on the methodology. Its value was 174℃.

Table 5 Values and unit of physical properties of eucalyptus oil.

| Physical properties | Results | unit | AFNOR standards |
|---|---|---|---|
| pH at 25 | 6.31 | - | 6.5-7 |
| Specific gravity at 25 | 0.829 | - | 0.81- 0.84 |
| Dynamic Viscosity at 19 | 3.65 | mpas | 3-9(mpas) |
| Flash point | 50 | ℃ | 49 |
| Boiling point | 174 | ℃ | 176 |
| Evaporation residue | 98.648 | % | - |
| Solubility in alcohol | soluble | - | Soluble in alcohol |
| Solubility in water | insoluble | - | Insoluble in water |

Quality Evaluation of the of eucalyptus oil

### 4.3.8 Acid value determination

The acid value of oil was determined as follows:

A.V = $\frac{\mathbf{56.1(volume\ of\ alcoholic\ sodium\ hydroxide\ solution*normality}}{\mathbf{mass\ of\ sample}}$ = $\frac{\mathbf{56.1(7ml*0.1)}}{\mathbf{2gram}}$ = **19.635**ml/gram

### 4.3.9 Saponification value determination

The saponification of eucalyptus oil was calculated as follow S.V =56($\frac{\boldsymbol{v1-v2}}{\mathbf{2*w}}$)

=56$\left(\frac{\mathbf{(11.5-11)ml}}{\mathbf{2*1gram}}\right)$= 14ml/gram

### 4.3.10 Iodine number determination

The iodine number calculated as I.N = 1.269 ($\frac{\mathbf{v1-v2}}{\mathbf{w}}$) = $\frac{\mathbf{1.269(10.5-8)}}{\mathbf{0.1}}$ =31.725ml/gram. Chemical properties of eucalyptus oil such as acid value, saponification value and iodine number are summarized in table 4.2.

Table 6 chemical properties of eucalyptus oil extracted using soxhlet.

| Chemical properties | **V**alue | Unit |
|---|---|---|
| Acid value | 19.635 | ml/g |
| Saponification value | 14.00 | ml/g |
| iodine number | 31.725 | ml/g |

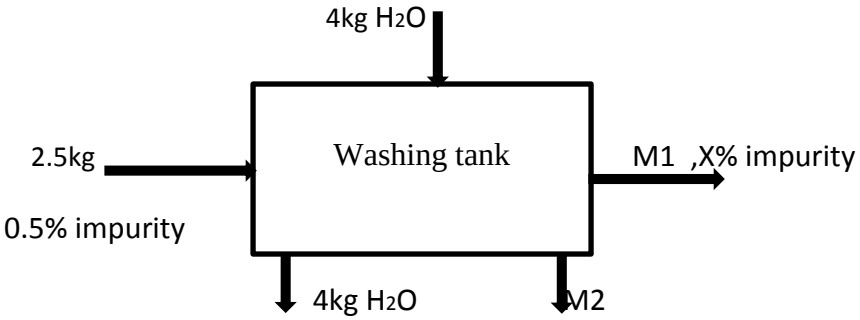
# 5. CHAPTER FIVE

## 5.1 Material balance and energy balance

Material quantities, as they pass through processing operations, can be described by material balances. Such balances are statements on the conservation of mass. Similarly, energy quantities can be described by energy balances, which are statements on the conservation of energy. If there is no accumulation, what goes into a process must come out. This is true for batch operation. It is equally true for continuous operation over any chosen time interval. Material and energy balances are very important in an industry. Material balances are fundamental to the control of processing, particularly in the control of yields of the products**.**

## 5.2 Material Balance in laboratory

**Material balance on washing tank**




Mass in + generation = mass out +consumption +accumulation

Mass in = mass out

Assume the efficiency of washer is 99.5% in removing the impurity from fresh eucalyptus leaves. Over all material balance on washer

$2.5kg = m_1+m_2$

Impurity removed ($m_2$) = (2.5×0.995×0.005) kg

= 12.44*10^-3kg

The impurity in the fresh eucalyptus leaves = 0.005×2.5kg

= 0.0125kg

So, the impurity remains after washing = 0.0125kg – 0.001244kg

= 0.000063kg

Substitute m2 to equation above

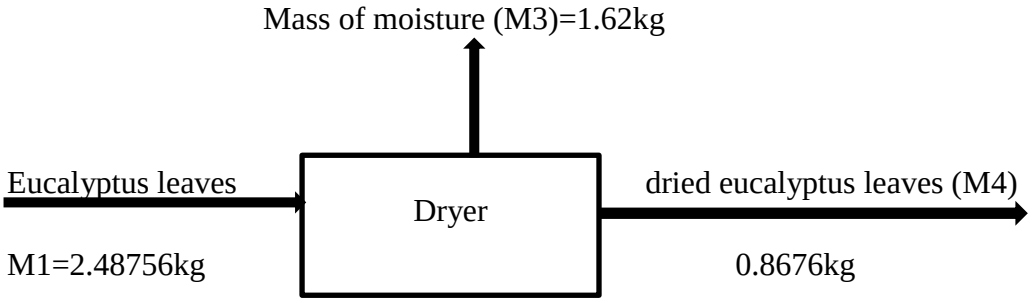
2.5kg = m1 + 0.001244kg

m1 =2.48756kg

x = $\frac{0.000063kg}{2.48756Kg}$×100%=0.002533%=0.00002533 it is almost negligible.

