# EXTRACTION OF ESSENTIAL OIL FROM PLANT FOR PRODUCTION OF PERFUME

## A RESEARCH PROJECT

## BY
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## BEING, PROJECT SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENT FOR THE AWARD OF DEGREE OF BACHELOR OF ENGINEERING

## DECEMBER,1998

# DECLARATION

I, MACHIE CHINYERE hereby declare that this project report is an authentic report of my experimental work and that to the best of my knowledge has not been presented elsewhere for the award of any degree of Diploma. All published work used has been duly acknowledge in the references.
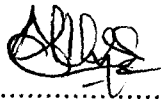
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SIGNATURE

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DATE

# CERTIFICATION

I certify that this project work was carried out by MACHIE CHINYERE of the Chemicals Engineering Department in Federal University of Technology, Minna in partial fulfillment of Bachelor of Engineering in Chemical Engineering.

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# DEDICATION

This Project is dedicated first and foremost to the God Almighty, and to Jesus Christ - the one I worship, and my Source of straight. This work is dedicated to my Parents Mr and Mrs R.E. MACHIE and my brothers and sisters for their love, sacrifices and assistance.

# ACKNOWLEDGEMENT

First of all, I acknowledge God Almighty for his guidance, grace, mercy, provision, protection against all devices of the enemy throughout the duration of my stay in this school. It looked hectic and tedious at the beginning but for straight given by jesus christ, here we are now to God be the glory and adoration.

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My special thanks to my friends Ebby Agbo Omoifo U.K Meka, Oby Chigozie Udeegbonam Chichi and all my course mate

My greatest thanks goes to my parents and my brothers and sisters for their financial and moral support throughout the programme. Thanks alot and God bless.May Almighty God bless you all.Thanks and remain blessed.

## ABSTRACT

This research project is on the prodution of essential oil to produce perfume using Eucalyptus Macarthuri leaves.

To achieve this,specified amount of E. Macarthuri leaves was used and the extraction was carried out by using Petroleum Ether as solvent.

The process of the extraction was of the continuous type using sohlex apparatus.The effect of particles size and time on the amount of oil extracted was studied. It was found that for a particle size of $850\mu$ m, $710\mu$ m, $500 \mu m$, $250 \mu m$, and the amount of oil recovered was 0.4, 0.14, 0.65 and 0.58 respetively.The trend shows that the amount of oil recovered increased as particle size is increase up to an optimum size of $500\mu$ m.

It was also shown that the optimum time for production of oil from particular size is three hours.

It is recommended that the essential oil produced in this work be characterized for use in the production of perfume.

Finally the extractor equipment which will produced 3000kg was design for the industrial production of perfume.

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# CHAPTER ONE

## 1.0 INTRODUCTION

The origins of perfumery are lost in the mists of antiquity. The first few recorded facts take the form either of recipes, like that inscribed (176 - 147 B.C) on a laboratory wall in temple of Horus at Edfu, or of reliefs depicting the offering of incense to gods, sacred objects or the deified dead. Reliefs of this kind are also to be seen in the same remarkably preserved temple of the sun god, side by side with a representation of sight, hearing and taste in which the latter is symbolised by a tongue.

The Edfu recipe, now over 2000 years old, was for a perfume dedicated to the sky deity and goddess of love, Hathor. It contained strong wine and water as well as ingredients that have been translated as incense, calamus root, mastic, styrax bark and more dubiously, as carob beans, violet seed and a wood related to rhodium root. A full account of the recipe and its rather tedious method of preparation has been published ina french perfumery journal(1).

Rameses II, most celebrated of all Egytain kings, lived from 13243 to 1258 B.C in the temple of Abu - Simbel he was shown offering incense to ptah, god - guardian of artists, and in the Ramesseum at theber making a similar offering to the ithyphallic god of the harvest, min or pan, in the gold, cornelian and lapis lazuli cosmetic containers found in the tomb of princess sit - Hatter of the 12th Dynasty (2200 - 1700 BC) and still to be sen in the Museum of Gizeh.

These and other examples indicate that perfumes were used in Ancient Egypt in the following ways:

(a) As incense or perfumed unquents, offered to the gods in religious ceremonies.

(b) As one of the three main techniques of embalming the dead, and others being the removal of the brain and inestines and treatment with the desiccant natron Canatural hydrous sodium carbonate).© For personal enjoyment and adernment, as today but then only by the priviledge few.

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The tomb of Tukankhamun cntained some veses of perfumed unguent that were subjected to analysis in 1926.[2] Although there were said to be still elusively fragant, the method of analysis available at that time were only able to reveal that 90 percent of the unguent or pomade had been containing resin or balsam. Here it may be mentioned tht a mere modern scheme of analytical investigation was adopted in 1969 by zolotovitch and Popov [3] when examining the perfumed residues remaining in some vessels found ina thracian tomb dating back to the 1st century A.D. These investigators used a combination of chromatographic procedures with UV, IR and .other techniques and they were unable to identify the essential oil and balsams whose perovidised tepenic and lighly polymerised traces were still in evidence. It seems reasonably certain that the Egyptains of these remote periods had very little knowledge of flower perfumes.

Perfumes and body oils were familiar in China by 300 B.C and probably long before that incense had been mentioned by confucus 6th century B.C. in some of his aphanisms, though little is known about the use of perfumes in ancient China, it seems very probable that developments ran parallel there to those in the middle east and Egypt indeed, having regard to the innovations and inventions introduced into global culture by the chinese, such for example as paper (the Egyptain papyrus) ink pne, ceramics and gun powder, and bearing in mind also the profusion of odorous materials available inthat vast country, ancient China may indeed have been advance of factorily of Egypt, the Chinese were the first to know about and to value musk, to which their doctors attributed therapentic properties but which afterwards become perhaps the most widely and persistently used and appreciated of all odorants.

In 538 A.D Buddhism was first introduced in Japan from China. One of the features of Buddhist ritual was the use of incense and this was the first time in Japanese history that perfume had entered into the life of the people. Do Teikichi Hiraizumi, at one time President of the Japan perfumery and flavoring association, has written authoritatively on the Ancient Art of incense in Japan[4]. He recalls how,

inthe 11th century, the incense competition , a kind of game using incense became popular. This was a game in which interested persons got together with their own compounds of incense that had been mixed before hand, and tried to guess at the composition of their felow contestants compounds. The use of incense in this way per entertainment led people to devise paste or semi solid incenses, He guotes three recipes which illustate year 1400. Here is the recipe of formula for `Bare of early Winter: Agar wood 40.0, Clove buds 40.0, Sandalwood 1-3, Rhizomes of cyperus rotundus 3.0, musk grians 2.0, Thais rudolphi (Shell) 1-2 pistacia khinjuk (fossi resin) 1.0, Liguidambar orientalis (Styrax) 10.0.

In another interesting account of this incense ceremony, referred to also as Kode E.F. stange [5] says that it was essentially a highly elaborated from intellectual culture, framed and garnish with a charming ritual and most curious and significant of all, based on the cultivation of one of the senses of smell - which has absolutely no plate whatever in western aesttetics. Here incense is used in the popular sense of assistance burnt in order to produce fragrant smoke. To the perfumer incense' has a more specialised meaning and refers to the resin Olibanum or frankincense.

Aristophanes (444 - 385 B.C) mentioned three special types f myron, a generic name for perfume viz - Bacharis, megaceion, psagdes.

Most of the leading items in our perfumery vacabulary are words of Roman orgin; perfume, fragrance, odour and olfection though others go back to sascrit roots and some (e.g Osmics) are of Greek origin.

The next great impetus to the development of perfumery came from the Arab world, in the centuries following the defeat of Rustum and his persians at Kalessia in 637 A.D as H.G wells phrases it: learning sprang up everywhere in the footsteps of the Arab conquerous [5] the very words algebra, chemistry alcohol and alembic are Arabic in origin. Arab alchemists inherited distillation techniques from the syriacs. They improved both the techniques and the equipment used in distillation although their knowledge of the theoretical basis of what they were doing remained imperfect ·

[6]. Nevertheless, they produced a typical of rose - water as well as tinctures and essences. The name of the Famous physician, Avicenna, has in fact been connected with an early process for extracting essential oils or attars by distilling with steam and in the case of roses, of producing both attar and rose - water, it will be apparent that all these primitive methods were only marginally efficient. Ambergris, much praised by Rhazes, was used by the disorders. It is said to have been inlcuded in the precious aromatic of the east presented by the Calip Haroun al Rschid (C.800 A.D) to the Emperor charlemagne. A few centuries later, ambergris and musk were being used in Europe for the embalming of the royal dead.

Mahomet himself is reperted to have said: `Women, Children and Perfume are what I most cherish in this World'. Mosques were in those days built with a small quantity of musk incorporated in the mortar and even today, when the sun shines on them, a faint but detectable odouris given off by the walls, in which a trained lose can detect beyond dispute the odour of musk.

Islam's passion for perfume is well illustrated in the voluminous pages of the ArabianNights.

The first revolution in perfumery did not really take place until the `7th and 18th centuries, when alcohol took over form oils, Pomades and powder bases as the inseparable vehicle - solvent for perfumery materials. The second major development was undoutedly the introduction and use in the early 19th century of flower pomades and a much wider and better selection of essential oils. It was only with the introduction of an increasing number of perfumery chemicals and isolates towards the end of the 19th and beginning of the 20th century, however, that modern perfumery began to emerge and it is even possible that the Golden age of fine perfumes, combining new ideas and individual artistry with a profusion of natural material and many of the more important synthetics, has already gone, never to return.

Billot has thus described this third stage of the revolutionary - evolutionary process [8] it was in1882 that Paul pergnet, then joint owner of the perfumerie

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Houbigent, was to create the soap fougere Royale, based on bergamot and Coumarin. In 1896 he created Le parfum ideal, the model composition, in which synthetics in ·conjunction with natural products put perfumery on a new trock.

Finally two minor evolutionary charges took place in the development of leather and leather - mossy twenty perfumes and, rather later of `green' and green fruity composition.

A short history of the great perfumers first published in 1962[9] attracted a great deal of attention, largely because most people had formerly been kept in ignorance of the function significance and identity of the perfumer. A perfume was, in many cases, a kind of anonymous production a mystic pallas. Athene springing, complete to its name and stylish packaging, direct from the brain of the manufacturing perfumer or couturier. As the author of the History wrote at the time: it is somewhat difficult to speak freely in this sphere of activity of personal creations, because certain firms are not anxious for their commercial success to be even partly attributed to an individual who is not in fact the prorietor of the concern. Perfumers themselves, whether employed by such firsm or working from their own laboratories, are unable to reveal their secrets for very similar reason.

All perfumes, moreover, had a faint fatty note in common, to which one became accustomed, they were not very stable and deterkiorated at the of a certain time despite all the tratments and chilling to which we subjected them[10].

For today in Nigeria there is no company that produces perfume, they only do dilution of perfume and packaging.

The brief biographical record that follows is based on the much longer and more informative short History[11] and can only be regarded as illustrative rather than comprehensive.

## OBJECTIVES OF THE STUDY

**The objective of the work include**:

(1) To produce an affordable perfume from locally sourced material like sucalyptus Macarthuri leaves. This will be achieved by extraction of essential oils from the leaves by use of solvent such as petroleum ether and ethanol/acetene mixture and the blending with additives such as labdanum, frankinces, myrrh and perubalsam.

(ii) To determine the effect of extracts obtained from the leaves.

(iii) To determine the effect of time on amount of extract obtainable from the leaves.

(iv) To use the data from above to design a process for the industrial production of the perfume.

## SCOPE OF WORK

The scope of work involves the use of sochlet extraction for the extractionofessential oil from ground encelptus maca-thuri ethanol/acetene at temperature of 70°C. Particle size will be investigated.

## LIMITATION

(1) The major limitation of this work is time constraint. This is because a minimum period of 6 months is required for perfume to age and nature.

(2) The project is capital intensive and this worked against producing it in a reasonable quantity.

(3) Most of the apparatus used in the laboratory for carrying out this project are absolute.

# CHAPTER TWO

## 2.0 LITERATURE REVIEW

## 2.0 SIGNIFICANCE OF PERFUME

Perfume derived from per and fumum is derived from incendo. Perfumes are pleasent scent or smeel or substance which have a pleasing odour. The word has a more esoteric connotation, which might perhaps be stated as a judicious blend of odorants.

## TO DISTINGUISH BETWEEN THE TERMS PERFUME, SCENT, ODOUR, ODORANT AND FRAGRANCE

### 2.1.1 ODOUR:

Derived from the latin odor - oris, cognate with the Greek, signifies any scent or smell whether pleasant or offensive.

### 2.1.2 ODORANT

This is an odorous substances perfumery materials whether natural or synthetic are simply described as odorants.

### 2.1.3 PERFUME

Perfume is in form of incense but nowadays it is usually as a liquid perfumes are available in many forms and find ina specialised sense, perfume is the term applied to a conventionally strong alcoholic solution of odorants as opposed to such weaker solutions are toil waters.

