COMPARATIVE STUDY OF THE PRODUCTION OF OIL FROM AVOCADO APPLE (*Persea americana*) USING EXTRACTION AND STEAM DISTILLATION

BY

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M.ENG/SEET/2005/1136

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IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE
AWARD OF DEGREE OF M.ENG IN CHEMICAL ENGINEERING

OCTOBER, 2006

DECLARATION

I, ADAMA KENNETH KENNEDY, hereby declare that this thesis was written solely by me under the supervision of Dr. M.O. Edoga of Department of Chemical Engineering, Federal University of Technology, Minna and has never been presented elsewhere for any degree.

2/07/08.

Signature & Date

CERTIFICATION

This thesis titled: COMPARATIVE STUDY OF THE PRODUCTION OF OIL FROM AVOCADO APPLE (*persea americana*) USING EXTRACTION AND STEAM DISTILLATION by KENNNETH KENNEDY ADAMA (M.ENG/SEET/2005/1136) meets the regulations governing the award of the degree of M.Eng. Chemical Engineering of the Federal University of Technology, Minna and is approved for its contribution to scientific knowledge and literacy presentation.

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DEDICATION

This work is dedicated to God Almighty for His mercy and love upon me and all those who contributed immensely to my academic achievement.

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My sincere appreciation goes to those who have in one way or the other helped to make this research thesis a success.

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ABSTRACT

This work compared the production of oil from the pericarps (peels) of avocado apple using steam distillation and extraction (direct and indirect leaching) methods with respect to the yields and economic analysis. An improvised steam distillation set-up was used at three different steam heating rates. It entailed the comminution of the sample materials suspended on a grid (perforated metal plate) 90 mm above the water level and the application of steam through the material. Variations in time and volume of water used to generate the steam for the process are as follows: 30, 60, 90,120,150 and 180 mins. and 1000 ml, 1250 ml and 1500 ml respectively. The improvised pilot plant was designed with appropriate provision for scaling up. In contrast, leaching method using two different solvents, n-hexane and anhydrous ethanol, were used. The extraction was carried out at different particle sizes, times and boiling points of the solvents. These were as follows: 0.75 mm, 1.00 mm, 1.59 mm and 2.00 mm; 30, 60, 90,120,150 and 180 mins.; 60^0C and 78^0C for n-hexane and anhydrous ethanol respectively. The active principles (oils) from both methods were further processed by treating them with activated clay and heating to a constant temperature of 100^0C for 30 min. to produce high quality refined oil. Finally, the crude and refined oils were characterized in order to determine their suitability for industrial applications. The results from steam distillation revealed that the heating rate of 2.57 ml/min (moderate) gave the maximum oil yield of 59.8% whereas 4.72 ml/min (low) and 4.94 ml/min (high) heating rates gave oil yields of 38.7% and 39.6% respectively. On the other hand, the results from leaching method at the optimum extraction time (120 min.) and particle size (0.75 mm), gave optimum oil yields of 21.2% and 23.9% for indirect and direct leaching respectively using n-hexane, 35.9% and 42.0% for indirect and direct leaching using anhydrous ethanol respectively. The physicochemical analysis of the refined oil showed that the oils had a saponification value of 196.4, acid value of 5.653, iodine value of 1.27, specific gravity of 0.90261, viscosity of 181.180cp and refractive index of 1,600. The chemical engineering design of the steam distillation pilot plant gave the following specification: Diameter, 0.231 m; Height, 0.464 m and volume, 0.01944 m^3 representing the geometry of the reactor with the overall rate controlled by the physical rate processes. The properties of the present oils are in conformity with the international standards (ISO/TR 21092, ISO 212, ISO/TR 210, and AOCS) and as such are potential substitutes for the most oils used for cosmetics and pharmaceutical drugs production. The comparative study of steam distillation and leaching revealed that, based on yields, steam distillation gave a yield of 59.8% compared with 35.2% for indirect and 42.0% for direct leaching. Also, based on economic analysis of the three production method tested, steam distillation method gave a rate of return (ROI) on investment of 78.88% and a payback period (PBP) of 1.3 years compared with 39.8% and 2.5 years for indirect leaching and 61.04% and 1.6 years for direct leaching respectively.

TABLE OF CONTENT

Content Pages

CHAPTER ONE

CHAPTER TWO

APTER THREE

CHAPTER FOUR

CHAPTER FIVE

CHAPTER SIX

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS/NOTATIONS

AVOA	Avocado Apple
AVOP	Avocado Peel/Pericarp
ISO	International Standard Organization
AOCS	American Oil Chemists' Society
ROR	Rate of Return
PBP	Pay Back Period
Ml	Millilitre
Mins	Minutes

CHAPTER ONE

1.0 INTRODUCTION

Avocado apple (*Persea americana*) variously known as the avocado, avocado pear, alligator pear, ahuacatl or agvacate, is a highly nutritional fruit rich in proteins, fats and oils and low in sugar. The total food value is high, providing nearly twice the energy of an equivalent weight of meat as well as an abundance of several vitamins such as A, B, C, D and E. Generally, the fruit composition by weight is about 65% mesocarp, 20% endocarp (seed) and 15% pericarp (peels) (Kirk Othmer, 1983; New Encyclopedia Britannica Vols. 1, 13 & 19). In addition to the oil, the avocado contains a small amount of about 1.5% by weight of oil of unsaponifiable matter, as a distinct fraction, which by itself is a very valuable component useful in many creams and medicated ointments (Werman, M.J. et. al. 1990, Kirk Othmer, 1983). It is readily available in the south western, south eastern and north central states of Nigeria. The fruit contains appreciable quantity of oil in the mesocarp as well as the pericarp. The lack of proper technical know-how and standard processing equipment had frustrated the local production of oils from the mesocarp and pericarp of the apples. However, with the abundant production of avocado apple in Nigeria, 15% of avocado apple (pericarps/peels) is discarded as waste which ought to have been a potential source for the production of oil for industrial applications due to its antiviral, antibiotic and insecticidal properties (Thomas, W.W. et. al., 2000; Daguet, D. 2000; Werman, M. et al.; 1990; David, R.M. et. al. 2000).

The method of production of oil from the avocado apple's pericarp is as varied as the species, though of most importance is the use of steam distillation and extraction with appropriate solvents. Various technologies have been developed for the production of oil from the avocado apples. This includes rendering also known as manual extraction which entailed the use of clamp to squeeze out the oil from the sample material (Julius and Francis, 2003). Rendering technology is cumbersome and does not enhance or improve the efficiency of the oil extraction in terms of yield and quality; the use of pressing machine (cold-press and not press equipment) which entails maceration of the sample by a high-speed grinder, followed by mixing of the pulp in malaxers (Werman and Neeman, 1987; Wicken,G.S. et. al. 1989). This gives only minute quantities of oils; finest product (in the case of cold pressing) but large quantities of undesirable components with stronger odour (hot pressing). Extraction with volatile solvent (leaching) which is in particular applied when the volume of the oil is considerably greater as oil than as part of the meal, thus requiring a complete extraction with volatile solvents (Romeo, T.T, 2000; Henley and Seader, 1999; Prabhudesai, R.K 1979; King, C.J. 1980). Extraction with volatile solvents gives higher yields of oils though a major drawback is the difficulty encountered in completely removing the solvent from the oil extract.

Steam distillation involves the use of steam to percolate and vaporize out the oil from the sample material with the subsequent condensation of steam and oil prior to their separation using a settling tank, separation funnel or any similar vessel. Three different methods of steam distillation are practiced (Kirk Othmer, 1983; New Encyclopedia Britannica, Vols. 1 and 13). The advantage of steam distillation, in addition to effective pressure reduction is that when condensed, the distillate will often break into two liquid phase; the water not diluting the organic distillate product significantly.

1.1 Aims and Objective of the Research

The main aim of the research was to find possible uses of the avocado apple's pericarps/peels that hitherto had been discarded as wastes. Specifically, the work was aimed at producing oil from the pericarps of avocado apple using steam distillation from an improvised pilot plant and extraction (direct and indirect leaching) methods; thus identifying which of the methods gave the highest yields and economical profitability. Furthermore, the characterization of the oils (active principle) from the avocado apple's pericarps was be undertaken in addition to developing a cost effective and environmentally friendly process for the production of avocado pericarp oil for cosmetic and pharmaceutical industries..

1.2 Scope of Work

The work focused on the production methods of steam distillation at three different heating rates using an improvised pilot plant and leaching (direct and indirect extraction) to produce oil from the pericarps of avocado apple. The solvents used for the leaching process were n-hexane and anhydrous ethanol. The scope also included the engineering design and economic analysis of both the steam distillation and direct and indirect leaching for the production of oils from avocado pericarps.

1.3 Significance of Research

The study evaluated the steam distillation and leaching(direct and indirect) methods employed for the production of oil from the avocado apple's pericarps vis-à-vis the yields of oils from either methods. Also, the characterization of the oils was carried out so as to know its physiochemical properties cum suitability for industrial applications while justifying the process design on the following criteria: low cost of production; efficiency and economic desirability; less expensive equipment; reduction to the bearest minimum problems of agricultural waste disposal and pollution; new and improved production processes and industrial employment generation and wealth creation.

CHAPTER TWO

2.0 LITERATURE REVIEW

Avocado apple (*Persea americana*) variously called avocado, avocado pear, alligator pear, ahuacatl or agvacate is a tropical fruit with hard, dark green pericarp, light green mesocarp and a large seed (endocarp) inside. It is a highly nutritional fruit rich in proteins, fats and oils and low in sugar (Rice, et al., 1986). The total food value as well as its oil content is high and it provides nearly twice the energy of an equivalent weight of meat as well as an abundance of several vitamins. A typical composition of the avocado apple show a 65% mesocarp (flesh), 20% endocarp (seed) and 15% pericarp/peels. Other well-known members of the laurales of which the Persea americana belong are cinnamon, sassatras, gratissima and greenheart (Samson, 1986). Oil expressed from the flesh is rich in vitamin A,B,C and E (Koch, D.F; 1983, Betser, et. al. 1999). It has a digestibility coefficient of 93.8% but has remained too costly to be utilized extensively as salad oil. The amino acid content has been reported as: podmitic, 7.0; stearic, 1.0; oleic, 79.0; linoleic, 13.0. Gachanja and Ilg, 1990 identified two methods of extracting oil from the avocado apples. These are the use of pressing machine and manual extraction method. The use of the pressing machine was known to improve the efficiency of the oil extraction. Lawrence, et. al. 2005 obtained oil from the flesh of the avocado apple and this finds commercial importance in several countries notably Mexico, South Africa and Israel. This oil however has been initially extracted from poor quality and waste avocado using solvents then refined, bleached and deodorized. It has mainly been used as a first-class cosmetic raw material and has not been viewed as edible oil

2.1 Historical Development of Avocado Apple

The avocado apple tree was introduced into West Africa years ago and is now thoroughly naturalized in many parts of Nigeria. In Nigeria, the ecology of the tree crop is closely related to the situation of the rainforest belt along the coast and savannah zones. It is still known as 'pear' in some vernaculars. However, the concentration is in the southern part of the country (Opeke, K. L; 1979).