**Material Balance on Dryer**

From the literature review the fresh eucalyptus leaves after drying lose its weight from 60-70%. Taking the average 65% weight loses. It is then dried in electrically operated tray drier or oven at a temperature of about 105℃. Assume cleaned eucalyptus leaves its moisture content should be decreased by sun light.




Mass in + generation = mass out +consumption +accumulation

Mass in = mass out

Over all material balance:

2.48756kg = m3 + m4

Weight loss after drying (m3) = 0.65×2.48756kg

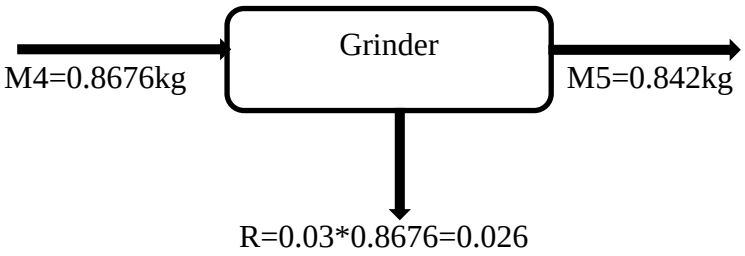
m3 = 1.62kg

Substitute m3 in equation: 2.48756kg-1.62kg = m4

m4=0.8676kg

**Material Balance on Grinder**

The amount of dry raw material left to the grinding vessel. Some amount of mass are loss during grinding that are between 2-4% are loses then we can take the average 3%.



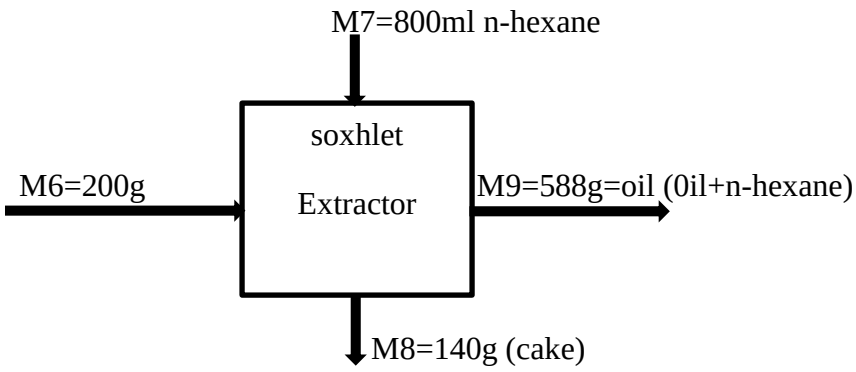

Mass in + generation = mass out +consumption +accumulation

Mass in = mass out

0.8676=0.026+ m5

0.842 kg =m5

**Material Balance on Soxhelt extraction**




Mass in + generation = mass out +consumption +accumulation

Mass in = mass out

Mass of sample powder eucalyptus leaves + mass of n-hexane = Mass of (oil +n-hexane) +mass of cake

200g +528g = 140g +588g

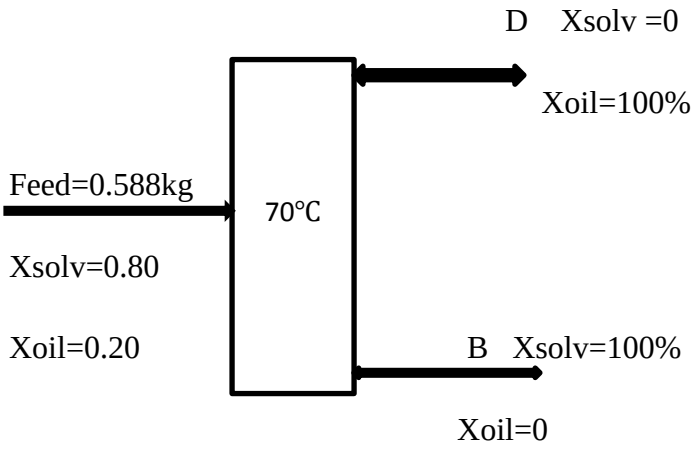
728g = 728g

Density of hexane is 0.66g/mL =0.66kg/L

Mass of solvent = Density of solvent × Volume of solvent

=0.66kg/L×0.8L=0.528kg

**Material balance on Distillation Unit**




Assumptions

- Steady state conditions
- At the top pure oil only exist
- At the bottom pure solvent only exist