### 2.1.4 SCENT

Scent has a specialised meaning referring to odour left behind by an animal during a chase scent is not a better, apter or more euphonious word than perfume indeed the reverse is the case even so, we should perhaps welcome the rehabilitation

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of scent because it permits us to use as a reasonable alternative.

## 2.2 CONSTITUENTS

A perfume may be defined as any mixture of pleasantly odorous substances incorporated in a suitable vehicles. All the products used when humans first started synthesizing materials for use in this field, they endeavored to duplicate the finest in nature. The finest modern perfumes are neither synthetic or completely natural, the best product of the art is a judicious blend of the two in order to enhance the natural perfume. The constituents of perfumes are the vehicle or solvent, the fixative and the odoriferous element.

## 2.2.1 VEHICLES

The modern solvent for blending and holding perfume materials is highly refined ethyl alcohol mixed with more or less water according to the solubilities of the oils employed. This solvent, with its volatile nature, helps to project the scent it carries, is fairly inert to the solutes and not too irritating to the human skin. The slight natural odour of the alcohol is removed by deodorizing or prefixation of the alcohol. This is accomplished by adding a small amount of gum benzoin or other resinous fixatives to the alcohol and allowing it to mature for a week or two. The result is an almost odorless alcohol, the natural rawness having been neutralized by the resins.

## 2.2.2 FIXATIVES

In an ordinary solution of perfume substance in alcohol, the moare volatile materials evaporate first, and odor of the perfume consists of series of impressions rather than the desired ensemble. Fixatives may be designed as substances of lower volatility than the perfume oils, which retard and even up the rate of evaporation of the various odorous constituents. The type of fixative considered are animal secretions, resinous products, essential oils and synthetic chemicals. Any of these fixatives may or may not contribute to the odor of the finished product but if they do they must blend with and cmplement the main fragrance.

### 2.2.3 ANIMAL FIXATIVE

Castor or castoreum, a brownish orange exudate of the perineal glands of the beaver is employed in the greatest quantity. Among the odoriferous compounds of the volatile oil of castor are benzyl acetophenone and castorin. A volatile resinous component of unkown structure.

### 2.2.4 ESSENTIAL OIL FIXATIVES

A few essential oils are used for their fixative properties as well as their odor, the more important of these are clary stage, vetives, patchouli, orris and sandalwood. These oils have boiling points higher than normal (285 to290oC).

### 2.2.5 SYNTHETIC FIXATIVES

Certain high boiling comparatively odorless esters are used as fixatives to replace some important animal fixatives. Among them are glyceryl diacelate (259oC) eltyl phitacate (295oC) and benzyl benzoate (32oC) other synthetics are used as fixatives although they have a definite odor of their own that contributes to the asemble in which they are used.

### 2.2.6 RESINDUS FIXATIVES ·

Resinous fixatives are normal or pathological exudates from certain plants, which are more important historically than commercially. These hard resins e.g benzoinand gums softer resins e.g myrrh and labdanum, balsams, moderately soft e.g peru balsam and tolu balsams.

### · 2.3 SCIENTIFIC AND MEDICAL ASPECTS

The application of perfumes to the skin is an age - old and widespread custom some perfumes condemn the practice on the grounds that the living skin, with its excretory and respiratory mechanism, its secretious and variable temperature is too changeable a medium to act as a good carrier of perfumes and frequently distorts the odour of substances in contact with it.

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## 2.3.1. TECHNOLOGICAL ASPECTS

The effect of applied science and improved technology upon perfumery practice in a larger manufacturing concerns, has been literally spectacle during recent years. Nuclear magnetic resonance, gas chromatography, mass spectrometry equipment and improved IR spectroscopy facilities computerisation. Some automated processes and a correspondingly different emphasis in the arrangement and equipment of the conventional analytical laboratory. The growing use of such instrument leads to a vast increase in the output.

## 2.3.2. HOW PERFUME APPEAL

The appeal of perfume reaches through the sense of smell. Smell might be described as the sense of memory because it recalls more directly than it does in any other sense. A fine perfume should have fashionable odour.

## 2.3.3 CHOOSING A PERFUME

This involve sprinkling of a few drops of the perfume on to a cotton handkerchief and putting 2 or 3 drops on the inner wrist. The handkerchief should always be of cotton, not silk or synthetic fabrics. As perfume applied to the skin, this should be gently spread with fingers but without rubbing too violently. Excessive fraction tends to denature the perfumes by the development of heat, the undue acceleration of evaporation and some extent the intensification of skin odour.

## 2.4 FRAGRANCE

Fragrance is unique in the realm of cosmetics for it is both an end result in its own right and a quality of product having a diversity of other purposes. Fragrance is a significant part of many other products like creams, lotious, face bath powder and shaving creams. In other to impart fragrance to an alcoholic solution or to a cosmetic preparation, the perfumer blends a variety of natural and synthetic raw material into a complex and usually difficult to describe mixture. This blend is known as a compound or perfume compound. The blend is also called a perfume oil.

### 2.4.1  USES AND ECONOMICS

Fragrances make a major contribution to cosmetic industry, fragrances are used industrially in neutralizing and altering the odor of various products, as well as in creating a distinctive aroma far normally odorless objects.

### 2.4.2  DESCRIBING THE PERFUME OIL

The description of the frangrance that is created or that is sought is one of the most difficult problems be setting the perfumer.  Perfumes that closely simulate the fragrance ofan individual known material, usually by a flower are simply described in terms of that material.

### 2.5 EUCALYPTUS GLOBULUS LABILL AUSTRALIAN FEVER TREE BLUE GUM

FR. EUCALYPTUS, GOMMIER BLEY GER. BLAUER EUCALYPTUS BAUM

The stom of Eucalypts a group of ever green trees and shrubs, began in 1770 when Joseph Banks a botanist, and Daniel Carl Solander, his assistant, collected the first specimen onthe shores of Botany Bay on the East coast of Austrialia daring Captain James cook's first voyage to the pacific ovan.

### 2.5.1  DESCRIPTION

Orignally from Australia and Tasmania, the eucalyptus is now cultivated in the mediterrarean region.  It is a very large tree with smooth bark.  It is the leaves from the older branches that are officinal they are falcate, alternate, petiolate with a pleasant smell.

### 2.5.2  ACTIVE CONSTITUENTS

The leaves contain tannink a bitter, a flavonoid pigment, eucalyptine,k a balsamic essential oil containing mainly cineol or eucalyptol and a bitter resin.

### 2.5.3  PROPERTIES

The essence is antibiotic, a respiratory disintectant and balsamic producing a cooling sensation in bronchitis and asthma, it arrests excessive secretion by the

bronchi.

## 2.5.4 APPLICATION

Inhalations or fumigations may be prepared from the leaves or the essence. Eucalyptus oil is also often used in cough pastives.

## 2.5.5 CLASSIFICATION OF EUCALYPTUS

The leaves, bark oils, buds, fruit, cotuledons the genus eucalyptus belongs to the family myrtaceae which contains about 90 gensa and over 3,000 species. The myrtaceac originally included a tribe, the lecyhideac, which is now generally treated as a separate family the lecyhidaceae as two separate families, the lecyhidaceae and the barringtoniaceae.

## 2.5.6 THE LEAF

The leaves of the eucalypts show two very striking characteristics. Firstly each individual tree develops different kinds of leaves at different stages of its life cycle. The most familiar types being the jurenice and adult leaves. Secondly the leafy crown of the eucalypts in general is built up with unusual rapidity. The ability to build a crown so quickly is due to the naked buds which enable the tree to develop a number of branch orders in the space of a few weeks.

## 2.5.7 CULTIVATION

The parent tree from which seed is collected should be carefully selected from trees showing a high degree of desirable characteristics such as rapid growth, good form, high percentage of oil. The soil used for sowing is usually fairly light and permeable. It consists of 100ms or various soils mixed with river sand, together with a reasonable amount of organic matter.

## 2.6 EXTRACTION

Extraction is a process in which a soluble constituent present either in a liquid mixture or solid is

## 2.6 EXRACTION

Extraction is a process in which a soluble constituent present either in a liquid mixture or solid is selectively removed using a liquid solvent in contact with it. An extractive process usually involve contacting, solvent recovery and raffinate.

### 2.6.1 EXTRACTION WITH VOLATIVE SOLVENT

Three successive washings are usually made carried out similarly to systematic extraction method. The most saturated washing is then pumped into the evaporator concentrated by distilling off the solvent. After the third washing has been drawn off the exhausted flower material is blown into the extractor to distill over and recover the petroleum ether.

### 2.6.1 SOLVENT EXTRACTION

Oil from oil - bearing material is taken into solution with a solvent residue and the oil is recovered from the solvent solution.

## 2.7 LEACHING

Leaching which is also know as solid - liquid extraction, is the removal of a soluble fraction in the form of a solution from an insoluble permeable solid phase with which it is associate. The seperation usually involves selective dissolution with or without diffusion, leaching is favoured increased volume of solids to be leached and by decreased radical distance that must be transversed with the solids, both of which are favoured by decreased particle size on the other hand cause slow percolation rate. The mechanism of leaching may involve simple physical solution or dissolution made possible by chemical reaction.

## 2.8 FACTORS AFFECTING RATE OF EXTRACTION SOLVENT

The liquid choosen should be a good selection solvent and its viscosity should be sufficiently low for it to circulate freely and high polarity of the solvent is important because it makes seperation of oil from solvent by evaporation or

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distillation faster and easier.

## 2.8.1 PARTICLE SIZE

The particle size influence the extraction rate in number if ways, the smaller the size the greater is the interfacial area between the solid and the liquid, therefore the higher is the rate transfer and smaller in the distance the solute must diffuse within the solid.

## 2.8.2 TEMPERATURE

The solubility of the material increases with rise in temperature to give a faster rate of extraction ,the diffusion coefficient will be expected to increase with rise in temperature and this will improve the rate.

## 2.8.3 CHOICE OF SOLVENT

Solvent vary considerably in chemical and physical properties and solvent selected will offer the best balance of number of desirable characteristics high saturation limit and selectively for the solute to be extracted.

## 2.8.4 SOLUBILITY

A low solubility of extraction solvent in the raffinate generally leads to a high relative volatility in a raffinate stripper or low solvent loss of the raffinate is not desolventized. A low solubility of feed solvent in the extract leads to high relative seperation and generally to low solute recovery costs.

## 2.8.5 SELECTIVELY

The most important factor for the success of the extraction process is the quality of the solvent employed. The ideal solvent should possess several properties.
(a) It should completely and quickly dissolve all the odoriferous principle of the flower yet at little as possible of such inert matter as wax, pigments albuminous compounds and the solvent should be selective.
(b) It should possess a sufficiently low boiling point to permit its being easily removed without resorting to higher temperature and the boiling point should not be

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low.

(c) The solvent must not dissolve water since the water present in the flowers would dissolve and accumulate in the solvent.

(d) The solvent must be chemically inert, not react with the constituents of flower oil.

(e) The solvent should have a uniform boiling point when evaporated it must not leave any residue and the solvent should be low - priced and non- flammable.

## 2.8.6 STABILITY

The solvent for extraction should be chemically stable to heat, light and water under process conditions. It must with stand repeated cycles of heating, evaporation and cooling.

## 2.8.7 DENSITY

The difference in density between the liquid phase in equilbrium affects the counter current flow rates that can be achieved in extraction equipment as well as the coalescence rates.

## 2.8.8 VISCOSITY

This is a physical property of solvent and it is the measure of internal molecular friction which hinders flow. A flow viscosity is desirable since extraction is the part governed by capillary flow.

## 2.8.9 TOXICITY RECOVERABILITY AND NON-REACTIVETY WITH EQUIPMENT

In toxiciy low foxicity from solvent vapour inhalation or skin contact is preferred because of potential exposure. In recoverbility the solvent for extraction must normally be recovered from the extract stream and from raffinate stream in a extraction process. Non-reactively with equipment, in this solvent selected for extraction should not react with equipment and some solvents are corrosive to piping and metal components.

## 2.9  ADVANTAGES AND DISADVANTAGES OF SOLVENT EXTRACTION

The extraction rate using this process are usually higher than that from mechanical methods and residues normally contain less than two percent oil at which it could be carried out in the laboratory. The disadvantage are separation of liquid from solid and providing efficient contacting and extracted time.

## 2.10  ESSENTIAL OIL

The term essential oil is often used in the cosmetic and perfume industries as synonmous with perfume oil, base, or compound. It is an odoriferous bodies of an oily nature obtained from vegetable sources,  generally liquid some times semi-solid or solid at ordinary temperature and volatile without  decomposition. Essential oil is a complex mixture of many different chemical bodies, some of which have been isolated, analyzed and reconstituted synthetically.

## 2.10.1  THE ORIGIN OF ESSENTIAL OILS

The components of the volatile oils consists of a variety of compounds which belong to all chemical classes.  There is however certain chemical relations between a number of the component.  It was this similarity that led to chemical research in terms of four groups like straight chain hydrocarbons, benzene derivatives, terpenes and miscellaneous compounds.