2.1.1 Varieties

There are great many varieties of avocado apple. Martin et al; 1987, identified three different species which are Mexican, Guatamalan and West Indian prevalent in the subtropical, semitropical and tropical climatic areas respectively. All the three species are largely cultivated and produced to varying degrees in the tropical rainforest and savannah belts of Nigeria (Opeke, K. L., 1979; Samson, A. R., 1986). These races or species can be distinguished from one another using several attributes as given in Table 2.1 (Samson, 1986).

Table 2.1: Properties of Some Avocado Varieties

Main attribute	Specific attribute	Properties by Varieties		
		Mexican	**Guatemala**	**West Indian**
Oil	Scent	Nice	None	None
Leaf	Size	Small	Various	Various
	Skin	Thin	Warty	Leathery
Seed	Size	Big	Small	Big
	Cavity	Loose	Tight	Loose
Tolerance	Cold	Yes	Medium	No
	Salt	No	No	No
Fruits	Oil content	High	Medium	Low
	Maturity (Months)	6	9	6

2.1.2 Uses

Avocado apples are an important addition to diet as they are of high food value. They are rich in sugar, in mineral matter and in easily digested oil. They contain a high percentage of vitamins A, B, C, D and E. The apples are used as fruits or vegetable, flavored with salt, pepper, vinegar or lime. They are excellent eaten with bread or other starchy food and make a good soup. The average protein content is 2.5 percent which is higher than many fresh fruits. In the West Indies, a type of ink is made from the nut. The seed yields a milky fluid with the odor and taste of almond. Because of it tannin content, it turns red on exposure, providing an indelible red-brown or blackish ink which was used to write many documents in the days of the Spanish conquest. Also, the fruits pericarp (peels) are reported to have antiviral, antibiotic and insecticidal properties (Thomson, W. W et. al; 2000) and employed as a vermifuge and remedy for dysentery and in the production of cosmetics.

2.2 Extraction Processes

Extraction is amongst the oldest methods of separation being practiced by human beings. It is a process that separates components or constituents of a mixture based upon physical properties by utilization of the solubility of one or more components in a second phase.

Basically, the separation process involves two phases. The solvent is the material added to form a phase different from that in which material is to be separated was originally present. Separation is achieved when the compound to be separated dissolves in the solvent while the rest of the components remain where they were originally. The two phases may be solid and liquid, immiscible liquid phases or solid and gas. The added phase is liquid, while that to be separated may either be a solid or a liquid. Generally, solid-liquid extraction is also called leaching (solvent extraction).

2.2.1 Techniques in the extraction of oil

Several methods have been developed for the extraction of oil from fruits and other oil-bearing plant materials. Generally, the techniques of extraction are mainly mechanical and chemical.

2.2.2 Mechanical extraction process

The mechanical extraction process is the oldest method of oil extraction which involves the application of pressures to batches of oil-bearing materials contained in bags, cloths, cages, etc. The pressing of the sample material to obtain oil is divided into two parts; cold pressing and hot pressing (Wicken, G. S, et. al; 1989).

In cold pressing, the sample material is initially preheated and reduced to certain particle sizes. Then pressing is done by using a hydraulic machine to squeeze out the oil. Only minute quantities of oils are obtained by simply pressing the seed. Cold pressing gives the finest product and is used for producing the highest grade of oil e.g. peanut oil, olive oil, cotton seed oils.

In hot pressing, preheating of the meal is done to bring about the coagulation of the protein matter which is largely responsible for the extremely fine state of division of the oil within the cells. Hot pressing gives a higher yield, but large quantities of undesirable components with stronger odour.

2.2.3 Chemical extraction processes

The chemical extraction processes includes steam distillation and leaching also called solvent extraction, or solid-liquid extraction methods.

i. Leaching

Leaching, sometimes also called lixiviation, decoction, solvent extraction or solid-liquid extraction is in particular applied when the value of the oil is considerably greater as oil than as part of the meal, thus requiring a more complex extraction with volatile solvents. It involves separating the components or constituents of the mixture based on the differences in physical properties. It depends on the selective dissolution of one or more liquid constituents of the mixture into a suitable immiscible liquid solvent. The basic principle behind the extraction involves the contacting of a solution with another solvent that is immiscible with the original. The solvent is also soluble with a specific solute contained in the solution (Hanson, et al; 1983, Felder, et al; 1978). Two phases are formed after the addition of the solvent due to the differences in densities. The solvent is chosen so that the solute in the solution has more affinity towards the solvent. Hence, mass transfer of the solute from the solution to the solvent occurs and thus, separation is achieved when the components to be separated dissolve in the solvent while the rest of the components remain in the solution. This is based on the concept of an "equilibrium" or "ideal" stage which is the stage from which the resultant solution is of the same composition as the solution adhering to the solids leaving the stage (Henley and Seader; 1981, Hanson, et al; 1983).

A well known method of extracting oil is the use of the soxhlet extractor. This is an indirect method of extracting oil from oil-bearing tissues and cells. It involves flooding the solute containing the material with solvent and then allowing it to soak for some period of time. During this period, the oil being the solute leaves its initial location and diffuses preferentially into the solvent. Maximum extraction is obtained by continuously contacting the feed with either fresh solvent or recycled solvent. This method however, gives a comparatively low yield of oils and requires large quantities of solvent for small oil extracted (Prabhudesai, R. K; 1979).

Direct leaching is usually employed for oil-bearing fruits of low oil content containing less than 20% oil. Prepress solvent extraction is used for high oil content (more than 20% oil) oil-bearing fruits. However, in all types of oil-bearing fruits, through solvent extraction, about 90% of oil is extracted and only 10% remain in the meal (Schweitzer, P. A. 1997; Hanson, et al; 1983, King, C. J; 1980).

ii. Phase equilibrium

From the standpoint of the phase rule given thus

$F = C - P + 2$ 2.1

Where F = number of degrees of freedom i.e. number of independent properties which have to be specified to determine all the intensive properties of each phase of the system of interest.

C = number of components in the system and

P = number of phases that can exist in the system..

There is no difference between a liquid-liquid and a solid-liquid (leaching) system. For this reason, phase equilibrium data for a three-component mixture of solute, solid and solvent at constant temperature and pressure can be represented on an equilateral or right-triangular diagrams as was done for liquid-liquid systems using weight fractions or weight ratios.

In spite of this, there are major differences between liquid-liquid and solid-liquid contacting because in the latter case, diffusion in the solid is so slow that true equilibrium is rarely achieved in practice. Also, drainage is equally slow that complete phase separations are seldom realized in mixer-settlers which is the most common type of leaching equipment employed. Therefore, rather than use thermodynamic equilibrium data to calculate stage requirements, a pragmatic approach is to use, for such calculations, pilot-plant or bench-scale data taken in prototype equipment where residence times, particle size, drainage conditions and level of agitation are such that the data can be extrapolated to plant size leaching equipment. Thus, several scaling up methods can be used as conditions change with scale and these include chemical similitude or dimensional similitude, mathematical modeling, scale-up by combined method, scale-up by conventional method and statistical method. But of most importance is the method of chemical similitude and mathematical modeling. However, where either method is inadequate, scale-up by combined method is the best. This method provides a technique of using dimensional similitude in combination with mathematical modeling in the design of a pilot plant and in evaluating the results to provide the basis for scale-up to a commercial scale plant. Stage efficiencies are therefore inherently included in the equilibrium diagrams. In addition, instead of having equilibrium phase, it is possible and practicable to have an overflow solution in equilibrium with solution adhering to an underflow of solids and solution.

If the carrier solid is completely inert and is not dissolved or entrained in the solvent, the solute is infinitely soluble in the solvent and if sufficient contact time for the solvent to penetrate the solute completely is permitted, then ideal leaching conditions exist and phase equilibrium diagrams such as the right triangular plots shown below can be utilized.

A typical right-triangular plot is shown in Figure 2.1 where the mass fraction of solvent in overflow (y_S) is plotted against the mass fraction of solute in underflow (x_A). In the figure, EF represents the locus of overflow solutions for the case where the overflow streams containing some inert solids, either by entrainment or by partial solubility in the overflow solutions. Lines GF, GL, and GM represent loci of stream composition for three different conditions, namely, constant underflow, variable underflow, and constant solvent-to-inert in underflow. The constant underflow line GM is parallel to EF, the hypotenuse of the triangle, whereas GF passes through the right-hand vertex representing 100% solute.