The composition of the feed, the top and bottom product was assumed. Mole fraction of solvent at feed = 0.80, Mole fraction of top product = 100% and Mole fraction of bottom product = 100%

F=D+B

Oil balance around the column

0.2×0.588Kg = 0×B + 1× D,

D= 0.176 Kg

Solvent balance

0.8×0.588Kg= 1×B + 0× D, B=0.4704 Kg

## 5.3 Energy Balance in laboratory

**Energy Balance Drying**

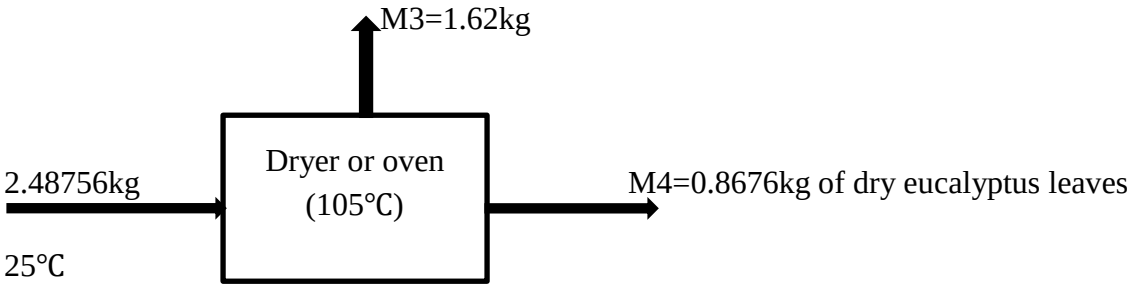



Table 7 Specific Heat capacity from table at d/t temperature

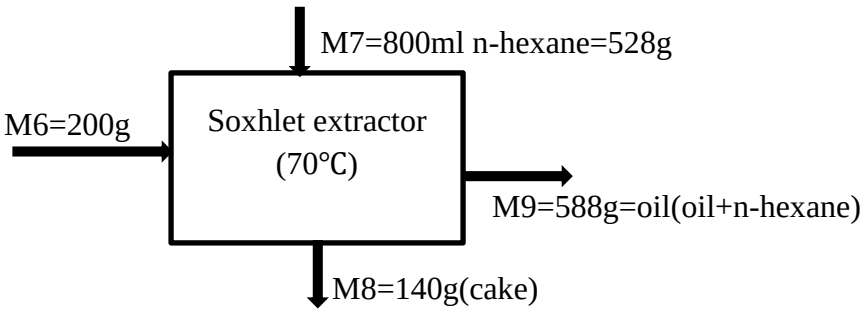
| T($^0$C) | Specific heat capacity(kJ/Kg.$^0$C) |
|---|---|
| 25 | 1.235 |
| 105 | 2.435 |

**From conservation of energy**,

Energy in+ energy generation = energy out + energy consumption + energy accumulation

Energy in = energy out Cp=0.86+0.015T is relationship b/n temperature and specific heat capacity eucalyptus. Q= MCP ΔT =(2.4875kg) ×(2.435KJ/Kg.$^0$C) ×(105-25)$^0$C Q =483.474KJ

**Energy balance on Soxhelt extraction**



**Energy balance on the soxhlet**

Assuming for extraction of oil from the eucalyptus at $70^0$C; taking 1.91KJ/kg.$^0$C is Specific heat capacity. From conservation of energy

Energy in+ energy generation = energy out + energy consumption + energy accumulation

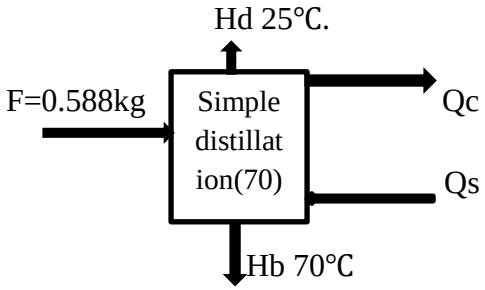
Energy in = energy out

Q= MCP $\Delta$T= (0.2+0.528) kg×(1.91+2.195) KJ/Kg.$^0$C×(70-25)$^0$C =Q =134.5KJ

**Energy balance on Distillation Unit**

**Energy balance calculation:**

Balance on simple distillation: During energy balance calculation we have to account sensible heat of feed (Hf ), sensible heat of distillate (Hd), sensible heat of spent eucalyptus (Hd), heat lost in the condenser (Qc) and heat supply by the steam (Qs). Let base temperature is at room temperature i.e. 25℃. If the temperature of feed, distillate, and bottom product will be 25℃., 70℃. And 25℃.