## 2.10.2 THE COMPOSITION OF ESSENTIAL OILS

The heating of the plant caused the odoriferous pinciple to evaporate and that upon condensation and subsequent cooling.  The water from the plant is used to carry over the oils, additional water or steam was introduced to obtain better yields and quality.

## 2.10.3 ESSENTIAL OILS

Many plants contain oils, either in the bark, flowers, fruit, leaves, roots or wood, which can be removed by distillation in a current of stem such oils are called volatile or essential oils and ocurs in the leaves of all the eucalypts.

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## 2.11 THE EUALYPTUS OILS OF COMMERCE CAN BE GROUPED OR CONVENIENCE INTO THREE CLASSES.

(a) Medicinal oils

(b) Industrial oils

(c) Perfumery oils

So perfumery oils being the oil were more interested in will be discussed in details.

### 2.11.1 PERFUMERY OILS

Despite the diversity of chemical compositio of essential oils derived from the eucalypts, very few species yield oil containing substances of value to the perfumery and allied industries so far only 3 species have been distilled for commercial purposes.

### (a) E. MACARTHURI

The odour of the leaves when crushed between the hands readily distinguishes the species from practically all other eucalypts. The oil differs from most other eucalypts oils of commerce in the absence of cineole or phallandrene the crude oil is used on perfumery.

### (b) E. CITRIODORA

It is known as the lemon - scented gum or spotted gum. It is readily identified by deliciously fragment citromella like odour of the crushed leaves.

### (C)   E. STAIGERIANA

It is commonly called lemon scented ironbark on account of the fragrant oil from the leaves.

### 2.11.2 USE OF PERFUMERY OILS

The oils of E. Macarthuri and E. Citriodora both of which posses pleansant characteristic, odour are used as denaturants of alcohol for the manufacture of perfumes. E. Macarthuri is a source of geranyl acetate and the fixative eudesmol.

## 2.11.3 PERFUME RAW MATERIALS

Perfume raw material can be divided into several categories they are often called natural or synthetic.

The following schematic division is relatively all inclusive.

(1) Plant materials:

    (a) Essential oils

    (b) Flower oils

    (c) Resine, gums and excidations.

(2) Animal secretions

(3) Chemical substances:

    (a) Isolates from plant materials

    (b) Derivatives of plant material

    (c) Synethetic organic substances.

## 2.11.4 DESCRIBING THE PERFUME OIL CONTROL

Adequate checking and control methods are of course essential at all stages of perfume production. From the intake and assay of raw materials, through storge and processing to filling and packaging. There are six main check point in perfumery.

(a) Control of raw materials

(b) Control of product creation and development

(c) Control of manufacture

(d) Control of packaging like presentation accessories, packaging design and production an container, deliveries.

(e) Control of finished products

(f) Control of storage and dispatch.

Control help to ensure the sustained high quality of a firm's products and makes a vital contribution to its reputation.

## 2.11.5 STORAGE OF MATERIALS

Natural or synthetic products should for preference be stored in a fresh, dark place and healthy basement. All to odoriferous product should be kept for preference in glass containers as a protection against light - induced reactions. the perfume material should not be allowed to stand in open vessels for any length of time before being added to the batch. Proper sealing o tanks as of glass bettles is essential to prevent access of air and loss of volatice materials.

## 2.12 AGEING AND MATURATION

Time is of course an important factor in the ageing and maturing of alcoholic tincture aand alcohol perfumes as well as perfume concentrates. It is only time whether of long or short duration that can transform the initial, rater crude and raw alcoholic solution into a true perfume of mellow distinction.

# CHAPTER THREE

## 3.0 INSTRUMENTATION / EQUIPMENT AND MATERIALS

The apparatus (instrument / equipment )the raw materia 1 and chemical used for this research work were listed in table 3.1 &3.2 below

## 3.1 LIST OF INSTRUMENT AND EQUIPMENTS

| | INSTRUMENT/EQUIPMENTS | MANUFACTURALS |
|---|---|---|
| 1 | Soxhlet extraction apparatus | Smith ,Garmany |
| 2 | simple distillation | England |
| 3 | sleving machine | England |
| 4 | Grinder | Nigeria |
| 5 | oven | Quick fit , England |
| 6 | weighing balance | New Jersey U.S.A |
| 7 | Density bottle | England |
| 8 | Heating mantle | Isopad Isomantle Boehamwood |
| | | Herts England |
| 9 | Stop watch | Germany |
| 10 | Vacuum filter | Zeal, England |
| 11 | PH metre | Germany |
| 12 | Refractometer | Japan |
| 13 | filter paper | England |
| 14 | Measuring cylinder/tortlestand | England |
| 15 | burrette/pipette | England |
| 16 | Bottom flask, round bottom flask beaker conical flask | |
| 17 | Thermometer(N2 filled ) | England |

**TABLE 2**

## LIST OF CHEMICALS AND MATERIAL

| CHEMICAL/MATERIALS | MANUFACTURALS/SOURCE |
|---|---|
| 1 Acetone | National oil and chemical marketing PLC |
| 2 Ethanol | " |
| 3 Petroleum ether | " |
| 4 Eucalyptus leave | Nigeria |
| 5 Perubalsam(alcoholic extract) | Italy |
| 6 frankincense | Italy |
| 7 Myrrh | Italy |
| 8 Labdanum | Italy |

## DIAGRAM OF SOXHLET EXTRACTION EQUIPMENT SET UP

thermometer adapter

distilling
head

condenser

vacuum adapter

distilling flask

open to air

water

receiving
flask

supporting
blocks
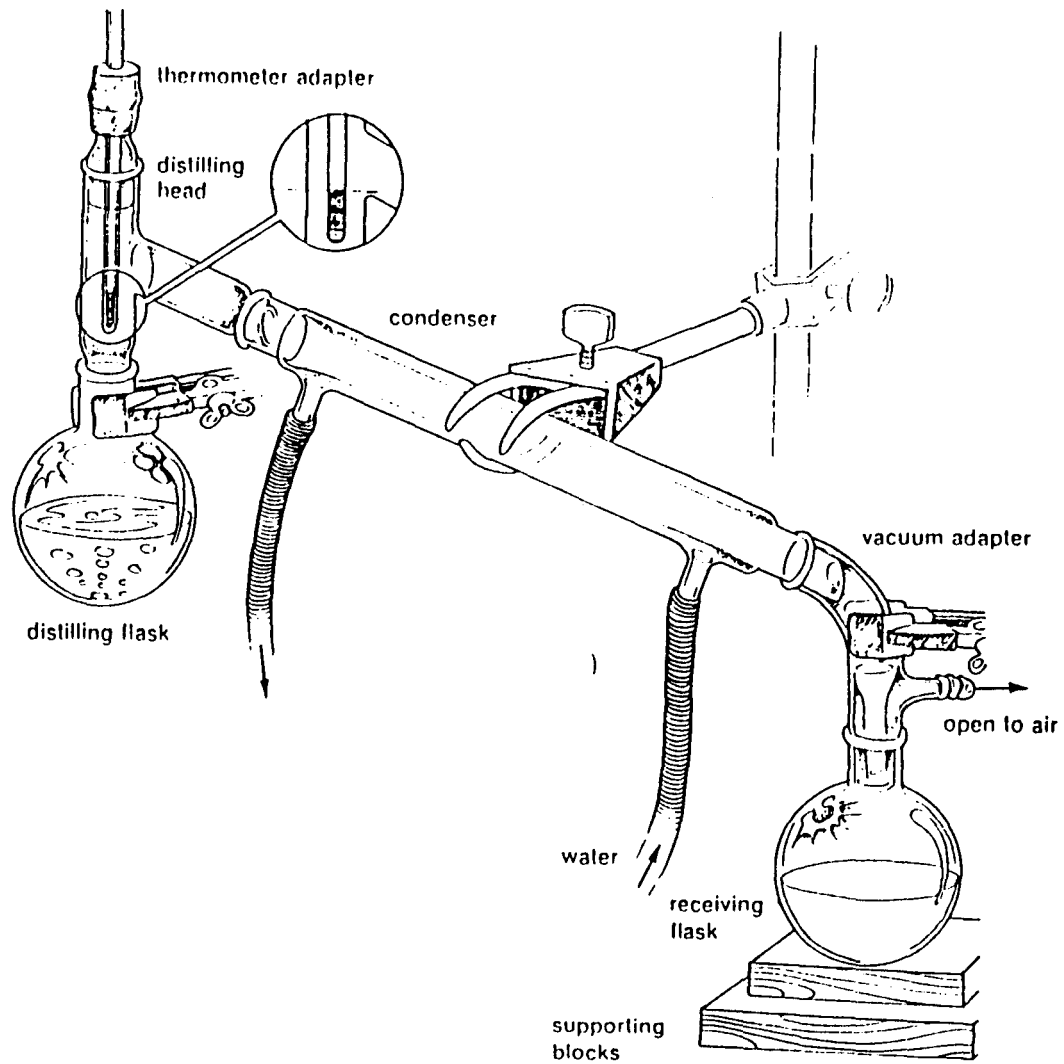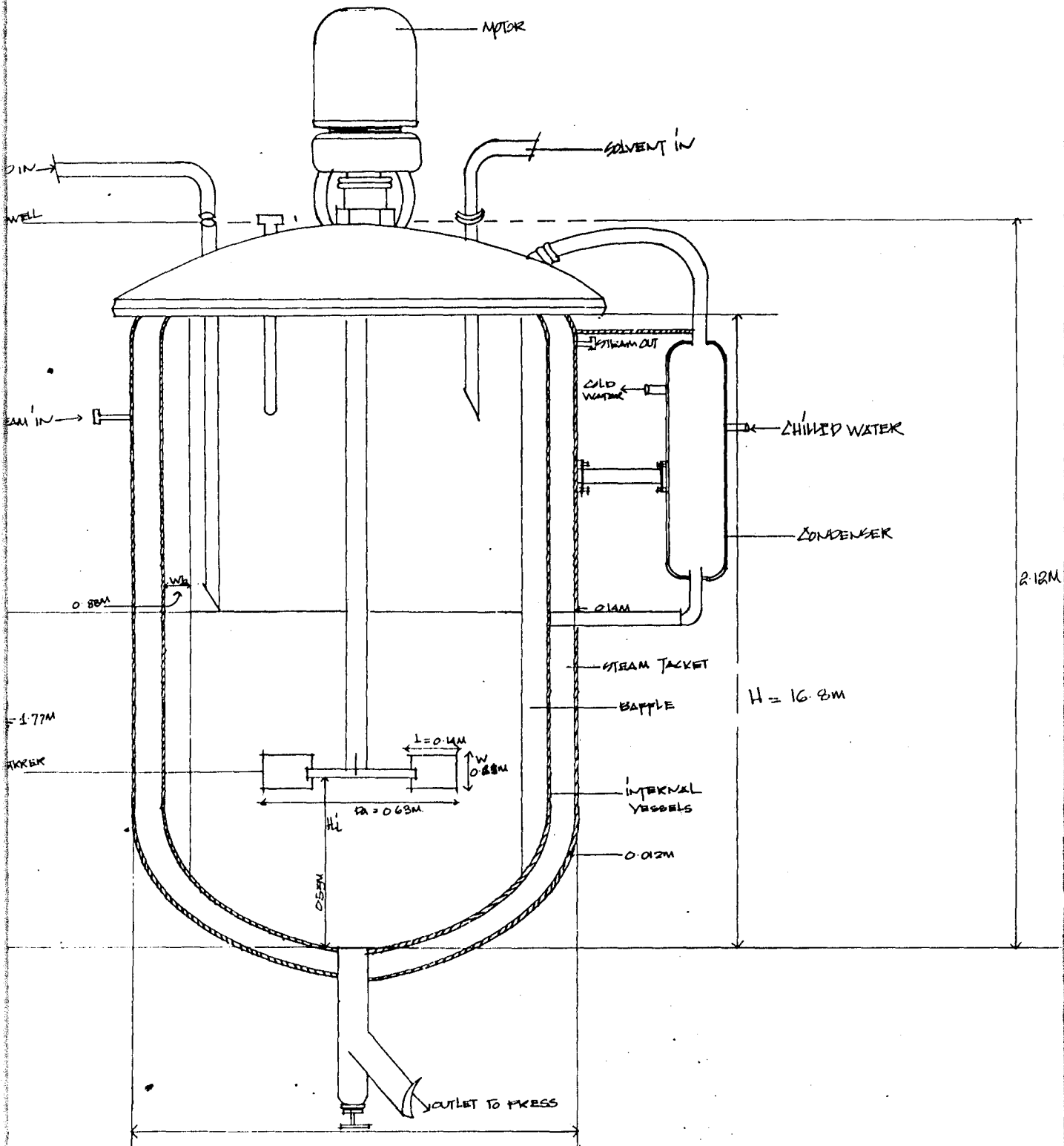
DIAGRAM SHOWING THE DESIGNED EXTRACTOR

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# EXPERIMENTAL PROCEDURE AND CHARACTERISATION OF ESSENTIAL OILS

This chapter described the various experimental procedure and analysis carried out for the purpose of this research work.