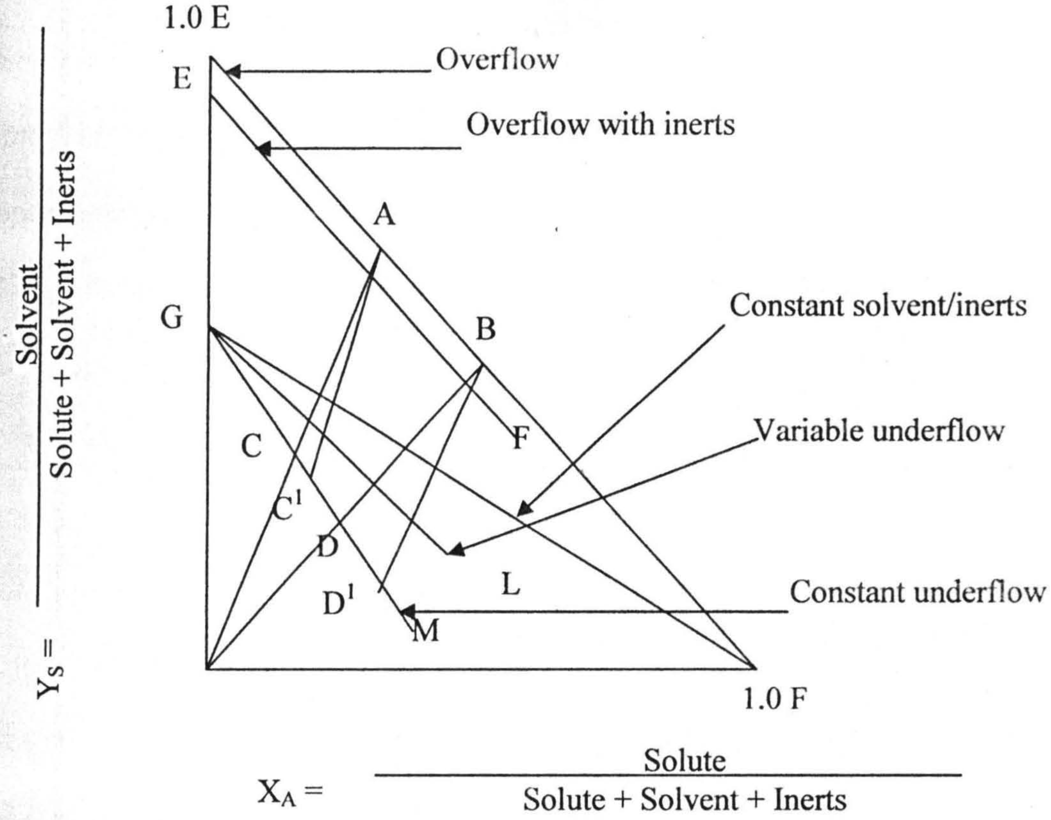

Figure 2.1: Typical Equilibrium Diagram for Leaching Operation

If all the solute is in solution and if the solution adhering to the solid has the same composition as that of the overflow stream, then lines such as AC and BD represent the ideal equilibrium tie line which passes through the origin O (representing 100% inerts).

For non-equilibrium conditions or for equilibrium position such as AC^1 and BD^1. Point C^1 is to the right of C, if the solute concentration in the overflow solution is less than that in the underflow solution adhering to the solids. Unequal concentration in the two solutions indicates insufficient contact time and/or preferential adsorption of one of the components on the inert solids. Tie-lines such as AC^1 may be considered "practical tie lines" if data on underflow and overflow composition are obtained experimentally under conditions simulating actual operation, particularly with respect to contact time, agitation and particle size of solids.

In non-ideal leaching, the tie lines slant to the right, indicating that the solute is more highly concentrated in the underflow, either because of equilibrium solubility or because of incomplete leaching (the latter is more likely when the solute and solvent are completely miscible).

Construction of material balance lines on solid-liquid diagrams depends critically on the coordinates used to represent the experimental data.

2.2.4 Factors influencing the rate of extraction (leaching)

Two steps which are always involved in solid-liquid extraction (Leaching) are (1) contact of liquid solvent with the solid to effect transfer of solute from the solid to the solvent and (2) separation of the resulting solution from the residual solid. Two other auxiliary operations that are involved include preparation of the solid for extraction and recovery of solute from the solvent, usually by evaporation or distillation. To meet these objectives, it is necessary to select the most practical and economic contacting and separation equipment in a batch or continuous operation. Equipment choice and method of operation for a particular extraction depend on various factors such as physical characteristics of the solid to be leached, and quantity and size of the solid relative to the amount of solute to be recovered.

i. Physical characteristics of solids

Knowledge of the physical characteristics of the carrier solid is very important in order to determine if it needs prior treatment to make the solute more accessible to the solvent. Prior treatment may involve crushing, grinding, cutting into pieces, or re-forming into special shapes such as flakes.

Solute particles may exist in the inert solid in a variety of ways. It may exist on the surface of the solid, may be surrounded by a matrix of inert material, may be chemically combined, or may exist inside cells as in the case of many vegetable and animal bodies.

Solute adhering to the solid surface is readily removable by the solvent. When the solute exists in pores surrounded by a matrix of inert material, the solvent has to diffuse to the interior of the solid to capture solute and then diffuse out before a separation can result. In such cases, subdivision of the solid by crushing, grinding, or cutting increases the surface exposed to the solvent. However, reduction of solid to finer particle size has its limitations. In some cases, the amount of solute to be recovered is small in comparison to the amount of solid to be treated, in which case grinding becomes uneconomical. Too-fine division may result in packing of solid during extraction, preventing free flow of solvent though the solid bed. In such a case, the extraction is much more difficult, especially when finely divided solids are treated in an unagitated state. Dispersion of particles in liquid solvent by agitation permits thorough contacting of the solid with the solvent. Agitation, while giving good extraction, may cause suspension of fine particles in out-flowing solution, which may subsequently require a difficult filtration or clarification step.

ii. Solvent selection

Properties of a solvent such as its boiling point, density and viscosity affect its suitability and selectivity to effect more complex and economical extraction of the desired solute. The extent and rate of solution of the desired solute and impurities in the solid by a solvent influence considerably the size of an extractor, overall operating costs, and type and cost of separation as well as solvent and soluble recovery equipment and product quality. The solvent should be selective, relatively cheap, nontoxic, and readily available. Very often a compromise in the selection of the solvent is required.

iii. Temperature of extraction (leaching)

Higher temperatures result in higher solubility of the solute in solvent and are therefore desirable to realize higher solute concentrations in the extract. Increased rates of leaching are obtained because of lower viscosity of the liquid and higher diffusivity of the solute and the solvent. Higher temperatures in the case of some natural materials, however, result in excessive extraction of undesirable material. High temperature operation may not be permissible because of unacceptable solvent losses and safety consideration. Again, a compromise is necessary in the selection of leaching temperature.

2.2.5 Methods of contacting phases

The leaching process is generally of two output streams: One being simply a solution of solute and solvent and the other containing the inert solid, mixed with some solute and solvent.

Two types of solid-liquid contacting methods may be distinguished: fixed-bed contacting in which the solid particles are stationary, and dispersed contact which involves motion of particles relative to each other and also relative to the liquid. Solvent can be contacted with a fixed bed of solids in three different ways.

The three different ways are: spray percolation, full immersion, and intermittent drainage.

In spray percolation method, the solvent is sprayed on the solid and allowed to drain through it continuously. In full immersion, the solid remains submerged in liquid and may be treated with solvent being supplied batch wise or continuously. The third method involves intermittent drainage of the solvent from the solids. A dispersed contact is usually effected by suitable agitation.

The simplest fixed-bed contacting equipment consists of an open tank with a perforated bottom in which the solvent is allowed to percolate by gravity through the bed of undissolved solids. An example of dispersed contact equipment is a leaching tank in which the solids are dispersed in the solvent by agitation and then allowed to separate from extract solution by settling. Separation of extract solution from the solids may be effected in the same tank to a separate settling unit, or by the use of various types of filtration equipment. When volatile solvents are used or when percolation under gravity is too slow, a closed percolation tank with a solvent circulation pump may be used.

2.2.6 Flow configuration of extraction process

The principal types of operating or extraction methods used in leaching systems are single-stage batch processing, multi-stage cross-flow extraction, multi-stage counter current extraction and continuous countercurrent extraction.

i. Single-stage batch processing

In single-stage batch processing, the solid is contacted with solute-free solvent until equilibrium is reached. The solvent may be pumped through the beds of solids and recirculated, or the solids may be soaked in the solvent with or without agitation. After equilibrium, the solvent phase is drained out of the solids. Examples are brewing of coffee or tea and water decaffeination of raw coffee beans.

ii. Multistage cross-flow extraction

In this process, the solid is contacted repeatedly, each time with solute-free solvent as in the soxhlet extraction of fats and oil in food/oil analysis. The procedure requires a lot of solvent or in the case of a soxhlet, a lot of energy in vaporizing and condensing the solvent for recycling; hence, it is not used as an industrial separation process.

iii. Multistage countercurrent extraction

This process utilizes a battery of extractors; solute-free solvent enters the system at the opposite end from the point of entry of the unextracted solids. The solute-free solvent contacts the solids in the last extraction stage; resulting in the least concentration of solute in the solvent phase at equilibrium. Thus, the solute carried over by the solids after separation from the solvent phase at this stage is minimal. Solute-rich solvent, called the extract, emerges from the system at the first extraction stage after contacting the solids which have just entered the system. A stage-to-stage flow of the solids. The same solvent is used from stage to stage; therefore, the solute concentration in the solvent phase increases as the solvent moves from one stage to the next, while the solute concentration in the solids decreases as the solids move in the opposite direction.

iv. Continuous countercurrent extraction

In this system, the physical appearance of an extraction stage is not well defined. In its most simple form it may consist of an inclined screw conveyor. The conveyor is initially filled with the solvent to the overflow level at the lower end, and solids are introduced at the lower end. The screw moves the solids up through the solvent. Fresh solvent introduced at the highest end moves countercurrent to the flow of solids, picking up solute from the solids as the solvent moves down. Eventually, the solute-rich solvent collects at the lowermost end of the conveyor and is withdrawn through the overflow. In this type of extraction system, the term 'height of transfer unit' (HTU) is used to represent the length of the conveyor, where the solute transfer from the solids to the solvent is equivalent to one equilibrium stage in a multistage system. The height of transfer unit (HTU) describes the quantity obtained by dividing values of individual coefficients of mass transfer by the flow rates of the phase to which it applies. The quantity obtained by this division is called height of transfer unit since it expresses in terms of a single length dimensions the height of apparatus required to accomplish a separation of standard difficulty.

2.3 Solid-Liquid Extraction (Leaching) Calculations

The calculations for a solid-liquid extraction system may be based on the use of material and energy balances and the concept of an ideal stage. An ideal stage is reached in leaching when the liquid in the overflow has the same concentration as the liquid in the underflow.

The lesser importance of the energy changes in actual extraction processes usually leads to omission of the energy-balance equations. As a result, the calculations are based on the material balances and the ideal-stage concept.