For 100% n-hexane/eucalyptus oil composition specific latent heat is 2256kJ/kg. To find heat lost balance around the condenser must be done. Heat of vaporization of the vapor is the sum of latent heat of the vapor and sensible heat to raise the temperature to the boiling point. Sensible heat of the distillate is zero because it is in liquid phase and exists in the base temperature i.e. $\Delta$T=0. The specific heat capacity of eucalyptus oil is 2KJ/kg℃ and specific heat capacity of n-hexane is assumed to be 2.195kJ/kg℃. Average water oil specific heat capacity is calculated as;

Cpavg = 0.8×2.195KJ/kg.℃ +0.2×2KJ/kg ℃ =2.156KJ/kg0C Sensible heat to raise the temperature at the boiling point of 700C is calculated as; mdCpavg$\Delta$T=0.588Kg×2.156J/kg℃×(70-25)℃= 57.05KJ

Since we assumed no reflux, mvapor = mdistillate

Hv= latent heat of vaporization at boiling point + sensible heat to raise liquid to the boiling points.

Hv = 57.05kJ +2256kJ/kg×0.588kg =1.384MJ

$H_v = H_d + H_l + Q_c$ where $H_l$=heat of reflux. $H_d = H_l = 0$

Therefore, Hv= Qc = 1.384MJ

Assume, since it is non-reacting mixture, if all amount of supplied heat to the simple distillation is liberated at the condenser (approximately 100% efficiency) and the simple distillation is well insulated, therefore amount of heat supplied will be equal to the amount of heat removed. Therefore, $Q_s$ = 1.384MJ

## 5.4 Energy and material balance in industrial scale up

### 5.4.1 Material balance in industrial scale up

Table8:Import of essential oils.

| Year | Quantity(Ton) | Rate(r) |
|---|---|---|
| 2002 | 577.6 | - |
| 2003 | 632.4 | 0.094 |
| 2004 | 728.6 | 0.1521 |
| 2005 | 929.4 | 0.2756 |
| 2006 | 1106.8 | 0.19 |
| 2007 | 1026 | 0.073 |
| 2008 | 1420.5 | 0.3845 |
| 2009 | 1428.6 | 0.0057 |
| 2010 | 1530.6 | 0.0714 |
| 2011 | 1596.5 | 0.043 |

Using the formula Q=P (1+r), n=9. Find the growth rate(r) value in each year and take an average value r= (0.094+0.1521+0.2756+0.19+0.073+0.3845+0.0057+0.0714+0.043)/9, r=0.143 or 14.3%

Annual average growth of import production during the period considered is 14.3%. By taking an average growth rate r=14.3% we can forecast import amount.

Table 9 Forecast import amount

| Year | Forecasted import ( in ton) |
|---|---|
| 2012 | 1596.5×1.143=1824.8 |
| 2013 | 2085.74 |
| 2014 | 2384 |
| 2015 | 2724.9 |
| 2016 | 3114.6 |
| 2017 | 3560 |
| 2018 | 4069 |
| 2019 | 4650.8 |
| 2020 | 5315.8 |
| 2021 | 6075.9 |
| 2022 | 6944.8 |

**Essential oil demand**

The future demand for essential oil very much depends on the growth of the industrial sector. However, since all industrial production processes do not use essential oil, the growth of the sector as a whole could not be used. But, since the growth of industrial outputs that make use of essential oil (such as food industry, soap industry, cosmetics, pharmaceutical industry, plastic industry etc.) Is related with the growth of the urban population growth rate, population based forecast can be made.

Table 10 Forecasted demand for essential oil.

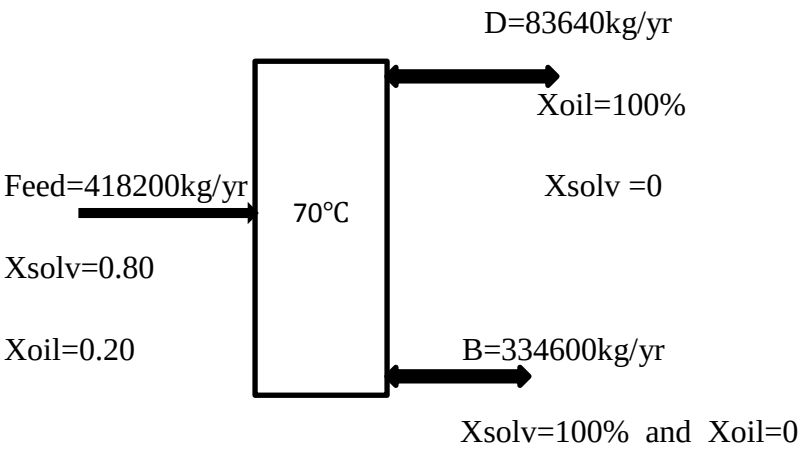
| Year | Forecasted import ( in ton) |
|---|---|
| 2013 | 2093 |
| 2014 | 2407 |
| 2015 | 2768 |

| | |
|---|---|
| 2016 | 3183 |
| 2017 | 3660 |
| 2018 | 4210 |
| 2019 | 4841 |
| 2020 | 5567 |
| 2021 | 6407 |
| 2022 | 7363 |