## 3.4.1 PRETREATMENT AND SIZE REDUCTION

Fresh leaves of eucalyptus where obtained,the leaves where then rinse,dried and grinded into fine particles for easy extraction.

## 3.4.2 EXTRACTION WITH SOLVENTS

Using continous extraction with soxhlet·apparatus in extracting the solution.

## 3.5 EXTRACTION OF ESSENTIAL OIL FROM PLANT SPECIES USING SOXHLET EXTRACTION APPARATUS

PROCEDURE:The grounded eucalyptus leaves was seived using seiveing machine into various particles size (850$\mu$m,710$\mu$m,500 -m,250 -m) and 2g of each particles were used as sample.

The filter paper and filter paper particle were weight and their weight(F1)and (F2) were noted respectively.Carefully,2g of each particle size was transferred into the filter paper and the weigth of the filter paper plus the particle (F2) and the weigth of particle(F1) were noted.

The 2g of each particle size were inserted into the xetraction at different hours

300ml of volume of the solvent (petroleum ether and ethanol /acetone) was poured into the flask

The extractor was then connected to a condenser above it  and flask at it bottom ,heated by heating mantle set at 70$^0$Cin each of the flask (see the appar atus arrangement in chapter three page)

This heat made the solvent in the. flask to evaporate and condense by aconden ser into the extractor in which a very light green colour of a solution was obse vered in the extractor. This coloured slution rose up to the level of the extractor capillary

### 3.5.1 SOLVENT RECOVERY

The filter paper was removed from the extractor, after the apparatus was disconnected. The apparatus was connected back without the filter paper, for recovery of the solvent and to obtain the oil the solvent of the solvent flask and condensed into the extractor, but this time the condensed into the extractor was not allowed to react the level of the capillary tube of the extractor when it reached one - third (1/3) of the capillary the height , the extractor was removed and the solvent inside it was poured into the measuring cylinder and the volume noted then poured into it container.This process continued until the solvent was recovered, and in the flask only the oil was left with small amount of solvent.

### 3.5.2 FILTERATION

A 850 air compressor vacuum filter was used for his purpose. A perforated glass funnel was place on a conical flask having a projected mouth at the neck. Filter paper was placed on the perforations. A project mouth of the flask and the compressor. Solution of the sample was then introduced in the funnel and the power swithed on. Vaccum was created and sucking continuous until all the solution is separated from inert solids. The inert solids were collected, dried and weighed.

### 3.6 PREPARATION OF RESINOIDS

#### LABDANUM

130g of grinded labdanum was put into a bottle and 80ml of acetone was then added. This was allowed to stand for three weeks until acetone become saturated with labdanum. This was then filtered by simple filteration.

### 3.6.1 FRANKINCENSE

80g of frankincense was put into a beaker and about 100ml of water was added the solution was then heated to about 50°C when the frankincense was totally dissolved it was allowed to cool then filtered.

### 3.6.2 MYRRH

80g Myrrh (Soft gum) was put into a beaker and about 100ml of water was added. The solution was then heated to about 50°C when the Myrrh was totally dissolved it was allowed to cool then filtered.

### 3.6.3 PERUBALSAM DEODOURIZATION

150g of labdanum was introduced into 150ml of perubalsam. This was then left for two weeks. The resulting solution being odourless alcohol.

## 3.7 PERFUME FORMATION

### PERCENTAGE COMPOSITION BY WEIGHT USING EUCALYPTUS OIL BASE AS PERFUME CONCENTRATES.

### SAMPLE

| COMPONENT | % WE | GRAMME |
|-----------|------|--------|
| Perubalsam | 30.0 | 30g |
| Labdanum | 35.0 | 35g |
| Eucalyptu Oil | 22.0 | 22g |
| Frankincense | 8.0 | 8g |
| Myrrh | 5.0 | 5g |
|  | ----------------- |  |
|  | 100 |  |

## 3.8 CHARACTERIZATION OF THE PLANT SPECIES OIL

### SPECIFIC GRAVITY

PROCEDURE:

Empty density bottle ($W_d$) was washed, dried in an oven and its weight was noted. The weight of distilled water ($W_w$) was noted afterfilling the empty density bottle with distilled water. The temperature was also noted. The essential oil extracted from the plant and the weight of the oil ($W_o$) was noted and the filled inside

## SPECIFIC GRAVITY OF EUCALYPUS OIL

Weight of empty bottle ($W_d$) $= 27.02$g

Weight of densiy bottle t$H_2$O($W_e$) $= 74.52$g

Weight of $H_2$O ($W_w$) = ($W_e$ - $W_d$) $= 47.5$g

Temperature of $H_2$O ($t_w$) $= 28$°C

Weight of density bottle + oil ($W_i$) = 71.62g

Weight of oil ($W_o$) = ($W_i$ - $W_d$) = 44.6g

Temperature of oil ($t_o$) = 29.5₀C

$$S.g = \frac{W_o}{W_w (H \quad (t_o - t_w)}$$

where $= 0.00155$ (i.e expensity of oil or fat in general,

$$sg = \frac{44.6}{47.5 (1 + 0.000155 (29.5 - 28)}$$

$$= \frac{44.6}{47.51} = 0.939$$

s.g of Eucalyptus oil = 0.939

======

## MATERIAL BALANCE AROUND THE PROCESS

Material in = Material out

$F_1$ $= H_1 + F_2$

3000 $= 900 + 2100$

3000 $= 3000$

the density bottle that was emptied and dried the temperature of the oil ($t_o$) was noted. The specific gravity of the oil was calculated as

$$S.g = \frac{W_o}{W_w (1 + \propto (t_o - t_w))}$$

where $\propto$ = 0.000155 (expansivity of oil o fat in general)

## 3.8.1 VISCOSITY

Visco tester rotatory arm is attached directly to the scale which is in kg/ms. The extracted oil in the viscotester container was heated to 36°C and then attached to the hook of the rotatory arm which rotates within the container and the viscosity was recorded directly from the scale as $1.94 \times 10^{-3}$ kg/ms of Eucalyptus.

## 3.8.2 THE PH OF EXTRACTED OIL

The PH of extracted oil was determined, selected and the electrodes were lowered into buffer solution. The temperature control was adjusted to temperature of buffer. The calibrate control was adjusted, and the meter indicates exact PH of buffer. The electrodes were raised and rinsed with buffer, then with the oil. The temperature control was adjusted to temperature of the extracted oil. The electrodes were then lowered into the extracted oil and the meter reading was noted as 3.23 (25°C) of Eucalyptus.

## 3.8.3 BOILING POINT

The boiling point of the extracted oil was determined by this procedure.

The oil in a beaker and thermometer immersed in it was heated on a heating mantle after 12 minutes of heating, it was observed that the oil in the beaker started circulating and immediately the temperature was noted as 176.4°C of Eucalyptus.

## 3.8.4 MELTING AND SOLIDIFICATION TEMPERATURE OF OIL

The extracted oil was converted to solid oil using ice blocks with a thermometer immersed in it and the temperature was noted as 2.6°C, 1.8°C and 3.9°C for Eucalyptus. That is then solidification temperature after the solidified oil was

then melted over a boiling water batch and the temperatue at which it returned to liquid form was noted 1.5°C of Eucalyptus.

### 3.8.5 REFRACTIVE INDEX

**PROCEDURE:**

Few drops of oil were placed on the face of the prism of refractometer and gently spreaded, closed and tightened Ample time was allowed for the oil and the prism to attain a steadly temperature. The refractive index was then read from the demarcation line as 1.3652.

# CHAPTER FOUR

## TABLE 4.1

| Time hour | particle size | weight of particle beforee xtractio n(wpb) | weight of filter paper(F 1) | weight of filter +particl e(F2) | weight of particle after extracti on(wpa ) | weight of extract (we)= 2- wpa | % yield |
|---|---|---|---|---|---|---|---|
| 1 | 850 | 2 | 2.532 | 4.118 | 1.586 | 0.414 | 20.7 |
|   | 710 | 2 | 2.813 | 4.672 | 1.859 | 0.141 | 7.05 |
|   | 500 | 2 | 2.678 | 4.020 | 1.342 | 0.658 | 32.9 |
|   | 250 | 2 | 2.721 | 4.134 | 1.413 | 0.587 | 29.35 |
| 3 | 850 | 2 | 2.478 | 3.767 | 1.289 | 0.711 | 35.55 |
|   | 710 | 2 | 2.729 | 3.944 | 1.215 | 0.785 | 39.25 |
|   | 500 | 2 | 2.628 | 3.784 | 1.156 | 0.844 | 42.20 |
|   | 250 | 2 | 2.646 | 3.909 | 1.263 | 0.737 | 36.85 |
| 5 | 850 | 2 | 2.499 | 3.766 | 1.267 | 0.733 | 36.65 |
|   | 710 | 2 | 2.727 | 3.910 | 1.183 | 0.817 | 40.85 |
|   | 500 | 2 | 2.626 | 3.780 | 1.154 | 0.846 | 42.30 |
|   | 250 | 2 | 2.737 | 3.854 | 1.117 | 0.800 | 40.00 |