In leaching, true equilibrium between solution and solute is rarely achieved because there is not usually sufficient time of contact for complete dissolution of the solute. Also, it is impractical to make separation of solid and solution perfect, and therefore solids leaving the stage will always retain some liquid and its associated dissolved solute. In case the solute is adsorbed by the solids, imperfect settling or drainage will result in lowering of stage efficiency even though equilibrium between the solution and solid phases is established.

Both algebraic and graphic methods of solution may be used since they are equivalent procedures for obtaining solutions to the materials balances and ideal – stage relationships.

The graphic solution (J.C. Elgin, 1936) possessed advantages in permitting a generalized treatment of the more complex cases and in permitting a better visualization of what is occurring in the process, although it may be inconvenient to use if a large number of stages is involved.

2.3.1 Assumptions made when calculating the number of stages in leaching process.

i. All solutes goes into solution

ii. No insoluble solutes present in extract.

iii. Ratio of solvent in extract to solvent in raffinate is constant

2.3.2 Procedure

A given leaching system is assumed to consist of the following three components:

i. Inert, insoluble solids

ii. A single solute which may be a single solid or liquid or a mixture of soluble components.

iii. A solvent which dissolves the solute but has little or no effect on the inert solid.

The solubility of the inerts and adsorption of solute by the inert solid can be accounted for in the calculations if data are available on the solubility of inerts in the solvent and on adsorption of solute by inerts as a function of solution concentration. The computation of the number of ideal stages required is then based on material balances, knowledge of solution quantity retained by the inert solids as a function of solute concentration in solution, and the definition of an ideal stage. Either stage to stage algebraic methods, graphical methods, or in some special cases, analytical methods can be applied. As a result of its relative importance, these methods can be applied to the design of a continuous countercurrent multistage system, after considering the single stage system. However, design parameters such as diffusion coefficients and mass transfer coefficients must be ascertain to determine scaling up process.

2.3.3 Mass transfer in leaching operations

As in other mass-transfer operations, leaching calculation are based on the concept of "equilibrium" or "ideal" stage which is defined as a stage from which the resultant solution is of the same composition as the solution adhering to the solids leaving the stage. Thus;

$$\frac{dm}{dt} = \frac{k' A(c_S - c)}{b} \qquad - \quad - \quad - \quad - \quad - \qquad 2.2$$

Where A = area of solid-liquid interphase

b = effective thickness of the liquid film surrounding the particle

C = concentration of the solute in the bulb of the solution at time, t.

C_S = concentration of the saturated solution in contact with the particle.

m = mass of solute transferred in time, t.

k^1 = diffusion coefficient

Hence,

$$dm = vdc \text{ and } \frac{dc}{dt} = \frac{k^{'}A(c_S - c)}{Vb} \tag{2.3}$$

Where v = volume of solution assumed to be constant.

Integrating thus, we have

$$\int \frac{dc}{c_s - c} = \int \frac{k^{'}A}{Vb} dt \tag{2.4}$$

This yield

$$\frac{c_s - c_0}{c_s - c} = \frac{k^{'}A}{Vb} dt \qquad - \quad - \quad - \quad - \tag{2.5}$$

If pure solvent is used initially, then $C_0 = 0$ and

$$1 - \frac{c}{c_S} = \ell^{-\left(k^{'}A/Vb\right)t} \qquad - \quad - \quad - \quad - \tag{2.6}$$

$$C = C_S\left[1 - \ell^{-\left(K^{1}A/bV\right)t}\right] \qquad - \quad - \quad - \tag{2.7}$$

2.3.4 Single stage extraction system

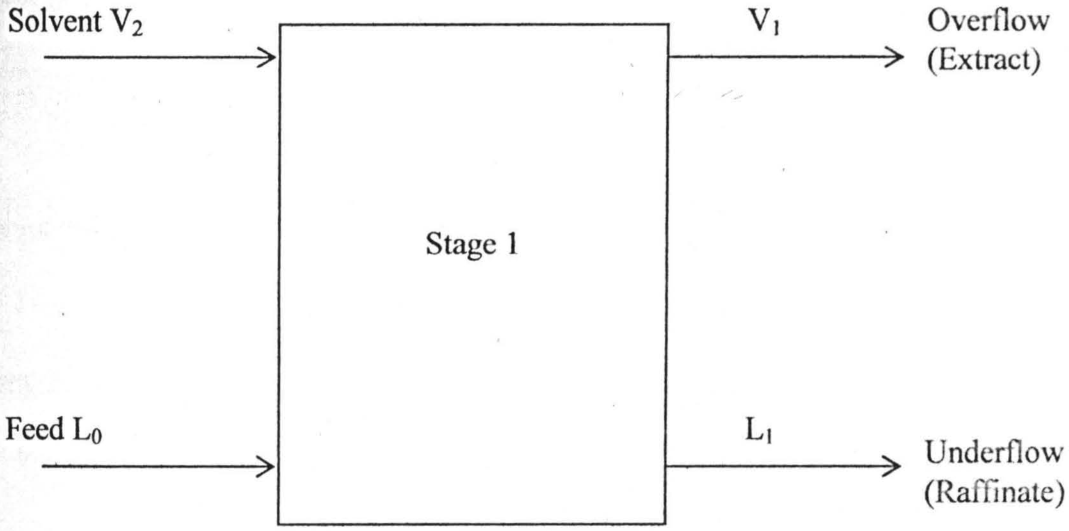

Figure 2.2: Single Stage Extraction System

Figure 2.2 above represents the nomenclature commonly used to describe the various streams in a single-stage system. A total material balance on the stage gives:

$$V_2 + L_0 = V_1 + L_1 = M \qquad 2.8$$

Where all the streams have units of kg/hr or g/minute (i.e., in mass flow rate).

The material balances for component A (solute) and component S (solvent) are, respectively.

$$V_2 y_{A,2} + L_0 x_{A,0} = V_1 y_{A,1} + L_1 x_{A,1} = MX_{A,M} \qquad 2.9$$

$$V_2 y_{S,2} + L_0 x_{S,0} = V_1 y_{S,1} + L_1 x_{S,1} = MX_{S,M} \qquad 2.10$$

Where:

x_A = weight fraction of solute in underflow

x_S = weight fraction of solvent in underflow

y_A = weight fraction of solute in overflow

y_S = weight fraction of solvent in overflow

Note that the symbols used in leaching are defined thus:

$$y = \frac{\text{mass of solute in overflow}}{\text{mass of solvent}}$$

$$x = \frac{\text{mass of solute in underflow}}{\text{mass of insoluble}}$$

$$z = \frac{\text{mass of solvent}\left(\text{overflow}\right)}{\text{mass of insoluble}\left(\text{underflow}\right)}$$

As usual, M, the mixture point can be located by the inverse lever rule. It is knowledgeable also that the exit equilibrium concentrations x_{A_1} (or x_{S1}) and y_{A1} (or

y_{S1}) must be collinear with M for an ideal stage (since the solution retained by the solids has the same composition as the overflow solution). If the solute is completely dissolved, assuming sufficient time and agitation are provided, and the resulting solution is not saturated, then the underflow stream may be considered to consist of a mixture of undissolved solids and a solution of composition

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2.3.5 Continuous countercurrent multistage leaching system

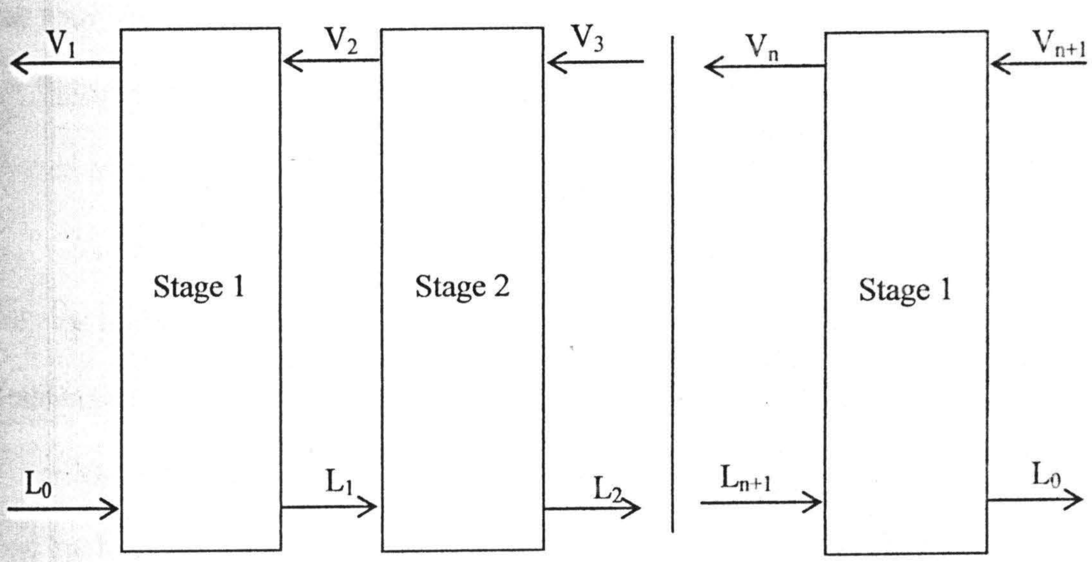


Figure 2.3: Countercurrent Multistage Leaching System

Figure 2.3 above is a schematic diagram of a countercurrent multistage leaching system containing n-ideal stages. The rate of feed to the system is arbitrarily designated by the symbol L_0, and the rate of solvent supply to the system by the symbol V_{n+1}.

When the system is operating under steady state conditions, the material balance equations on the entire system is as follows:

Total matérial balance is given thus:

$$V_{n+1} + L_o = V_1 + L_n \qquad \text{.. 2.11}$$

And the balance for either A (solute) and/or S (solvent) is given thus also:

$$L_0 x_0 + V_{n+1} y_{n+1} = L_n x_n + V_1 y_1 \qquad \text{.............................. 2.12}$$

Thus, a solute balance is given as follows:

$$y_{A,n+1} = \frac{L_n}{L_n + (V_1 - L_0)} x_{A,n} + \frac{V_1 y_{A,1} - L_0 x_{A,0}}{L_n + +(V_1 - L_0)} \qquad \text{........... 2.13}$$

Where:

L_n = Total mass of underflow leaving stage n

L_0 = Total mass of solids feed entering first stage

V_1 = Total mass of overflow leaving first stage

V_{1+1} = Total mass of fresh solvent entering stage n

L_n = Total mass of underflow leaving stage n

The operating line as represented from each stage is constant. Otherwise the slope of the operating line varies from stage to stage. Algebraic, graphical and analytical methods can be employed in conjunction with the operating line to calculate the number of equilibrium stages required to accomplish a specified separation.