**Plant Capacity**

If the plant/company starts its production in 2022, the production capacity is will be Production capacity =forecasted demand in 2022 - forecasted the import in 2022= (7363-6944.8) in ton = 418.2 ton/year. Based on the availability of raw material (eucalyptus leaves) we assume our company covers 20% in the total imported oil =418.2×0.2 = 83.64 ton per year 94.83m3=94830L/yr. since, density of essential oil is 882Kg/m3. So the plant capacity when it works in full capacity and efficiency is 94830 L/ year. On the other hand, the company works 300 days per year. Depending on the above sentence explanation our company should have a plan to produce 94830 L/yr of essential oil.

**Material balance on Distillation Unit**




**Assumptions**

Steady state conditions ,At the top pure only exist , At the bottom pure solvent only exist the

composition of the feed, the top and bottom product was assumed. Mole fraction of solvent at feed = 0.80, Mole fraction of top product = 100% and Mole fraction of bottom product = 100%

F=D+B

Oil balance around the column

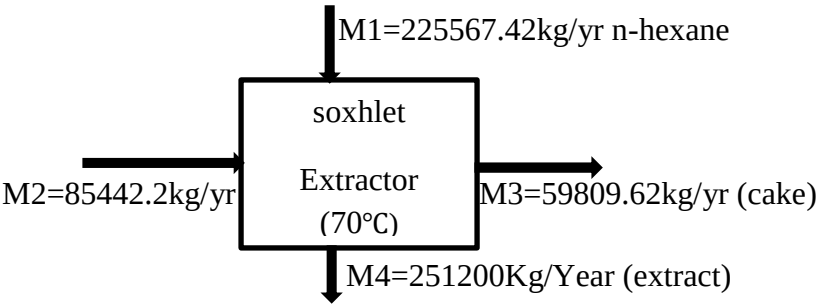
0.2×F= 0×B + 1×83640kg/yr , F=418200Kg/Year

Solvent balance

0.8×F = 1×B + 0× D, B=334560Kg/Year

From these two equations using simultaneous method calculation the bottom product and the feed flow rate contains 334560and418200Kg/Year, respectively.

**Material Balance on Soxhelt extraction**




Take ratios solid to solvent =200g/800mL=1g/4mL then

One mass of solid = 2.64 mass of solvent because mass of solvent = Density of solvent × Volume of solvent.

=0.66kg/L×0.8L =0.528kg

Solid to solvent ratios is 0.2: 0.528 and from laboratory relationship powder to removal of cake=200g/140g=20/14=10/7 then 10mass of solid powder=7 mass of cake=1ms=0.7mc ms=mass 0f solid=m2

Mc=mass of cake=m3 m1+m2=m3+m4 and relationship b/n powder and mixture of solvent and oil=200g/588g from this relation from 200g of powder 588g of mixture of oil is produced. 0.2Kg=0.588Kg m2= 251200Kg m2=251,200×0.2/0.588=85,442.2Kg/year and from relationship

0.2Kg powder=0.528Kg solvent. 0.2=0.528 85,442.2=m1 m1=225,567.42Kg/yr of powder is required to produce.

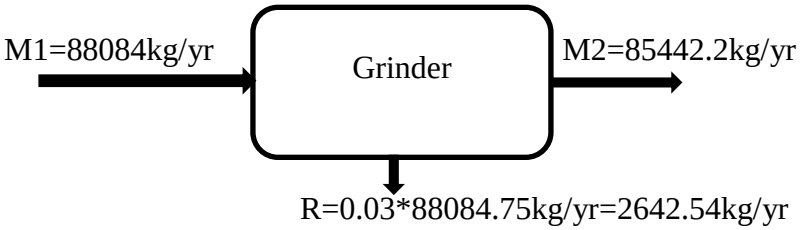
Mass in + generation = mass out +consumption +accumulation

Mass in = mass out m1+m2=m3+m4,

m3=m2+m1-m4=225,567.42Kg/yr+85,442.2Kg/yr-251,200Kg/Yr
m3=311,009.62Kg/yr-251,200Kg/Yr
=59,809.62Kg/yr.