* For the procedure on how the above varies were obtained see Appendix `A'
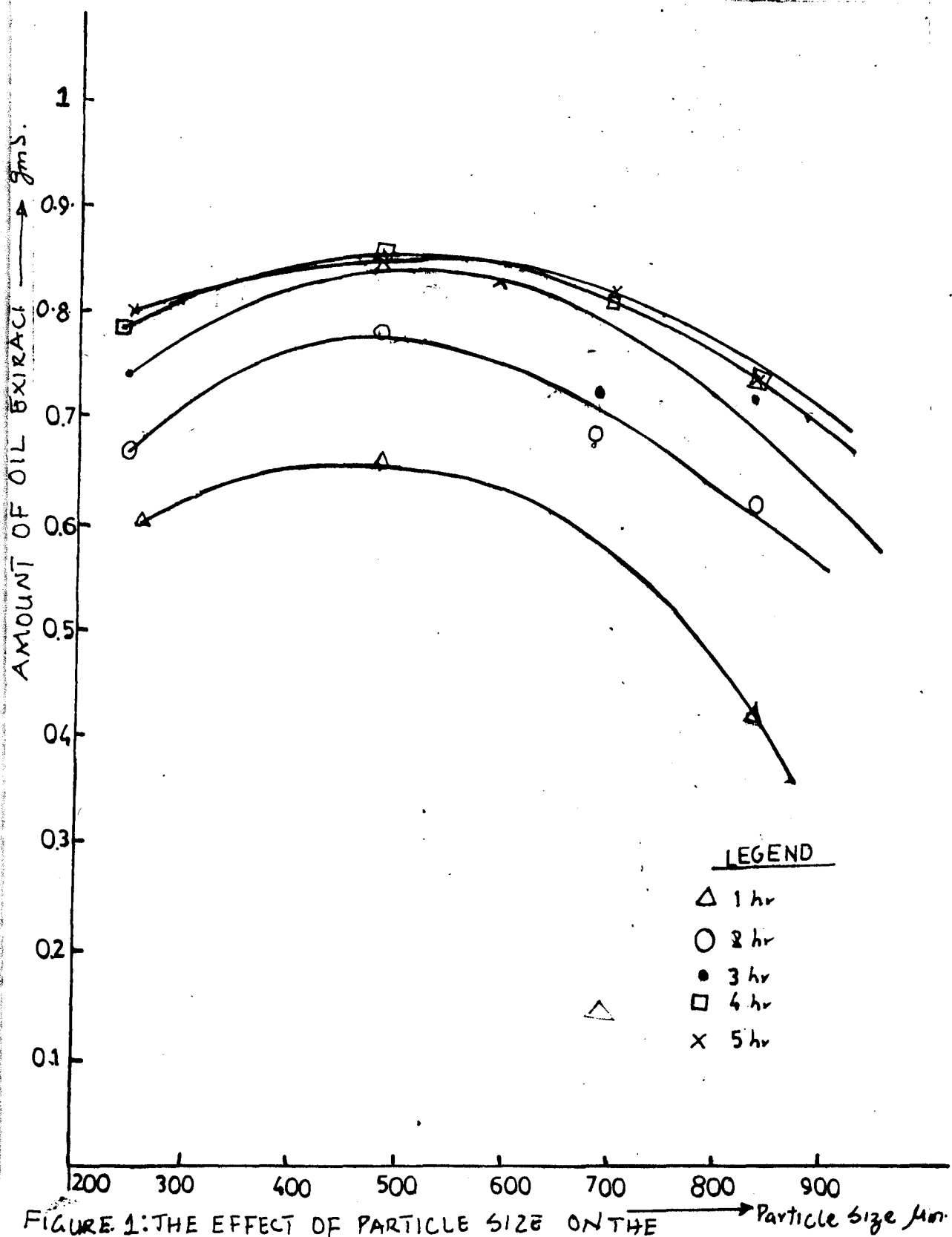
FIGURE 1: THE EFFECT OF PARTICLE SIZE ON THE
AMOUNT OF OIL EXTRACTED.
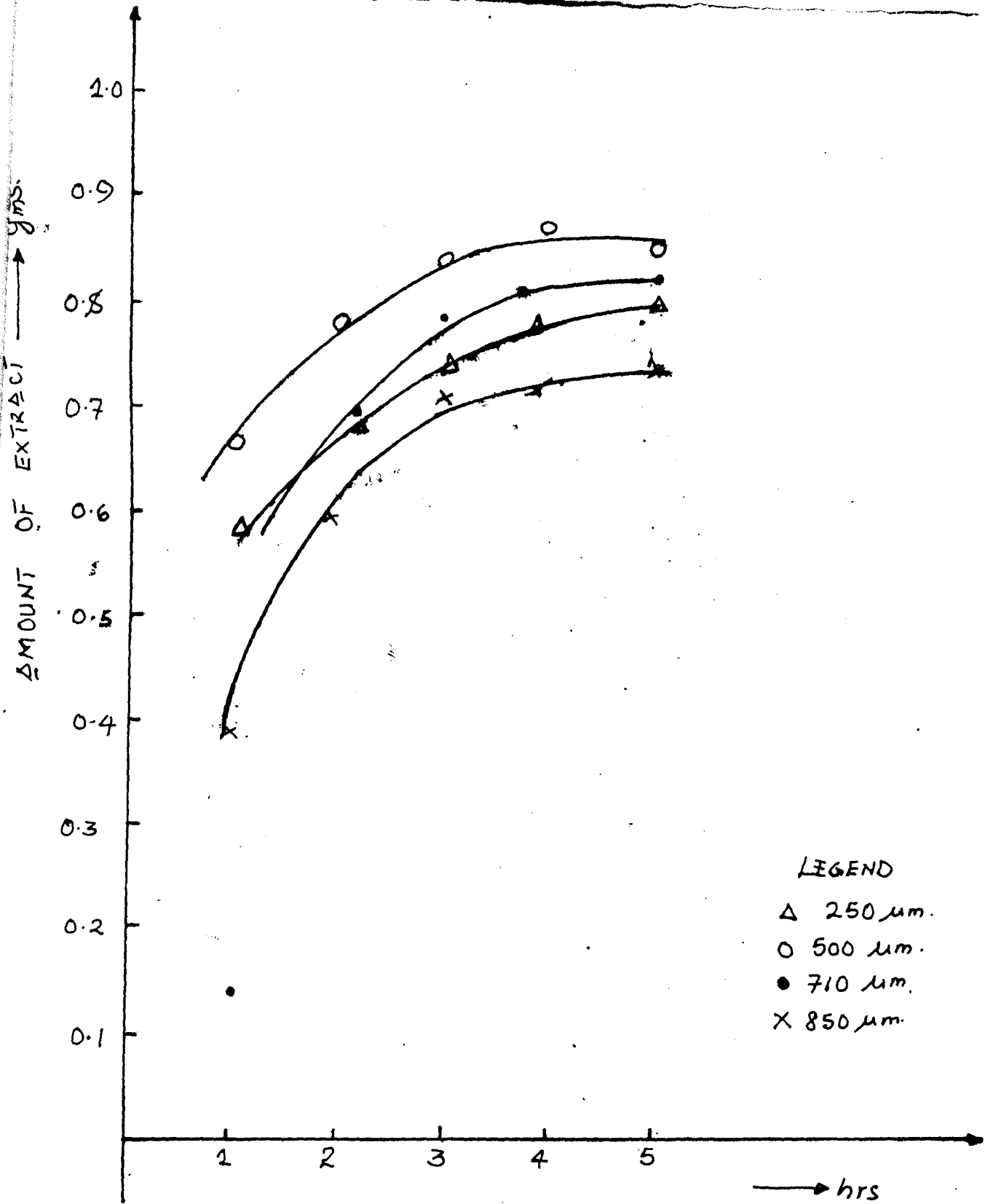
FIGURE 2: THE EFFECT OF TIME ON THE AMOUNT OF OIL EXTRACTED.

LEGEND

△ 250 μm.
O 500 μm.
● 710 μm.
X 850 μm.

# CHAPTER FIVE

## DESIGN OF AN EXTRACTOR

## 5.0 EQUIPMENT SIZING AND DESIGN SPECIFICATION

volume of the vessel = volume of the cylinderical part + volume of hemi elliposodial part

volume of cylinderical part = $\pi(D_T/2) + 3/4\ D_T$

volume of hemi - elliposdial part = $1/6 \times 1/4\ \pi D^3_T$

therefore volume of the vessel = $4m^3$

$4 = \pi(D_T/2)^2 \times 3/4\ D_T + 1/6 \times 1/4\ \pi D^3_T$

$4 = 3\pi D_T/16 + \pi D_T/24$

$4 = \pi D_T\ (3/16 + 1/24)$

$4 = 11/48\ \pi D_T$

$4 \times 48/11\pi = D_T$

$D_T = (4 \times 48/11 \times \pi)1/3 \qquad = \qquad 1.77mm$

Diameter of tank $D_T = 1.77m$

From the formula for geometric characteristic f vessels.

For two (2) blade paddles.

$D_T/Di = 2.82\ (=3)$ where Di is agitator diameter

$===> Di = D_T /2.82\ = 1.771/2.82 = 0.628 \qquad 0.63m$

**Critical stirrer speed calculation**

from dimension less

$\Omega c\ Di^{0.85} = SV^{0.1}\ D_p^{0.2}\ (gd\rho/\rho\ )^{0.45}\ \beta^{0.13}$

Where

$n_c$ = critical stirrer speed = ?

Di = agitator diameter = 0.63m

S = shape factor = 8 [for 2 blades]

V = kinematic viscosity

= Dynamic viscosity/density = 0.61 cp/866 = $6.23 \times 10^{-7}NS$

Dp = average particle size = 500um = $5 \times 10^{-4}m$

1

$g$ = gravitational acceleration = $9.81 ms^{-2}$

$\Delta \rho$ = density difference = $200 kg/m^3$

$\rho$ = liquid densities = $866 kg/m$

$\beta$ = 100 x weight of solid/weight of liquid

$\qquad$ = $100 \times 2000/80 = 2500$

$n_c (0.63)^{0.85} = 8 \times (6.23 \times 10^{-7})^{0.1} (5 \times 10^{-4})^{0.2}(9.81 \times 200/866)^{0.45} \times (2500)^{0.13}$

$n_c = 1.6954/(0.63)^{0.85} = 2.48 rev/s$

## 5.1 $\qquad$ POWER REQUIREMENT

power required P is

$$P = N_p \, n_c^3 \, Di^5 \, \rho/g_c$$

where $N_p$ = power number [for 2 blade $N_p$ = 2.5]

$\Longrightarrow$ $P = N_p \, n_c^3 \, Di^5 \, \rho/0.1 \times 9$

$\qquad$ = $2.5 \times (2.48)3 \times (0.63)5 \times 866/0.1 \times 9.81 = 346W$

$\qquad$ = $3.468kW$

Now we find the following parameters for the mixture

$\qquad \mu \qquad$ = $0.61 \times 10^{-3}$ NS/m² [for ethyl alcohol] - $0.61C_p$

## CALCULATION FOR THERMAL CONDUCTIVITY OF THE MIXTURE

$$K_{mix} = w_p k_p + w_c k_c/w_p + w_c$$

where $w_p$ = weight of the solvent = 80kg

$\qquad w_c$ = weight of the feed = 2000kg

$\qquad k_p$ = thermal conductivity solvent = 1.8 x 10-4

$\qquad K_c$ = thermal conductivity of feed = 2.8 x 10-4 [for cellulose]

$K_{mix}$ = 80 x 1.8 x $10^{-4}$ + 2000 x 2.8 x $10^{-4}$/200 + 800

$K_{mix}$ = 2.762 x $10^{-4}$ KJ/ms° C = 2.762 x $010^{-4}$kw/m°C

## CALCULATION FOR HEAT CAPACITY OF THE MIXTURE

$Cp_{mix} = w_p + w_c C_c/w_p + w_c$

where $C_p$ = heat capacity of the solvent = 2.3

$C_c$ = heat capacitor of the feed = 2.5

$\Longrightarrow Cp_{mix}$ = 80 x 2.3 + 2000 x 2.5/2000 + 80 = 2.492KF/kg°C

therefore,

$\mu$ = 0.61 x 10$^{-3}$ Ns/m² [for ethylalcolhol]

$K_{mix}$ = 2.762 x 10$^{-4}$ KJ/ms°C

$Cp_{mix}$ = 2.492 KJ/kg°C


## 5.2      TIME OF ISOTHMAL HEATING

The time required to head 4m³ of reaction mixture from 30°C to 70°C in a jacketed, 1.77m agitated vessel. The vessel is equipped with a 0.92m diameter 2 blades that paddle (45°) impeller at 2.48rev/s. The vessel is assumed to e clean and free from fouling films and heated by 100°C steam.

$\mu$ (30° C) = 1.1$C_p$ = 1.1 x 10$^{-3}$ Ns/m²

$\mu$(50° C) = 0.75$C_p$ = 0.75 x 10$^{-3}$ Ns/m²          For ethyl alcohol

$\mu$(70° C) = 0.5$C_p$ = 0.5 x 10$^{-3}$ Ns/m²

REYNOLD'S NUMBER CALCULATION FOR THE ABOVE TEMPERATURE

Using $N_{Re} = \rho N^{Di}/\mu$

where N = $n_c$

Therefore,

$N_{Re}$ (30°C) = 866 x 2.51 x (0.63)2/1.1 x 10$^{-3}$    = 784296

$N_{Re}$ (50°C) = 866 x 2.51 x (0.63)2/0.5 x 10$^{-3}$ = 1725451

PRANDTL NUMBER CALCULATION

$N_{Pr} = Cp_{mix}\mu/K_{mix}$

$N_{Pr}$ (30°C) = 2.492 x 1.1 x 10-3/2.762 x 10$^{-4}$ = 9.93

$N_{Pr}$ (50°C) = 2.492 x 0.75 x 10$^{-3}$/2.762 x 10$^{-4}$ = 6.77

$N_{Pr}$ (70°C) = 2.492 x 0.5 x 10$^{-3}$/2.762 x 10$^{-4}$ = 4.51

## INSIDE HEAT TRANSFER COEFFICIENT CALCULATION (hi)

## FOR THE ABOVE TEMPERATURES

Using

$h_i$ (approximate) = $0.73 (N_{Re})^{0.65} (N_{Pr})^{0.33} (K/D_T)$

($h_i$ is in Btu/hr ft$^2$ °F)

hi (30°C) = $0.73 (784296)^{0.65} (9.93)^{0.33} (2.762 \times 10^{-4}/1.771) = 2.2564 kW/m^2°C$

hi(50°C) = $0.73 (1150300)^{0.65} (6.77)^{0.33} (2.762 \times 10^{-4}/1.771) = 1.8619 W/m^2°C$

hi(70°C) = $0.73 (1725451)^{0.65} (4.51)^{0.33} (2.762 \times 10^{-4}/1.771) = 2.1193 kW/m^2°C$

## INTERNAL WALL OF THE VESSEL CALCULATION

Using

$T_w = T_s - [T_s - T_b/1 + h_s/h_i]$

where

$T_w$ = temperature of the internal wall

$T_s$ = Temperature of steam at 100C

$T_b$ = Temperature of the bulk

$h_s$ = heat capacity of steam

$h_i$ = Heat transfer coefficient approximates

From literature,

$h_s$ = 1000Btu/hr Ft$^2$°F

= $1000 \times 5.6783$ w/m$^2$ °C = 5.6783kw/m$^2$°C

Therefore,

At $T_b$ = 30 C

$T_w$ = 100 - [100 - 30/1 + 5.6783/2.2564] = 100 - 70/3.5165 = 80.09°C

At $T_b$ = 50°C

$T_w$ = 100 - [100 - 50/1 + 5.6783/1.8619 = 100 - 50/4.0497 = 87.65°C

At $T_b$ = 70°C

$T_w$ = 100 - [100 - 70/ 1 + 5.6783/2.1193] = 100 - 30/3.6793 = 91.85°C

## CALCULATION OF VISCOSITY NUMBER AT TEMPERATURE

## OF THE INTERNAL WALL

Using

$$N_{vis} = \mu_w/\mu_b$$

where

$N_{vis}$ = Viscosity number

$\mu_w$ = viscosity at the temperature of the wall

$\mu_b$ = Viscosity at the temperature of the bulk

Therefore

At $T_b$ = 30°C and $T_w$ = 80.09°C

$N_{vis}$ = 0.487$C_p$/1.15$C_p$ = 0.4235

At $T_b$ = 50°C, and $T_w$ = 87.65°C

$N_{vis}$ = 0.443/0.75Cp = 0.59

At $T_b$ = 70°C and $T_w$ = 91.85°C

$N_{vis}$ = 0.397Cp/0.575$C_p$ = 0.69

## RECALCULATION OF HEAT TRANSFER COEFFICIENT

## USING NULSELT NUMBER RELATION RELATIONSHIP

Using

$$N_{vis} = 0.73 \ (N_{Re})^{0.65} \ (N_{Pr})^{0.33} \ (N_{vis})^{-0.24} \text{ and } N_{vis} = h_i \ D_T/K$$

where $N_{nu}$ = Nulselt number

from equation (1) and (2)