2.3.6 The extraction battery: number of extraction stages

Figure 2.4 is a schematic diagram of an extraction battery with n-stages. The liquid phase is designated the overflow, the quantity of which is represented by V. The solid phase is designated the underflow, the quantity of which is represented by L. The extraction stages are numbered '1' after the mixing stage where the fresh solids first contact the solute – laden extract from the other stages and 'n' as the last stage where fresh solvent first enters the system and where the spent solids leave the system. The stage where extract from the first extraction stage contacts the fresh solids is called the mixing stage. It is different from the other stages in the extraction battery because at this stage, the solids have to absorb a much larger amount of solvent than in the other stages. The figure is shown below:

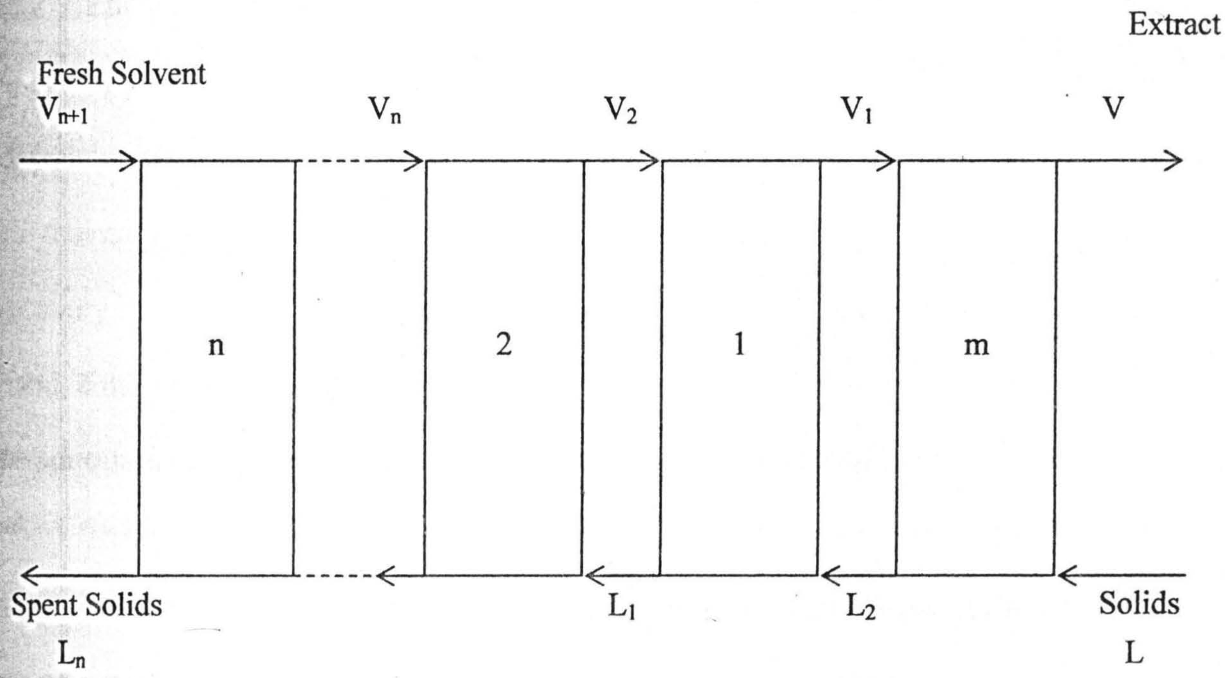


Figure 2.4: Schematic Diagram of an Extraction Battery for Multistage Countercurrent Extraction

Each of the stages is considered an ideal stage. Solute concentration in the underflow leaving stage 'n' must be at the designated level considered for completeness of the extraction process. Residual solute in the solids fraction must be maintained at a low level. If it is a valuable solute, the efficiency of solute recovery is limited only by the cost of adding more extraction stages. On the other hand, if the spent solids affect its value, then the number of extraction stages must be adequate to reduce the solute level to a minimum desirable value.

The number of extraction stages may be determined using a stage–by–stage material balance. Since the only conditions known are those at the entrance to and outlet from the extraction system, stage–by-stage material balancing involves setting up a system of equations which are solved simultaneously to determine the conditions of the solute concentration in the underflow and overflow leaving each stage. The procedure involves assuming a number of ideal stages, solving the equations, and determining if the calculated level of solute in the underflow from stage 'n' matches the specified value. The process is very tedious and a graphical method is generally used.

2.3.7 Determination of the number of extraction stages using the ponchon – savarit diagram

This graphical method for determining the number of extraction stages in a multistage extraction process involves the use of an X-Y diagram. The coordinates of this diagram are defined as follows:

$$Y = \frac{Solid}{Solute + solvent}$$

$$X = \frac{Solute}{Solute + solvent}$$

Where solid = concentration of insoluble solids,

Solute = concentration of solute, and

Solvent = concentration of solvent

Thus, the composition of any stream entering and leaving an extraction stage can be expressed in terms of the coordinates X, Y.

Figure 2.5 below is an X-Y diagram for a solid-liquid extraction process.

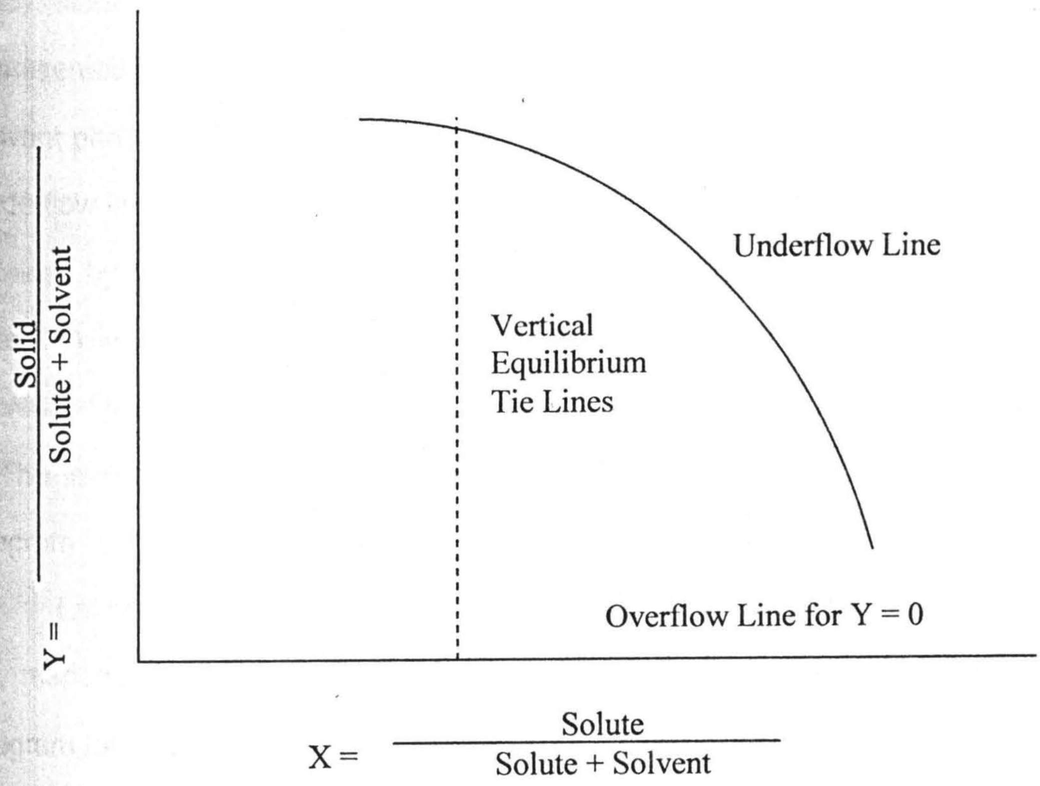


Figure 2.5: The Underflow and Overflow Curves and Equilibrium Tie Lines on a Ponchon-Savarit Diagram

The overflow line represents the composition of the solvent phase leaving each stage. If no solids entrainment occurs, the overflow line should be represented by $Y = 0$. The underflow line is dependent upon how much of the solvent phase is retained by the solids in moving from one stage to the next. The underflow line is linear if the solvent retained is constant and curved if the solvent retained by the solids varies with the concentration of the solute. Variable solvent retention in the underflow occurs when the presence of the solute significantly increases the viscosity of the solvent phase.

i. The lever rule in plotting the position of a mixture of two streams in an x-y diagram

Let two streams with mass R and S, and with coordinates X_r, Y_r and X_s and Y_s, respectively, be mixed together to form T with coordinates X_t and Y_t. The diagram for the material balance is shown in Figure 2.7-I. If R and S consist of only solute and solvent, solute results in

$$RX_r + SX_s = (R+S)X_t \quad \text{...} \; 2.14$$

A solids balance gives:

$$RY_r + SY_s = (R+S)Y_t \quad \text{...} \; 2.14$$

Solving for R in equation 2.14 and S in equation 2.15 and dividing yields:

$$\frac{R}{S} = \frac{X_s + X_t}{X_t + X_r} = \frac{Y_s + Y_t}{Y_t + Y_r} \quad \text{...} \; 2.16$$

Figure 2.7-II represents the term involving Y in equation 2.16. The coordinate for the mixture should always be between those of its components. The ratio of the distance between the line $Y = Y_s$ and the line $Y = Y_t$, represented on the diagram by A, and the distance between the line $Y = Y_t$ and the line $Y = Y_r$, represented on the diagram by B, equals the ratio of the mass of R and S.