**Material Balance on Grinder**

The amount of dry raw material left to the extraction vessel will be m2 = 85,442.2Kg/year powder and some amount of mass are loss during grinding that are between 2-4% are loses the we can take the average 3%.




Mass in + generation = mass out +consumption +accumulation

Mass in = mass out

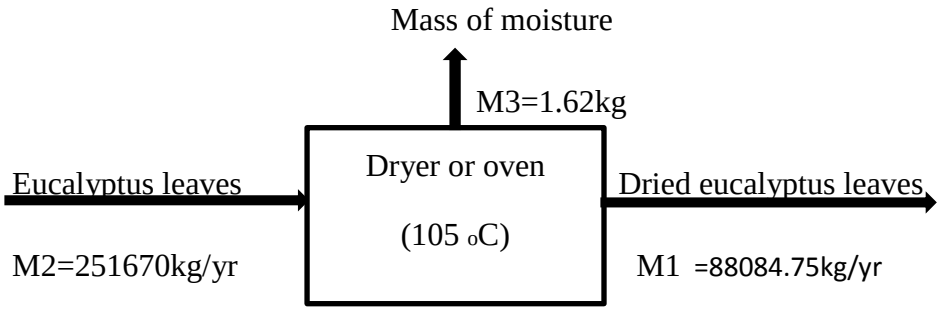
Mass of dried eucalyptus = Mass of powdered dried eucalyptus

m1=85,442.2Kg/year+0.03m1,
(1-0.03) ×m1=85,442.2Kg/year

m1=85,442.2Kg/0.97yr
=88,084.75Kg/yr

**Material balance on dryer:**

From the literature review the fresh eucalyptus leaves after drying lose its weight from 60-70%. Taking the average 65% weight loses. It is then dried in electrically operated tray drier or oven at a temperature of about 105$_o$C.




Mass in + generation = mass out +consumption +accumulation

Mass in = mass out

Over all material balance:

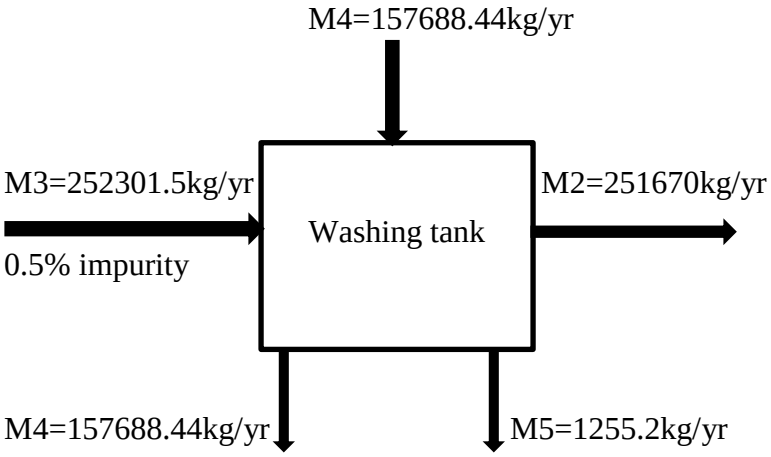
m2 = $m_3$ + m1 Weight loses after drying ($m_3$) = 0.65×m2

$m_3$ = 0.65m2=0.65×251,670.7Kg/yr=163,586Kg/yr

Substitute $m_3$in equation, m2=0,65m2+88,084.75Kg/yr

(1-0.65) m2=88,084.75Kg/yr, m2=251,670.7Kg/yr

**Material balance on washing tank**




Mass in + generation = mass out +consumption +accumulation

Mass in = mass out

Assume the efficiency of washer is 99.5% in removing the impurity from fresh eucalyptus leaves. Over all material balance on washer

$m3 = m_2 + m_5$

Impurity removed ($m_5$) = (m3×0.995×0.005) kg

$= 4.975\times10^{-3}m3 = 4.975\times10^{-3}\times252{,}301.5Kg/yr$

$=1{,}255.2Kg/yr$

The impurity in the fresh eucalyptus leaves= 0.005×m3 = 0.005m3=0.005×252,301.5Kg/yr =1261.52Kg/yr.

So, the impurity remains after washing = $0.005m3 - 4.975\times10^{-3}m3$

$= 2.5\times 10^{-5}m3 = 2.5\times 10^{-5}\times252{,}301.5Kg/yr.$

=6.31Kg/yr.

Substitute $m_5$ to equation (1): $m3 = 251{,}670.7Kg/yr + 2.5\times10^{-3}m3$

$(1-2.5\times10^{-3})$ m3=251,670.7Kg/yr.

m3=251,670.7Kg/0.9975yr

m3=252,301.5Kg/yr.

$x = \frac{6.31}{251{,}670.7}\times100\% = 0.00251\% = 0.0000251$ it is almost negligible.