$h_i = 0.73 \ (N_{Re})0.65 \ (N_{Pr})^{0.33} \ (N_{vis})^{-0.24} \ (K/D_T)$

$h_i (30°C) = 0.73 \ (784296)^{0.65} \ (9.93)^{0.33} \ (0.4235)^{-0.24} \ (2.762 \times 10^{-4}/1.771)$

$\qquad = \quad 2.0244 kW/m^{2}°C$

$h_i (50°C) = 0.73 \ (1150300)^{0.65} \ (6.77)^{0.33} \ (0.50)^{-0.24} \ (2.762 \times 10^{-4}/1.771)$

$\qquad = 2.1132 KW/m^{2}°C$

# CALCULATION OF OVERALL HEAT TRANSFER COEFFICIENT

Using

$$1/\mu_i = 1/h_i + x/K + 1/h_s$$

where

x = thickness of vessel = 10mm + 20% safety factor

$$= 1.2 \times 10^{-3}m$$

K = thermal conductivity for steel vessel

$$= 9.4 \text{ Btu/hr ft}^2 \text{ }^\circ F/ft$$

$$= 9.4 \times 1.73073 = 16.2686 W/m^\circ C$$

$$= 0.0163 KW/m^\circ C$$

Therefore,

$\mu_i$ (at $T_b = 30^\circ C$)

$1/\mu_i = 1/2.0224 + 1.2 \times 10^{-3}/0.0163 + 1/5.6783 = 0.7437kW/m^\circ C$

$\mu_i = 1.3446kW/m^\circ C$

$\mu_i$ (at $T_b = 50^\circ C$)

$1/\mu_i = 1/2.1132 + 1.2 \times 10^{-3}/0.0163 + 1/5.6783 = 0.72294$

$\mu_i = 1.3832kW/m^\circ C$

$\mu_i$ ($T_b = 70^\circ C$)

$1/\mu_i = 1/2.3167 + 1.2 \times 10^{-3}/0.0163 + 1/5.6783 = 0.6814kW/m^\circ C$

$\mu_i = 1.4675kW/^\circ C$

Graph of $\mu_i$'s against $T_b$ is plotted and $\mu_i$ located at the $T_b$ of 30, 50, and 70°C. Their

ranges is divided into 5 equal parts which is 8°C at interval and the values of their overall heat

transfer coefficient $\mu_i$ at the midpoint of each       range is read, that is,

$\mu_i$ (at 40°C) = 1.35kw/m°C, $\mu_i$ (at 60°C) = 1.14kW/m°C

See F.S. Champmann Lever brothers Company N.Y. Liquid Mixing and processing in stirred

tanks page 176 - 300.

## CALCULATION OF HEAT TRANSFER AREA CONTACTED BY THE LIQUID

$A_i$ = area of the cylinderical part + area of the = $\pi D_T (D_T - D_T/4) + \frac{1}{2} [2\pi(D_T/4)^2 +$

$2\pi \ (D_T/4 \ D_T/2) \ \text{Sine}^{-1}\rho/\rho$

where

$$\rho = [(D_T/2)^2 - (D_T/4)^2]^{\frac{1}{2}}/D_T/2$$

$$= \ [(1.771/2)^2 - (1.771/4)^2]^{\frac{1}{2}}/1.771/2$$

$$= \ (0.588206)^{\frac{1}{2}}/0.885569 = 0.76694/0.885569$$

$$\rho \quad = \quad 0.866$$

Therefore,

$A_i = 1.771 \ [1.771 - 1.771/4] + \frac{1}{2} \ [2\pi(1.771/4)^2 + 2\pi(1.771^2/8) \ \text{Sin}^{-1} \ 0.866$

$$= \quad 7.39006 + \frac{1}{2} \ [1.23167 + 2.97863]$$

$$= \quad 7.39006 + 2.10511 = 9.495m$$

## 5.8 TIME REQUIRE TO HEAT THE BULK MADE

## OVER A TEMPERATURE INCREMENT

Using

$$\Delta_t \ (\text{in hours}) = WC_p/\mu_iA_i \ \text{loge} \ )T_s - T_{bi}/T_s - T_{bf})$$

where

$T_{bi}$ = initial batch temperature at time interval dt

$T_{bf}$ = final batch temperature at time dt(hr)

w = Weight of the bulk liquid in the vessel in kg

$C_p$ = Specific heat capacity at constant pressure of the bulk liquid in KJ/kg C

W = 2080kg

$C_p$ = 2.492KJ/kg C

$T_s$ = 100 C

From (30 - 38°C), $\mu_i$ at midpoint = 1.34kW/m²°C

(38 - 46°C) $\mu_i$ at midpoint = 1.36kW/m²°C

(46-54 °C) $\mu_i$ at midpoint = 1.38kW/m²°C

(54 - 62°C) $\mu_i$ at midpoint = 1.42kW/m²°C

(62 - 70°C) $\mu_i$ at midpoint = 1.46 kW/m²°C

## CALCULATION OF THE REQUIRED HEAT

## FROM THE ABOVE RANGES

$\Delta_t$ (30 - 38°C) = 2080 x 2.4942/1.34 x 9.495 log (100 - 30/100 - 38)

$= \quad$ 49.4849secs = 0.014hr

$\Delta_t$ (38 - 46°C) = 2080 x 2.4942/1.36 x 9.495 log (100 - 38/100 - 46)

$= \quad$ 55.501 secs = 0.015hr

$\Delta_t$ (46 - 54°C) = 2080 x 2.4942/1.38 x 9.495 log (100 - 46/100 - 54)

$= \quad$ 63.48 secs = 0.018hr

At (54 - 62 C) = 2080 x 2.4942/1.42 x 9.495 log (100 - 54/100 - 62)

$= \quad$ 73.514secs = 0.020hr

At (64 - 70 C) = 2080 x 2.4942/1.46 x 9.495 log (100 - 62/100 70)

$= \quad$ 88.465 = 0.025hr

Total time required to heat the batch from 30 C -70 C is the sum of the individual time increment over the 80 C ranges

$\Delta_t$ total = $\Delta_t$ (30 - 38°C) + $\Delta_t$ (38-46°C) + (46 - 54° C) + $\Delta_t$ (54 - 62° C) + $\Delta_t$ (62 - 70° C)

$= \quad$ 0.014 + 0.015 + 0.018 + 0.02 + 0.025

$= \quad$ 0.092hr

Therefore, total time required for the mixture to reach the extraction temperature is 0.092hr.

## 5.9                 COOLING TIME CALCULATION

Time required to cool the medium to the extraction temperature of 60 C from 70 C is

$\Delta_t = wC_p/\mu_i A_i$ loge $(T_{b1} - T_c/T_{b2} - T_c)$

where $\mu_i$ = me value of $\mu_i$ from the graph at midpoint of 60°C = 1.44kW/m²°C

$T_c$ = the temperature of the cooling medium which is air at 30°C (temperature of the industrial

$T_{b1} = 70°C$

$T_{b2} = 60°C$

$\Delta_t$ = 2080 x 2.492/1.44 x 9.495 loge (70 - 30/60 - 30)

= 109.06 secs = 0.03hr

**TIME OF HEATING BACK**

Time required to heat the mixture from 60°C back to 70°C

$\Delta_t$ = $W_1C_p/\mu_iA_i$ loge (100 - 60/100 - 70)

= 109.06secs = 0.03hr

## CALCULATION FOR DENSITY OF THE MIXTURE

The density of mixture is $\rho_{mix}$

$\rho_{mix}$ = $\rho_f m_f + \rho_s\ m_s/m_f + m_s$

where $\rho_f$ = feed density

$\rho_s$ = solvent density

$m_f$ = mass of feed

$m_s$ = mass of solvent

$\rho_{mix}$ = 1066 x 2000 + 80 x 866/2000 + 80

= 1058.31kg/m²

## 5.10 PIPE DIAMETER CALCULATION

To calculate the diameter of pipe conveying the various stream we use

$d_p$ = $293G^{0.53}\ \rho^{-0.37}$

where   G = flowrate in kg/s

$d_p$ = pipe diameter

for me mixture flowing from the extractor, a rate of G = 14kg/s is desired

therefore

for the flow rate $G_f$ = 14/2080 x 2000/1 = 13.5kg/s

for the solvent flow rate $G_s$ = 14/2080 x 80 = 0.54kg/s

This implies,

for the solvent pipe

$d_p$ = 293 $(0.54)^{0.53}$ $(866)^{-0.37}$ = 17.304mm

for the feed piped

$$d_p = 294 \, (13.5)^{0.53} \, (1066)^{-0.37} = 88.24mm$$

for the mixture

$$d_p = 293 \, (14)^{0.53} \, (1058.31^{-0.37}) = 90.2mm$$

$$\rho_{mix} = 1058.\, 31kg/m^3$$

$$\mu liq = 0.61 \times 10 \, N/m \, s$$

$$D_T = 1.771m$$

Pipe size = 0.0902m

Total lenght of pipe = 2m (assuption)

Assuming height of extractor base to press = $Z_2 = 0.5m$

Height of the unit = $1.2D_T = 1.2 \times 1.771 = 2.125m$

$$Z_1 = 0.5 + 2.125m$$

$$Z = 0.5 + D_T = 0.5 + 1.771 = 2.271m$$

## 5.11 TIME REQUIRED TO THE EXTRACTOR [DISCHARGE TIME]

(1) $2D^2_T / d^2p = \dfrac{2 \times 1.771^2}{0.0902^2} = 770.99 = 771$

(2) $[Z_1 + P_A + P_B /\rho g]^{\frac{1}{2}}$

where $P_A - P_B = \rho_{air} \, g \, Z = 1.225 \times 9.81 \times 2.271$

$$= 27.29$$

Therefore

(3) $[Z_1 + P_A - P_B/\rho g]^{\frac{1}{2}} = [2.625 + 27.2/1058.31 \times 9.81]^{\frac{1}{2}}$     $= 0.70897$

$$= 0.7090$$

The actual length of conveying pipe to diameter ratio is L/d = 2/0.0902 = 22.17m

Since our two elbows will be at 90°C, from table of roughness of pipe surface, the value of absolute roughness for steel and wrought iron is = 0.00015 x 03048m = 4.57 x 10$^{-5}$ m

The gate valve which will be used for the discharge has equivalent length of pipe to diameter ratio of 7 and that of the elbow is 32 (64 for 2 elbows)from table therefore, the total equivalent length to pipe diameter ratio is L/d = 22.17 + 64 = 93.17

## FRICTIONAL LOAD CALCULATION

Frictional load $h_f = P_A - P_B/\rho g$ $= 27.29/9.81 \times 1058.31$

$$= 2.629 \times 10^{-3}$$

Estimating we have,

$$(R/\rho\mu^2)(N^2_{Re}) = h_f[\rho^2 d^2_p\, g/\, 4[L/d]\mu^2$$

$$= 2.629 \times 10^{-3} \quad [1058.31^2 \times 0.0902^2 \times 9.81]/4 \times 93.17 \times (0.61 \times 10^{-2})$$

$$= 16947\,38.6$$

$$= 1.695 \times 10^6$$

The diamensionless roughness factor is

$$E/d = 4.57 \times 10^{-5}/0.902 = 5.07 \times 10^{-4}$$

Then we calculate for the basic friction factor $R/\rho\mu^2$ From the graph of friction factor against Renolds number and roughness factor [from flow fluid through valves, fittings and pipes, Technical paper No 410 crane industrial products group chicago Illinois USA)

$t/d = 0.000507$ and $(R/\rho\mu^2)(N^2_{Re}) = 2.39 \times 10^6$

The Reynolds number $N_{Re} = 2.7 \times 10^4$

from the above equation

$$R/\rho\mu^2 = 2.39 \times 10^6/N^2_{Re} \qquad = 2.29 \times 10^6/(2.7 \times 10^4)^2$$

$$R/\rho\mu^2 = 3.28 \times 10^{-3}$$

Now

$$1 + 8(R/\rho\mu^2)L/d/2g]^{1/2} = [1 + 8(3.28 \times 10^{-3}) \times 93.17/2 \times 9.81]^{1/2}$$

$$R/\rho\mu^2 = 3.28 \times 10^{-3}$$

Now $[1 + 8(R/\rho\mu^2)L/d/2g]^{1/2} = [1 + 8(3.28 \times 10^{-3}) \times 93.17/2 \times 9.81]^{1/2}$

$$= \quad 0.419$$

Therefore, the time required to empty the extractor (discharge time) is

$$t = [Z_1 + P_A - P_b/\rho\, g]^{1/2} - [Z_2 + P_A - P_B/\rho g/^{1/2}\, 2D^2_T/d_p\, [1 + 8\,(R/\rho\mu^2)L/d)/2g]^{1/2}$$

$$= \qquad (1.9017 - 0.2096) \times 1654.08 \times 0.419$$

$$= \qquad 826.20 \text{ secs} = 0.23\text{hr}$$

**PIPE SPECIFICATION**

| Material | Inside diameter of pipe (in) | Outside diameter | Normal pipe size (in) | Wal thickness (in) | Schedule |
|----------|------------------------------|------------------|------------------------|---------------------|----------|
| Feed | 3.48 | 4.5 | 4 | 0.531 | 160 |
| Solvent | 0.68 | 0.84 | 0.5 | 0.083 | 10s |
| Bulk fluid | 3.55 | 4.0 | 3.33 | 0.226 | 40 st, 40s |

Impeller diameter to tank $D_i/D_T = 1/2.82 = 0.355$

Baffle width to tank diameter ratio, $W_b/D_T = 0.1$

$$W_b = 0.1D_T$$

$$= 0.1 \times 1.771 = 0.1771$$

Height of impeller from tank bottom to tank diameter ratio $H_i/D_T = 0.3$

## 5.12 CALCULATION OF BUCKLING PRESSURE

Thin - welled vessel subjected to external pressure are liable to failure that determine the wall thickness for short closed vessel and long vessel with stiffening rings the critical buckling pressure is given by

$$P_c = K_c E (t/D_o)^3$$

where $K_c$ is a function of the diameter and thickness of the vessel.