Figure 2.7-III represents the term involving X in equation 2.16. Again, the ratio of the masses of R and X equals the ratio of distance C to distance D in the X-Y diagram. The composite of the material balance is drawn in figure 2.7-IV. The coordinates of points S and R, when plotted on the X-Y diagram and joined together by a straight line, result in the point representing T being on the line between S and R. The ratio of the distance on the line between S and T, represented by E, and that between R and T, represented by F, is the ratio of the mass of R to S. These are shown in Figure 2.7-I-IV thus:

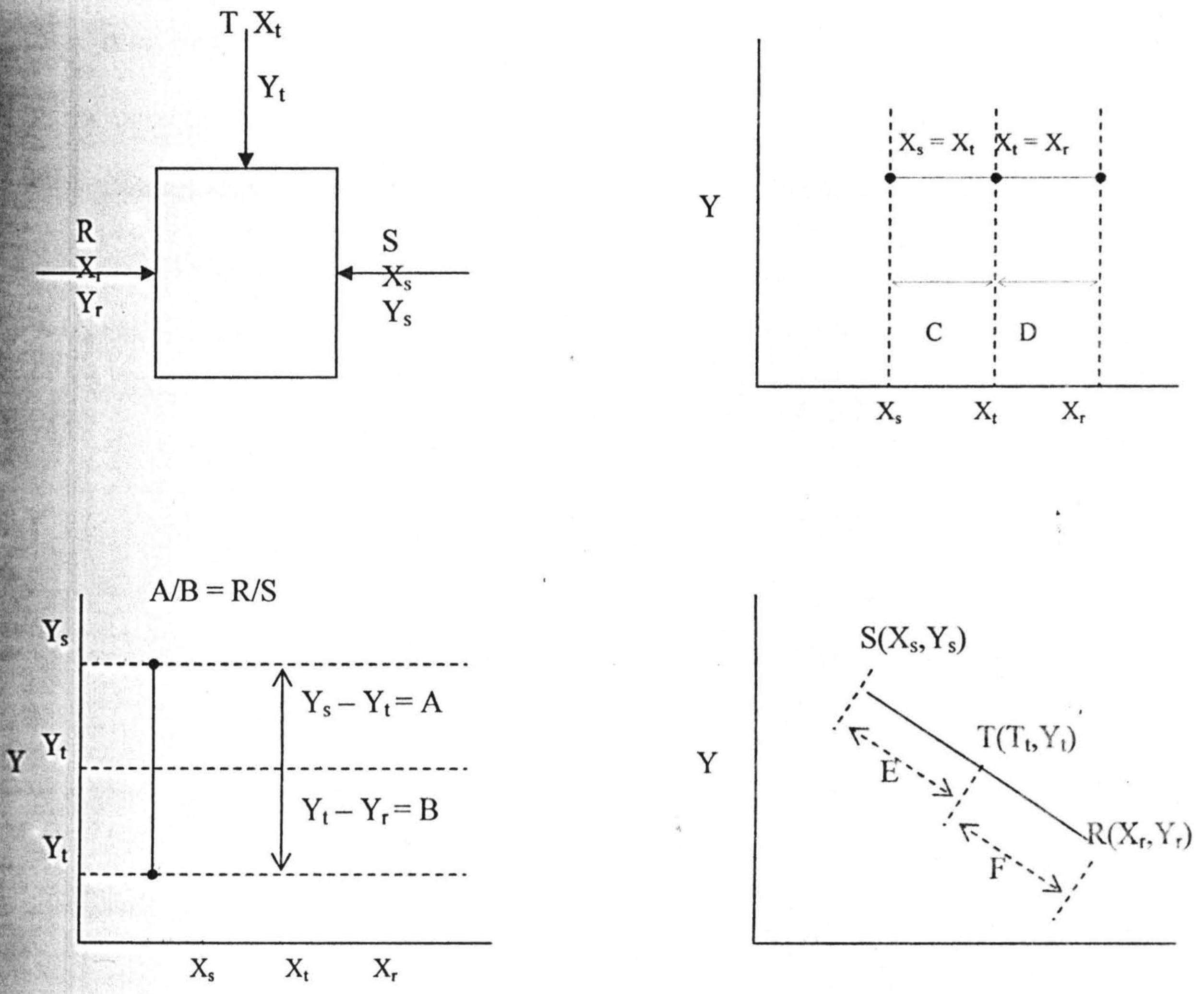


Figure 2.6: Representation of a Material Balance on the X-Y Diagram, and the Lever Rule for Plotting the Mass Ratio Of Process Streams as the Ratio of the Distances Between Points on the X-Y Diagram.

These principles show that a material balance can be shown on the X-Y diagram, with each process stream represented as a point on the diagram, and any mixture of streams can be represented by a point on the line drawn between the coordinates of the components of the mixture. Exact positioning of the location of the point representing the mixture can be done using the lever rule on distances between the points as represented in Figure 2.7- IV.

ii. Material and graphical representation of point j in the Ponchon-Savarit diagram

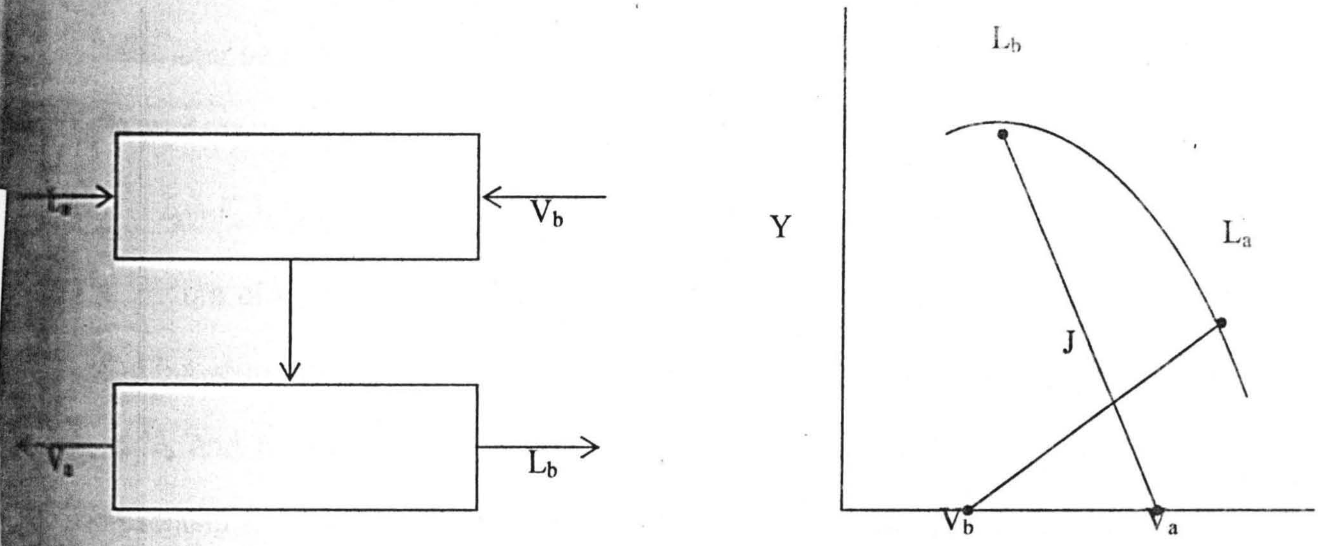


Figure 2.7: Representation of Point J as the Intersection of Lines Connecting Four Process Streams Entering and Leaving a System

Consider underflow, L_b, mixing with overflow, V_a, and the solid and solvent phases later separated to form the overflow stream, V_b, and the underflow stream, L_a. A total mass balance gives:

$$L_b + V_a = V_b + L_a = J \quad \text{...} \ 2.17$$

The mixture of L_b and V_a forms the point J, as shown in Figure 2.8 above. A line drawn between the coordinates of L_b and V_a, and another line drawn between the point V_b and L_a, intersect at point J. Point J may be used to help plot points representing the incoming and exiting streams in an extraction battery from the solvent- solids ratio.

Iii. Mathematical and graphical representation of point p

Point P is a mixture which results when the underflow leaving a stage mixes with the overflow entering that stage. It is also the mixture of the overflow leaving a stage and the underflow entering that stage. The mixture representing point P is shown schematically in Figure 2.8 below.

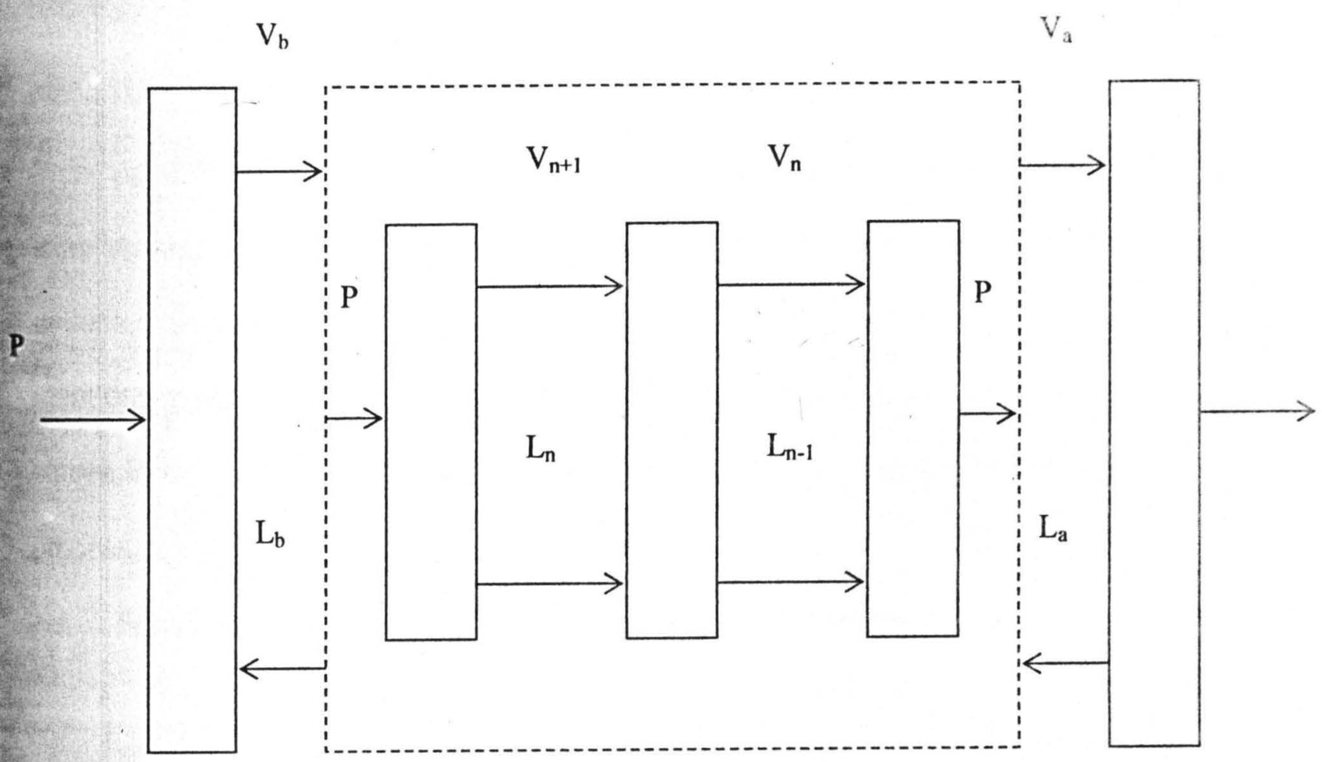


Figure 2.8: Representation of Point P as the Common Point through which all Lines Connecting the Underflow Leaving and the Overflow Entering an Extraction Stage must pass