To calculate the required mass of water there a relationship from laboratory that is 2.5Kg powder=4Kg of water

m4 =252,301.5 Kg/yr.,

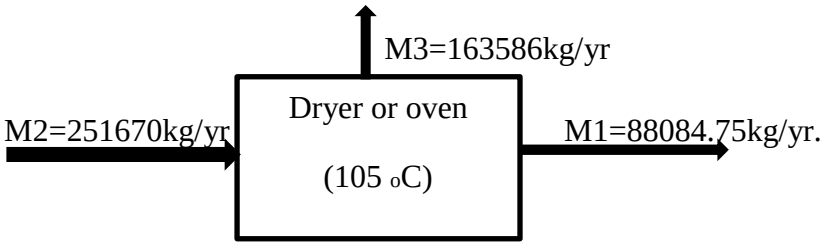
m4=252,301.5×2.5/4

=157,688.44Kg/yr. is water required.

## 5.5 Energy balance in industrial scale up

**Energy balance on Drying**

From the literature review the fresh eucalyptus leaves after drying lose its weight from 60-70%. Taking the average 65% weight loses. It is then dried in electrically operated tray drier or oven at a temperature of about 105oC.




From conservation of energy. Energy in+ energy generation = energy out + energy consumption + energy accumulation. Energy in = energy out

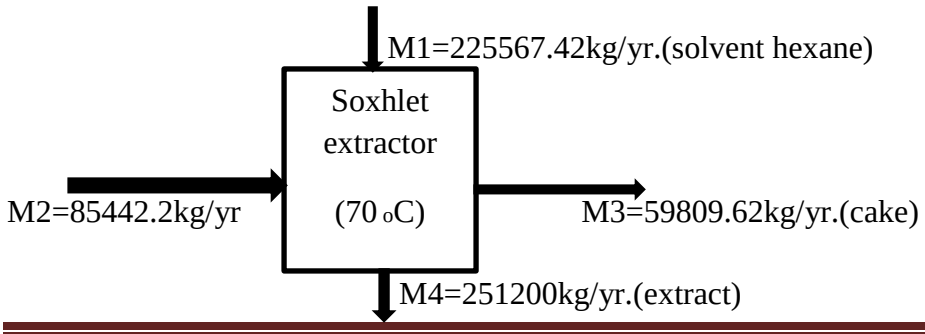
Table 11 Specific heat capacity from table at different temperature

| T(oc) | Specific heat capacity(kJ/kg.0C) |
|---|---|
| 20 | 1.16 |
| 25 | 1.235 |
| 70 | 1.91 |
| 105 | 2.435 |

Q= MCP ΔT = (251,670.7Kg/yr) ×(2.435KJ/Kg.0C) ×(105-25)0C Q =4.9×10^4MJ/yr.

**Energy balance on Soxhelt extraction**

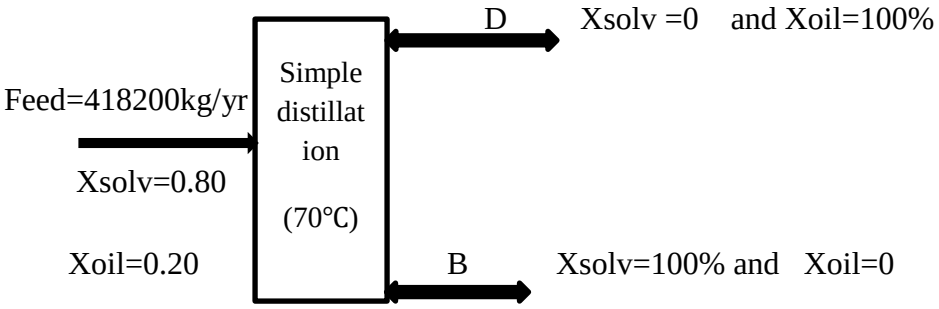
Solvent Hexane (m1) =225,567.42Kg/yr.



From conservation of energy, Energy in+ energy generation = energy out + energy consumption + energy accumulation. Energy in = energy out

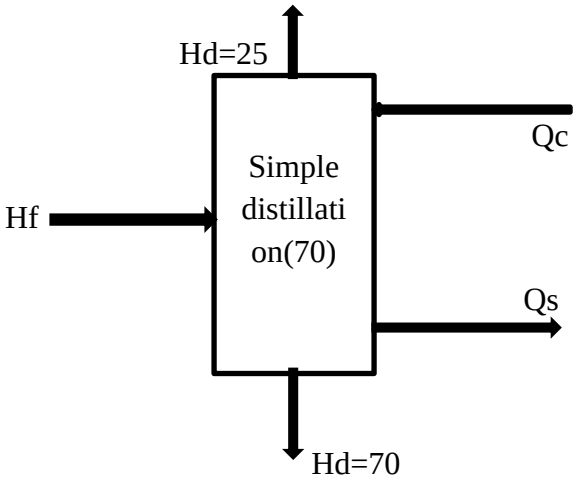
Q= MCP ΔT= (85,442.2Kg/yr +225,567.42Kg/yr)×(1.91+2.195) kJ/Kg.$_o$C ×(70-25)$_o$C
Q =5.6×10^$_4$MJ/yr.=4666.7MJ/month=155.6MJ/day