$E$ = Young modulus for steel = $2.14 \times 10^{11}$

$L/D_o = 1.2D_T/D_o$

where L = length of the extractor

$D_o$ = Outside diameter of vessel

$t$ = thickness of the vessel

Outside diameter $D_o$ is determine as

$$D_o = 2 \times t + D_T$$

$$= 2 \times (1.2 \times 10^{-3}) + 1.771 = 1.795$$

$L/D_o = 1.2D_T/D_o = 1.2 \times 1.771/1.7950 = 1.1839$

to determine $K_c$, we determine $D_o/t$

But $t/D_o = 12 \times 10^{-3}/1.795 = 6.69 \times 10^{-3}$

$D_o/t = 149.58$

From table of collapse coefficient of cylinderical shell after brownell and young 1959,

we find $K_c$ at $D_o/t = 1477.83$ and $L/D = 1.1984$

Therefore $K_c = 36$

This implies the buckling pressure $P_c$

$$P_c = 36 \times 2.14 \times 10^{11} \times (149.58^{-1})^3$$

$$= 2.3 \times 10^6 \, KN/m^2$$

## HEAT BALANCE AROUND THE CONDENSER

$$UA.\Delta T = ML$$

where

$\mu = \mu_i$ at $65°C = 1.41 KN/m°C$

A = Area of the extractor

$\Delta T$ = Final temperature - initial temperature

$$= 70 - 30°C = 40°C$$

m = mass flow rate of the solvent and

L = latent heat of vapourization of ethanol at

$$70°C = 8440.51 KJ/Kg$$

$A = A_i = 9.495m$

Therefore, the mass flow rate m

$$m = UA_1D_T/L = 1.41 \times 9.495 \times 40/8440.51$$

$$= 6.35 \times 10^{-2} \, kg/hr$$

### HEAT BALANCE THROUGH THE CONDENSER

$$q = m (H_b - H_a)$$

where

$H_a$ = enthalpy of solvent at extractor after cooling condenser

$H_b$ = enthalpy of solvent vapour at exit

Therefore, $H_x = C_p (T - 25°C)$

where

$$x = a \text{ or } b$$

$C_p$ = specific heat capacity of the solvent

T = Temperature at a or b

$\Longrightarrow$ $H_a$ = 2.3 (30 - 25)

$\qquad$ = $\qquad$ 2.3 x 5 = 11.5J/kg

$H_b$ = $C_p$ (70 - 25)

$\qquad$ = $\qquad$ 2.3 x 45 = 103.5 KJ/kg

So, the rate of heat balance through the exchange condenser

$\qquad$ q = m ($H_b$ - $H_a$)

$\qquad$ = $\qquad$ 0.0635 (103.5 - 11.5)

$\qquad$ q $\quad$ = $\quad$ 5.842KW

the energy required by condenser to cool the solvent is 5.842KW

## 5.13 DETERMINATION OF BAFFLE LENGTH

From

$\qquad$ $W_b/D_T$ = 0.10

$\Longrightarrow$ $W_b$ = 0.10 x $D_T$

$\qquad$ = 0.10 x 1.771

$W_b$ = 0.1771m

The length of baffle = 0.1771m

## CALCULATION OF THE HEIGHT OF IMPELLER ABOVE

## THE BOTTOM OF THE VESSEL

$\qquad$ $H_i/D_T$ = 0.30

$\Longrightarrow$ $H_i$ = $D_T$ x 0.30

$\qquad$ = 1.771 x 0.30

$\qquad$ $H_i$ = 0.5313m

The height of impeller above bottom of vessel $H_i$ = 0.5313m

## DETERMINATION OF FULL JACKETED PRESSURE

$\qquad$ Jacketed pressure = inner pressure of vessel - pressure on the jacketed

$\qquad$ = $\rho_{mix}$ gh + atmospheric pressure - atmospheric pressure

$$= \rho_{mix} \times 9.81 \times D_T$$

$$= 1058.31 \times 9.81 \times 1.771$$

$$= 18386.56 N/m$$

$$= 18.34 KN/m$$

**THICKNESS OF THE VESSEL**

The thickness of the vessel that is, the distance between the jacket and the internal vessel is $2/3 W_b$

$$= 2/3 \times 0.1771$$

$$= 0.1181 m$$

**5.14    CALCULATION OF HEAT REQUIRED FOR HEATING THE VESSEL**

Using $m_s \lambda_s = UA\Delta T$

where $\lambda_s$ =    Enthalpy of vaporisation of steam at $100°C$

$$= 1037 Btu/lb$$

$$= 1037 \times 1055.06/0.4536 = 2412030.9 J/kg$$

$$= 2412.031 KJ/kg$$

$\mu_i$ (at $T_b = 70\ °C$); $T = T_s - T_b = (100 - 70) = 30°C$

$M_s$ = Amount of system required

$\Longrightarrow M_s = UA\Delta T/\lambda_s = 1.4675 \times 9.495 \times 30/242.0309$

The amount of steam equired to heat the bulk is $0.1733 kg/s$

**5.15        QUANTITY OF WATER NEEDED TO COOLING**

From heat balance around the condenser
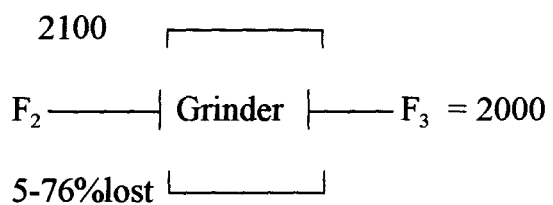
$$m\, C_w\, (\theta_2 - \theta_1) = UA\Delta T_{lm}$$

where

m = mass floor rate of cooling water required

$C_w$ = the specific heat capacity of water at the inlet temperature of 10 C = 4.1944 KJ/kg

$\theta_2$ = the inlet temp of cooling water = 10 C

## Material balance around the cylinder

```
   2100    ┌───────────┐
F₂ ─────── │  Grinder  │ ────── F₃ = 2000
           └───────────┘
5-76%lost
```

Material in = Materials out

$F_3 = F_4$

2100 = 2000

## Material balance arounds the extracts



$S' = 6000 kg$

Extractor

$f_3$
2000
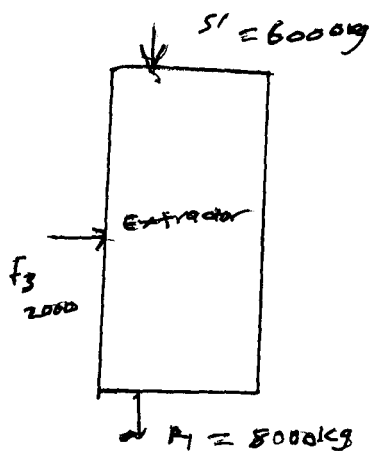
$R_1 = 8000 kg$

Material in = Material out

$F_3 + S = R_1$

2000 + 6000 = 8000kg

## Material balance around the filter press.



$R_1$ ──→ │ filter press │ ──→ $E_2 = 6700$

$R_2$ 1300 kg
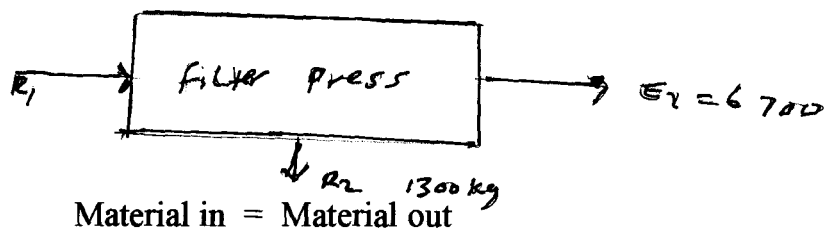
Material in = Material out

51

$$R_1 = R_2 + E_1$$

$$8000 = 1300 + 6700$$

$$8000 = 8000$$

Material balance around the distillation



$S' = 6000 kg$

$E_2 = 700 kg$

$\uparrow = 6700 kg$
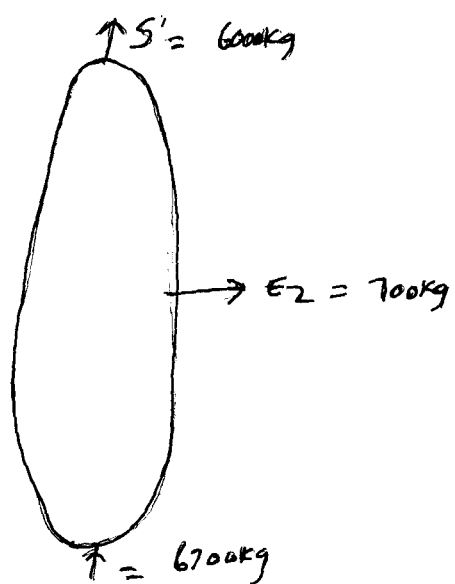
Material in = Material out

$$E_1 = E_2 + S'$$

$$6700 = 700 + 6000$$

$$6700 = 6700$$

$\theta_1$ = Outlet temperature of the cooling water = 30 C

A = the area of the extractor A = 9.495m

U = the overall heat transfer coefficient of the vessel at Tb = 70 C = 1.4675KN/m C

$T_{IM}$ =inlet temp of solvent - outlet temp/Ln (inlet temp/outlet)

= 70 - 30/Ln 70/30 = 40/Ln 7/3 = 47.21 C

The mass flow rate of the cooling water required is m = $UA\Delta T_{IM} /C_w (\theta_2 - \theta_1)$ = 1.4675 x

9.495 x 47.21/4.1944 x 20 = 9.45kg/s

## 5.16                              COST ANALYSIS

Costing of extractor which will contain 4m$^3$

Using

$Ce = CS^n$

where

$C_e$ = cost of the equipment

S = Size of unit

C = Cost constant

n = Index

Cost of agitator

$C_e = 69.574 \times 1000^{0.5} =$ £2200.123

Cost of pipe (carbon steel 15 - 350mm)

$C_c = 27d^{0.55}$ /m where £/m = pounds per meter

For solvent

$Ce = 27 \times (0.017304)^{0.55} =$ £2.9/m

per 2 meter length pipe = 2.9 x 2 = £5.8

For feed

$C_e = 27 \times (0.08824)^{0.55} =$ £7.104/m

For 2 meter length pipe the cost = 7.104 x 2 = £14.21

For mixture pipe

$C_e = 27 (0.0902)^{0.55} = £7.19/m$

cost for 2m length pipe = 7.19 x 2 = £1438

Cost of cooling water is 5 pound/ton

For 9.45kg/s of water for 4 hours (4 x 3600)

$\implies$ mass of water used = 9.45 x 4 x 3600 = 136080kg

$= 136.08ton$

Therefore, $C_e$ = 5 x 136.08p = £6.804

Cost of steam used = £6/ton

mass of steam used = 0.20885kg x 4 x 3600 = 3007.44kg

$= 3.007ton$

Ce = 6 x 3.007 = £18.042

**Note**    1£ = ₦150

## 5.17    SAFETY AND LOSS PREVENTION

Any organisation has a legal and moral obligation to safeguard the health and welfare of its employees and the general public. The security of the plant or unit as well as personnel working around it is a vital factor to be considered along side the plant design for smooth operation of the extractor and the entire plant in general, the safety precautions considered necessary includes

### 1)    OPERATIONAL CONDITIONS

Properties like temperature, pressure and concentrations must be strictly respected to ensure the life span of the reactor and the safety of the operators as well as to avoid lost production.