A total mass balance around the system represented by the dotted line in Figure 2.8 gives:

$$V_b + L_b = -V_a + L_a = -V_{n+1} + L_n = P \quad \text{.................................. 2.18}$$

Thus, point P is an extrapolation of the line which joins V_b and L_b, the line which joins V_a and L_a, and the line which joins V_{n+1} and L_n. All lines which join the underflow stream leaving a stage and the overflow stream entering that stage all meet at a common point, P. This is a basic principle used to draw the successive stages in a Ponchon-Savarit diagram for stage-by-stage analysis of an extraction process.

iv. Equation of the operation line and representation on the x-y diagram

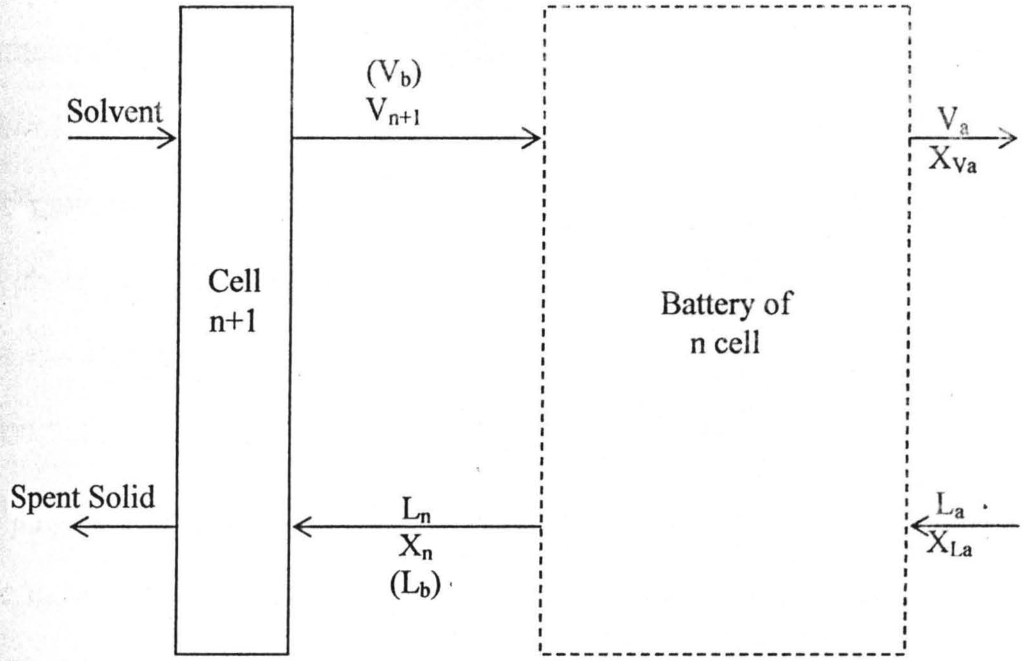


Figure 2.9: Diagram of the System Used to make a Material Balance for Deriving the Equation of the Operating Line in a Ponchon-Savarit Diagram

Figure 2.9 above represents an extraction battery with n cells. Each stage in the extractor may also be called an extraction cell. Cell n+1 is the cell to which underflow enters after cell n. The subscripts on the overflow stream, v, and the underflow stream, L, represent the cell from which the stream is leaving. Thus, the solvent phase entering cell n comes from cell n+1 and is designated V_{n+1}, and the solid phase leaving cell n is designated, L_n. If cell n is the last stage in the extraction battery, then, the following expression is attainable.

$V_{n+1} = V_b$ And $L_n = L_b$

Let V represent the mass of solute and solvent in an overflow stream, and let, L, represent the mass of solute and solvent in an underflow stream. A material balance around the battery of n cells is a follows.

Total mass balance:

$$V_{n+1} + L_a = L_n + V_a \quad \text{.......} \quad 2.19$$

or $V_{n+1} = L_n + V_a - L_a$

Solute balance:

$$V_{n+1} + X_{n+1} = V_a X_{va} + L_a X_{La} + L_n X_n \quad \text{.......} \quad 2.20$$

Substituting equation 2.19 in equation 2.20 and solving for X_{n+1};

$$X_{n+1} + \frac{L_n X_n}{L_n + V_a - L_a} + \frac{V_a X_{va} - L_a X_{La}}{L_n + V_a - L_a} \quad \text{.......} \quad 2.21$$

Equation 2.21 is the equation of an operating line. It shows that the points representing overflow and underflow leaving stage n+1, which have the X-coordinate X_{n+1}, are on the same line drawn through the points representing overflow and underflow leaving stage, n, which have the X-coordinates, X_n.

It can be shown that the coordinates of point P represented by equation 2.18 also satisfy equation 2.21. Thus, a line drawn from a point representing stage n to point P allows the determination of a point representing the coordinate of stage n+1.

2.3.8 Factorial analysis of the experiment

Factorial analysis is a methodological design plan followed to conduct the experiment. In factorial experiments, some factors are varied while others are kept constant which is followed by the real factorial analysis i.e the selective comparison of the variable factors (independent factors) to develop the response (dependent terms) of the process. Its mathematical representation that provides the relationship of these factors to one another and also the effect of these factors on the overall rate of a process.

In most cases, the factors affecting a process are obtained from literature; however, they can be predicted or assumed by considering the relationship between the physical or chemical properties of the system understudy.

2.3.9 Methods of statistical analysis

Statistical analysis is a body of techniques for deriving or organizing statistics, and for determining their essential significance. There are two types of data to which statistical methods may be applied: variable and attribute. Data are of the variable type when they can be considered, from a practical standpoint, as having some continuously measurable characteristics. Attribute on the other hand, are non-variable classification in that they are in the form of counts or number of things called enumerations.

The types of statistical analysis, which can apply to either variable or attribute data, are the following:

i. To test a given hypothesis concerning some observed characteristics
ii. To determine a reliable estimate of some factual value
iii. To represent a physical situation functionally

The reason for such an analysis is the fact that all data are to some extent, one way or the other, subject to chance error. These chance errors may arise whether the problem involves estimation-the test of a hypothesis-or the development of a reliable model. As a means of testing a hypothesis or determine the reliability of some factual value, a statistically designed experiment should be use. Basically, these design experiments enable the analyst to determine with a pre-assigned degree of confidence, the degree of variation in the experimental determinations which is due to chance and that which is the result of some possible known or unknown influence.

In addition, a statistical experiment is designed from the standpoint of been able to make a given number of reliable generalizations from a minimum number of experiments. It is for this reason that in modern design of experiment,.the statistical approach is needed from the beginning.

Statistical methods are predicted on the single concept of variability. Through it, a basis is determined for experimental design and analysis of data. In this sense, statistical methods are concerned with deriving maximum information from a given set of the data (analysis), and conversely minimizing the amount of the data (experiment design) to derive specific information. Some of the fundamental concepts which are utilized to describe statistical analysis methods includes standard deviation, true value, degree of freedom, normal frequency distribution and theoretical model distribution.

2.3.10 Design of experiment

Experimental designs are particularly applied to the study of process variables and how they affect the product. The experiments employ regression analysis, i.e their quantitative effects, to determine the effect of the variables. Data, which serve as the basis for regression analysis, can be obtained from the recorded experimental data. A selected procedure is employed to develop a controlled combination of variables so as to determine a reliable analysis. Three basic types of experiments are frequently used in the chemical industries and these are: factorial, fractional factorial and Box-Wilson.

For a process application, which includes the study of three variables, say, production method, particle size and time on the rate of extraction, a factorial experiment could be effectively used. For this a reasonable number of operating levels of production method, sample particle size and extraction time would be selected and with the selected levels for each, the factorial experiment would require all possible combinations. In the case where levels were specified this would include 27 sets of tests. These test result would be interpreted by a functional representation (regression analysis) and an analysis of variance. For process application including more than three variables, the number of test becomes excessive with a factorial experiment; hence, the fractional factorial or the Box-Wilson experiment can be effectively used.

2.4 Types of Extractor Systems and their Applicability

Three main types of extractor systems can be identified each of which finds application in several uses.

2.4.1 Mixer settlers

Mixer – settlers are used when there will only be one equilibrium stage in the process. For such a system, the two phases are added and mixed. Due to their density differences, one phase will settle out and the mixture will be separated. The downfall to this type of extractor is that it requires a large – volume vessel and a high liquid demand. (Seader and Henley, 1999).

2.4.2 Contacting columns

Contacting columns are practical for most solid-liquid extracting systems. The packing, trays, or sprays increase the surface area in which the two phases can intermingle. This also allows for a longer flow path that the solution can travel through. In the selection of a packing, it is necessary to select a material that is wetted by the continuous phase (Seader and Henley, 1999). Lastly the flow in a column should always be counter – current.

2.4.3 Centrifugal contractors

Centrifugal contractors are ideal for systems in which the density difference is less than 4%. In addition, this type of system should be utilized if process requires many equilibrium stages (Strigle, 1994). In these systems, mechanical devices are used to agitate the mixture to increase the interfacial area and decrease mass transfer resistances (Seader and Henley, 1999). Many different types of centrifugal contractors exist, and each has its own guidelines for operation and selection. A more detailed view can be found in Seader and Henley, 1999.

Table 2.2: Advantages and Disadvantages of the Various Solid-Liquid Extractor Types (Seader and Henley, 1999).