**Energy balance on Distillation Unit**




**Energy balance calculation:**

Balance on steam distillation: During energy balance calculation we have to account sensible heat of feed ($H_f$ ), sensible heat of distillate ($H_d$), sensible heat of spent eucalyptus leaves ($H_d$), heat lost in the condenser ($Q_c$) and heat supply by the steam ($Q_s$). Let base temperature is at room temperature i.e. 25$_o$c. if the temperature of feed, distillate, and bottom product will be 25$_o$c, 70$_0$c and 25$_o$c.




For 100% n-hexane /eucalyptus leaves oil composition specific latent heat is 2256kJ/kg. To find heat lost balance around the condenser must be done. Heat of vaporization of the vapor is the sum of

latent heat of the vapor and sensible heat to raise the temperature to the boiling point. Sensible heat of the distillate is zero because it is in liquid phase and exists in the base temperature i.e. ΔT=0. The specific heat capacity of eucalyptus leaves oil is 2kJ/kg $_{o}$c and specific heat capacity of n-hexane is assumed to be 2KJ/kg.$^{0}$C. Average water oil specific heat capacity is calculated as;

$C_{pavg}$ = 0.8×2.195KJ/kg.$^{0}$C +0.2×2KJ/kg.$^{0}$C

= 2.156KJ/kg.$^{0}$C

Sensible heat to raise the temperature at the boiling point of 70$^{0}$C is calculated as; $m_d C_{pavg} \Delta T$= 251,200Kg/yr ×2.156KJ/kg.$^{0}$C ×(70-25)$^{0}$C

Q=2.44×10$^{4}$MJ/yr

Since we assumed no reflux, $m_{vapor} = m_{distillate}$ $H_v$ = latent heat of vaporization at boiling point + sensible heat to raise liquid to the boiling points.

$H_v$ = 2.44×10$^{4}$MJ/yr+2256KJ/kg×251,200Kg/yr = 5.911×10$^{5}$MJ/yr.

$H_v = H_d + H_l + Q_c$ where $H_l$=heat of reflux. $H_d = H_l = 0$

Therefore, Hv= Qc =5.911×10$^{5}$MJ/yr.

Assume, since it is non-reacting mixture, if all amount of supplied heat to the simple distillation is liberated at the condenser (approximately 100% efficiency) and the simple distillation is well insulated, therefore amount of heat supplied will be equal to the amount of heat removed. Therefore, $Q_s$ =5.911×10$^{5}$MJ/yr.

# 6. CHAPTER SIX

## 6.1 Conclusion and recommendation

### 6.1.1 Conclusion

In this project, Soxhlet extraction was used. From the experimentation it was found that maximum oil yield of 3.117% was obtained at particle size range of 0.5-1mm and extraction time of 180min and solvent to solid ratio of 7:1. A minimum oil yield of 0.821% was obtained at particle size range of 3.35-1.7mm, extraction time of 60 min and solvent to solid ratio of 6:1(ml: gram). Most physical properties of eucalyptus oil were between standards value but some were not this could be due to different factors such as duration of characterization, place to put the oil (Veranda P. S. (2006-07)). Finally, it can be concluded that it is possible to extract essential Oil from eucalyptus (Globulus) leaves for pharmaceutical purpose using Soxhlet extraction method the simplest extraction method and available equipment in chemical and food engineering laboratory rooms for extraction of eucalyptus essential oil.

### 6.1.2 Recommendation

In this project, investigation of effects of extraction time, particle size and solvent to solid ratio on the oil extraction yield and product characterizations were made. However, there are comparison of different extraction technology such as steam distillation, supercritical fluid extraction and cold press with solvent extraction, investigation of temperature, pressure and solvent type, investigation of eucalyptus leaves in remaining Ethiopian regions, constitutes of essential oil using Gas chromatography mass spectroscope (Gc-Ms) has to be made to project successfully. Study can be carried on converting the residue or the waste to valuable product and using the hydrosol as integrated small scale industry. Therefore, anyone who has great interest in doing project in this area can go with those investigations.

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# APPENDICES

Removal of impurity of eucalyptus leaves by washing

Partial sun drying.

Oven drying

Size reduction by using rapid grinder.

Extraction of oil using soxhlet setup.