### 2)    ENVIRONMENTAL CONTROL

The control of the toxic flammable materials such as $CO_2$ and $H_2 S$ should be achieved to avoid pollution within the environment. The plant environment should be protected by sanitation as this provides clean, heating and safety working conditions.

### 3)    PROVISION OF SAFETY WEARS

The operating and laboratory personnel involved in obtaining samples should wear chemical type safety goggles or shield, solvent resistant gloves and approved respiratory protective equipment where ambient concentration exceeds allowable limits.

## 4) FIRE SAFETY

The function of this department would be

i.    To fight and quench all fire out break in the plant

ii.   To ensure that the personnel are well protected and follows all safety guidelines

iii.  Checking for leakages along all pipe networks

iv.   Provision of all necessary safety signboard, posters etc.

v.    Organisation of safety training for all staff.

## 4) INBUILT SAFETY GADGETS

Inbuilt safety gadgets are safety precautions installed in all equipment and around all building at strategic points in the plant especially areas demarcated as high risk areas. In addition, escape routes, fire extinguishers, loses etc. Should be made available, ever flour alarms and efficient process control mechanism should be installed on the equipment.

# CHAPTER SIX

## DISCUSSION OF RESULTS

Fig. 1. Show the effect of particule size on the amount of essential oil extracted from the figure it can be seen that for different times of extraction, the amount of oil extracted appear to rise through a maximum, for example at 1 hr of extraction, the maximum amount of extract was obtained for a particle size of $500\mu$ m. Similar particle size value was obtained for extraction time of 2 hrs, 3hrs, 4hrs, and 5hrs.

The figure further showed that the amount of extract obtained between 3 - 5 hrs pass through a narrow band.. Infact at the optimum size of 500 $\mu$m, the differences in the amount of extract are insignificant. Consequently extraction beyond 3hrs is unnecessary.

Fig.2. Shows the effect of time on the amount of extract at various particle size. The figure shows that the amount of extract increases with time of extraction between 1 - 3 hrs beyond which the amount of extract tails off. This clearly shows that the optimum time of extraction is 3 hrs. For example at a particle size of $850\mu$ m, the amount of extract was 0.4g which rose to 0.71g at 3 hrs. Beyond 3hrs, i.e 4 - 5 hrs extract values ranging between 0.73 and 0.733 were obtained of all the particle size investigated, the highest amount of extract at all times investigated were obtainted for $500\mu$ m size particles.

# CHAPTER SEVEN

## CONCLUSION AND RECOMMENDATION

**CONCLUSION**

Results of the experiments carried out shows the depending of the extract on particle size range.

From the results, the oil extract obtained from 1 - 5 hrs with the solvent are 0.414, 0 .61,0.711,0.73,0.733 respectively. Oil extracted from the small size range was substantially more than the one extracted from big size range. As such it is most suitable to finely grid the sample since it yield more oil.

Moreover increasing extraction time lead to more oil extract. Thus it could exclusively be said that an optimum or maximum percentage solvent extraction point was reached at 3hrs.

The perfume so formed from the extracted of Eucalyptus has good scent since it is combine with Frankense etc.

**RECOMMENDATION**

(1)     Chemical analysis could be carried out on the extracted oil to know the chemical composition.

(2)     Other part of the Eucalyptus citriodora, such as leave, shell, capsules, flower, back of the tree and roots can be used for its oil extraction.

(3)     Other organic solvent such as hexane, benzene could be used.

(4)     Since tertiary substance like thymol, menthol are of high demand by chemical and pharmaceutical industries, chemical analysis could be undertaken on the manufacture from Eucalyptus oil.

(5)     Because the oil is Eucalyptus citriodora is versative, plantation of the Eucalyptus in the country should be expanded for economic growth, its availability will encourage research findings.

# GLOSSARY OF TERMS

Essential oil: used in the cosmetic and perfume industries as synonymous with perfume oil, base or compound.

Fragrance: Unique in the realm of cosmetics for it is both an end result in its own right of other purposes.

Eucalyptus: Leaves used in extracting essential oil.

Perfume: Any mixture of pleasantly odorous sustance incorporated in a suitable vehicles.

Odorant: An odorous sustances, perfumery materials whether natural or syntheti.

Scent: A specialised meaning referring to odour left behind by an animal during a chase.

Vehicle: Compound that is used for blending and holding of the material of the oil or it is used ethyl alcohol.

Fixative: Component that help to lower the rate of evaporation of essential oil e.g myrrh or soft gum.

Extraction: A process which liquid component present in the solid is being extracted using a liquid.

Soxhlet extraction;Consist of conical flask, extractor,condenser,the extractor is mounted on a conical flask and the condense is mounted on the extractor.

Leaching: A solid - liquid extraction is the removal of a soluble fraction in the form of soluble from a insoluble permeable solid phase with which it is associated with or without diffusion.

# APPENDIX A

## CALCLULATION AND RESULTS

## PERCENTAGE OF OIL YIELD BY SoxHLET EXTRACTION EQUIPMENT USING ETHANOL/ACETONE AND PETROLEUM ETHER AS A SOLVENT

1ST EXPERIMENT

PARCTILE SIZE OF $850 \mu m$

Weight of the practice before extraction  $WP_b = 2g$

Weight of the filter paper $F_1$      $= 2.532g$

Weight of filter paper  $F_2$      $= 4.118g$

Weight of particle after extraction $WP_a = F_2, F_1 = 1.586$

Weight of the extract $WC = WP_b - WP_a$      $= 0.414g$

% yield oil $= \dfrac{WP_b - WP_a}{Wp_b} \times 100 \%$

$= \dfrac{W_c}{WP_b} \times \dfrac{100}{1} = \dfrac{0.414}{2} \times \dfrac{100}{1}$

$= 20.7\%$

========

## PARCTILE SIZE OF $710 \mu m$

Weight of the practice before extraction  $WP_a = 2g$

Weight of the filter paper $F_1$      $= 2.813g$

Weight of filter paper  $F_2$      $= 4.672g$

Weight of particle after extraction $WP_a = F_2, F_1 = 1859$

Weight of the extract $W_c = WP_b - WP_a$      $= 0.141g$

% yield oil $= \dfrac{WP_b - WP_a}{WP_b} \times 100 \%$

$$= \frac{W_e}{WP_b} \times \frac{100}{1} = \frac{0.141}{2} \times \frac{100}{1}$$

$$= 7.05\%$$

========

## PARCTILE SIZE OF 500$\mu$m

Weight of the practice before extraction  $WP_a = 2g$

Weight of the filter paper $F_1$  $= 2.678g$

Weight of filter paper  $F_2$  $= 4.020g$

Weight of particle after extraction $WP_a = F_2, F_1 = 1.3 + 2$

Weight of the extract $W_e = WP_b - WP_a$  $= 0.658g$

% yield oil $= \dfrac{WP_b - WP_a}{Wp_b} \times 100\,\%$

$$= \frac{W_e}{WP_b} \times \frac{100}{1} = \frac{0.658}{2} \times \frac{100}{1}$$

$$= 32.9\%$$

========

## PARCTILE SIZE OF 250$\mu$m

Weight of the practice before extraction  $WP_a = 2g$

Weight of the filter paper $F_1$  $= 2.721g$

Weight of filter paper  $F_2$  $= 4.134g$

Weight of particle after extraction $WP_a = F_2, F_1 = 1.413$

Weight of the extract $W_e = WP_b - WP_a$  $= 0.587g$

% yield oil $= \dfrac{WP_b - WP_a}{Wp_b} \times 100\,\%$

$$= \frac{W_c}{WP_b} \times \frac{100}{1} = \frac{0.587}{2} \times \frac{100}{1}$$

$$= 29.35\%$$

## 2ND EXPERIMENT

### PARCTILE SIZE OF 850$\mu$m

Weight of the practice before extraction $WP_b = 2g$

Weight of the filter paper $F_1$    = 2.478g

Weight of filter paper $F_2$      = 3.767g

Weight of particle after extraction $WP_a = F_2, F_1 = 1.289$

Weight of the extract $WC = WP_b - WP_a$     = 0.711g

% yield oil $= \dfrac{WP_b - WP_a}{Wp_b} \times 100\ \%$

$$= \frac{W_c}{WP_b} \times \frac{100}{1} = \frac{0.711}{2} \times \frac{100}{1}$$

$$= 35.55\%$$

### PARCTILE SIZE OF 710$\mu$m

Weight of the practice before extraction $WP_b = 2g$

Weight of the filter paper $F_1$    = 2.729g

Weight of filter paper $F_2$      = 3.944g

Weight of particle after extraction $WP_a = F_2, F_1 = 1.215$

Weight of the extract $WC = WP_b - WP_a$     = 0.785g

% yield oil $= \dfrac{WP_b - WP_a}{Wp_b} \times 100\ \%$

$$= \frac{W_c}{WP_b} \times \frac{100}{1} = \frac{0.785}{2} \times \frac{100}{1}$$

$$= 39.25\%$$

## PARCTILE SIZE OF 500$\mu$m

Weight of the practice before extraction $WP_b = 2g$

Weight of the filter paper $F_1$ $\quad = 2.628g$

Weight of filter paper $F_2$ $\quad = 3.784g$

Weight of particle after extraction $WP_a = F_2, F_1 = 1.156$

Weight of the extract $WC = WP_b - WP_a$ $\quad = 0.844g$

% yield oil $= \underline{WP_b - WP_a}$ x 100 %

$\qquad Wp_b$

$\qquad = \underline{W_c}$ x $\underline{100}$ $= \underline{0.844}$ x $\underline{100}$

$\qquad WP_b \quad 1 \qquad 2 \qquad 1$

$$= 42.2\%$$

## PARCTILE SIZE OF 250$\mu$m

Weight of the practice before extraction $WP_b = 2g$

Weight of the filter paper $F_1$ $\quad = 2.646g$

Weight of filter paper $F_2$ $\quad = 3.909g$

Weight of particle after extraction $WP_a = F_2, F_1 = 1.263$

Weight of the extract $WC = WP_b - WP_a$ $\quad = 0.737g$

% yield oil $= \underline{WP_b - WP_a}$ x 100 %

$\qquad Wp_b$

$\qquad = \underline{W_c}$ x $\underline{100}$ $= \underline{0.737}$ x $\underline{100}$

$\qquad WP_b \quad 1 \qquad 2 \qquad 1$

$$= 36.85\%$$

**3RD EXPERIMENT**

**PARCTILE SIZE OF 850$\mu$m**

Weight of the practice before extraction $WP_b$ = 2g

Weight of the filter paper $F_1$ = 2.499g

Weight of filter paper $F_2$ = 3.766g

Weight of particle after extraction $WP_a = F_2, F_1$ = 1.267'

Weight of the extract WC = $WP_b$ - $WP_a$ = 0.733g

% yield oil = $\dfrac{WP_b - WP_a}{Wp_b} \times 100$ %

$$= \dfrac{W_c}{WP_b} \times \dfrac{100}{1} = \dfrac{0.733}{2} \times \dfrac{100}{1}$$

$$= 36.65\%$$

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**PARCTILE SIZE OF 710$\mu$m**

Weight of the practice before extraction $WP_b$ = 2g

Weight of the filter paper $F_1$ = 2.727g

Weight of filter paper $F_2$ = 3.910g

Weight of particle after extraction $WP_a = F_2, F_1$ = 1.183

Weight of the extract WC = $WP_b$ - $WP_a$ = 0.817g

% yield oil = $\dfrac{WP_b - WP_a}{Wp_b} \times 100$ %

$$= \dfrac{W_c}{WP_b} \times \dfrac{100}{1} = \dfrac{0.817}{2} \times \dfrac{100}{1}$$

$$= 40.85\%$$

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**PARCTILE SIZE OF 500$\mu$m**

Weight of the practice before extraction $WP_b = 2g$

Weight of the filter paper $F_1$ = 2.626g

Weight of filter paper $F_2$ = 3.780g

Weight of particle after extraction $WP_a = F_2, F_1 = 1.154$

Weight of the extract $WC = WP_b - WP_a$ = 0.846g

% yield oil = $\dfrac{WP_b - WP_a}{Wp_b}$ x 100 %

= $\dfrac{W_c}{WP_b}$ x $\dfrac{100}{1}$ = $\dfrac{0.846}{2}$ x $\dfrac{100}{1}$

= 42.3%

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**PARCTILE SIZE OF 250$\mu$m**

Weight of the practice before extraction $WP_b = 2g$

Weight of the filter paper $F_1$ = 2.737g

Weight of filter paper $F_2$ = 3.854g

Weight of particle after extraction $WP_a = F_2, F_1 = 1.117$

Weight of the extract $WC = WP_b - WP_a$ = 0.800g

% yield oil = $\dfrac{WP_b - WP_a}{Wp_b}$ x 100 %

= $\dfrac{W_c}{WP_b}$ x $\dfrac{100}{1}$ = $\dfrac{0.800}{2}$ x $\dfrac{100}{1}$

= 40%

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