Extractor Types	**Advantages**	**Disadvantages**
Mixer – Settlers	Efficient low headroom induces good contacting can handle any number of stages.	Large floor High set-up costs High operation costs
Columns (Without agitation)	Small investment costs low operating costs.	High head room Difficult to scale up from laboratory Less efficient than mixer – settler.
Columns (With agitation)	Good dispersion Low investment costs Can handle any number of stages.	Difficult to separate small density differences Does not tolerate high flow ratio
Centrifugal Extractors	Can separate small density differences short holding time small liquid inventory.	High set-up cost High operating and maintenance costs cannot handle many stages.

2.5 Role of Solvent in the Extraction Process

Ideal solvents for extraction should meet the following criteria.

i. Selective in action: It should thoroughly dissolve oils and fats as far as possible and should not dissolve other constituents contained in the oil.

ii. Ease of removal: It should be volatile and should not disintegrate on distillation.

iii. Corrosive tendency: The solvent must not chemically react with the materials from which the processing equipment is fabricated or with any oil component.

iv. Boiling point: It should have a constant boiling point. It is desirable that the solvent should be removed at the lowest practicable temperature, otherwise the application of excessive heat may have adverse effect on oils and fats.

v. Stability: It should be recoverable in the same stable state so as to follow repeated use of the solvent.

vi. Availability: The solvent should be available and should not deteriorate during storage.

vii. Price structure: It should be cheap and readily available in appreciable quantity.

viii. Toxicity: It should not be toxic and should have no deleterious effect on either the residue or the oil. Its vapour too must not create any toxic effect when inhaled.

ix. Solubility in water: The solvent should be insoluble in water.

Despite the fact that n-hexane and anhydrous ethanol has been established as a universally accepted solvent for oil extraction because of its ability and disposition to the above criteria, choice can be made for other solvents such as petroleum spirit, benzene, acetone and petroleum ether.

2.6 Steam Distillation

Thermal degradation problems or the need for very high temperature heating media can result in a significant extraction problem for organic mixtures of oil-bearing fruits and seeds. Thus, steam distillation is sometimes used to solve temperature-related problems.

Steam distillation is a method used for the separation of high boiling substances from non-volatile impurity or for the removal of very high boiling volatile impurities from still higher boiling substances.

It involves the use of steam to percolate and vaporized out the essential oils from the plant material, with the subsequent condensation of steam and essential oil prior to their separation. It involves the communition of material usually in a large vat and the application of live steam through the material. The steam percolates the material and vaporizes the oil from the matrix, from which the mixture of steam and essential oil goes to a condenser where it is liquefied. Subsequently, the liquid mixture is separated using a setting tank, separating funnel or similar vessel.

From chemistry, it is known that a liquid boils when the vapour pressure of the liquid equals the external pressure of the system. Hence, boiling temperature can be reduced by reducing the pressure of the system either by boiling under a vacuum or by adding an inert vapour which contributes to the vapour pressure allowing the liquid to boil at a lower temperature. This is the exact principles upon which steam distillation is based.

Three different methods of steam distillation are practiced (Kirk Othmer, 1983; New Encyclopedia Britannica, Vols. 1 and 13). In the first method, a vessel containing water and sample material is heated by a direct flame and the water vapour and volatile oil are recovered by a water – cooled condenser. In the second method, the sample material is suspended on a grid above the water level and steam from a second vessel is introduced under the grid. The volatiles are condensed and the oil is separated; and in the third process, the vessel containing the sample material on a grid is heated to prevent condensation of steam so that dry distillation is attained.

In steam distillation, live steam is fed directly into the bottom of the column, serving as the heating medium and the source of a vapour steam. The steam serves, in effect to lower the pressure of the distillation for the organic mixture, since the steam occupies partial pressure within the vapour phase and thus the partial pressure of the organic components add up to less than the total pressure. The lower sum of partial pressure for the organic in turn lead to lower temperatures for the distillation.

A necessary condition for the application of steam distillation is that the desired product (whether for volatile material passing over from the still or the non-volatile residue remaining in the still) must be practically immiscible with water (McGraw, D.R., et al. ; 1999, www.uwichill.edu). The condition that the product desired is not soluble in water means that the distillate will form two layers on condensation from which the product desired can be taken off as one layer and the water as the other layer. (Philip, A. 1997, Perry and Green, 1997).

An advantage of steam distillation, in addition to the effective pressure reduction, is that when condensed, the distillate will often break into two liquid phase: the water not diluting the organic distillate product significantly.

A disadvantage is that the water effluent contains organic pollutants and requires treatment before discharge or return to a boiler for steam production or generation.

2.6.1 Steam and its role in the distillation

In steam distillation, the steam as a heat transfer medium possesses a high heat content, can be distributed easily, its flow is easily controllable, is non-combustible and will not support combustion, is non-toxic and relatively non-corrosive and is produced from water which is relatively cheap and abundant (Kern, D. Q., 1950). Thus, the steam serves to reduce or lower the pressure of the distillation for the organic sample material since the steam occupies partial pressure within the vapour phase and hence, the partial pressure of the organic component add up to less than the total pressure. The lower sum of partial pressure for the organic mixture in turn leads to lower temperature of the distillation (Kern, D. Q., 1950, Badger, et al; 1982).

2.6.2 Calculations in steam distillation

In steam distillation, the vapour generated by boiling the liquid is withdrawn from contact with the liquid (water) and condensed as fast as it is formed.

Consider a batch of W_0 moles of liquid. Suppose that at any given time during the distillation there are W moles of liquid left in the still (tank). At this time, let the liquid composition be x and the vapour composition y.

The total amount of component A in the liquid will be xW. Suppose a very small amount of liquid dW is vapourized. During the vaporization, the liquid composition will diminish from x to x – dx, and the weight of the liquid will diminish from W to W – dW. There will be left in the still $(x-dx)(w-dw)$ moles of A, while the amount ydw has been removed from the still. A material-balance equation with respect to component A is, therefore:

$$xW = (x-dx)(w-dw) + ydw \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \; 2.22$$

Expanding equation 2.44, it becomes:

$$xW = xW - xdW + dxdW - Wdx + ydW \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \; 2.23$$

From equation 2.23, it can be observed that there is a second-order differential equation which can be neglected, thus, the equation may be rewritten as:

$$\frac{dW}{W} = \frac{dx}{y-x} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \; 2.24$$

Integrating equation 2.24. between the limits of W_0, the initial weight in moles, and W_1, the final weight in moles, on the left hand sides, and between the limits x_0, the initial concentration, and x_1, the final concentration, on the right-hand side, equation 2.24 becomes:

$$\int_{W_1}^{W_0}\frac{dW}{W} = \ln\frac{W_0}{W_1} = \int_{x_1}^{x_0}\frac{dx}{y-x} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \ 2.25$$

Equation 2.25 above is the well known Rayleigh equation where the function $dx/(y-x)$ can be graphically integrated to give the relationship between x and y.

2.6.3 Heat and material balance calculations

In the design of a steam extraction tank still, with condensing units; the primary information required includes the determination of the number of plates or the packed height and the column diameter required.

Now, suppose that F g/min of a mixture of components A and B, containing, x_f, weight fraction of component A, is to be separated continuously operating at a constant pressure, P. The product i.e. distillate is to contain, x_0, and the residue (raffinate) is to contain x_w, weight fraction of component A. The temperatures of the feed, extract or distillate and the residue (raffinate) are specified and are t_f, t_0 and t_w respectively. The corresponding enthalpies of these streams are h_f, h_D and h_W.

The flow rates of the terminal streams D and W may be calculated from over-all material balances.

Under steady-state conditions,

$$F = D + W \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \ 2.26$$

$$Fxf = Dx_D + Wx_W \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \ 2.27$$

Solving equations 2.26 and 2.27 simultaneously for the distillate, D, and residue (raffinate), W,

$$Fxf = Dx_D + (F - D)x_W \quad \text{2.28}$$

$$D = \frac{F(x_f - x_w)}{x_D - x_w} \quad \text{2.29}$$

The amount of stream w is similarly obtained thus as:

$$W = \frac{F(x_D - x_F)}{x_D - x_w} \quad \text{2.30}$$

Now, assuming no heat loss from the system to the surroundings, the energy balance for the system may be written as:

$$Fh_f + q_r = Dh_D + Wh_W + q_C \quad \text{2.31}$$

Where q_r = heat added in heating mantle (reboiler), Btu/hr

q_C = heat removed in condenser, Btu/hr.

By equation 2.31, it implies that if the heat input to the heater (reboiler) is fixed, then the heat removed in the condenser cannot be varied but is fixed at a definite valve. Likewise, if the heat removed in the condenser is set, then the heat input to the reboiler is also fixed.

Thus, the energy and material balances for the condenser alone maybe written as:

$$V_1 = L_0 + D \quad \text{2.32}$$

$$V_1y_1 = L_0x_0 + Dx_0 \quad \text{2.33}$$

$$V_1H_1 = q_C + L_0h_0 + Dh_0 \quad \text{2.34}$$

Now, assuming that all the vapour entering the condenser is condensed, then, $y_1 = x_0 = x_D$ and $h_0 = h_D$, and solving equation 2.34 for q_C, we have.

$$q_C = V_1H_1 - (L_0 + D)h_D \quad \text{...} \quad 2.35$$

Substituting for V_1 from equation 2.32 we have

$$q_C = (L_0 + D)H_1 - (L_0 + D)h_D = (L_0 + D)(H_1 - h_D) \quad \text{..........} \quad 2.36$$

And dividing both side of equation 2.58 by D, we have

$$\frac{q_C}{D} = \left(\frac{L_0}{D} + 1\right)(H_1 - h_D) \quad \text{.......................................} \quad 2.37$$

Thus, the input heat may now be calculated from equation 2.37

Now, the following nomenclature is here defined

V = Vapour rate, L = Liquid rate, H = Enthalpy of vapour stream, h = enthalpy of liquid stream, F = Feed rate.

2.6.4 Effect of heat losses

The effect of heat losses from a steam extraction tank still may be taken into account by including such a term in the energy-balance equation (2.30)

Representing q_h as the total heat losses in Btu per hour, the energy balance becomes:

$$Fh_f + q_r = Dh_D + Wh_W + q_C + q_h \quad \text{..................................} \quad 2.38$$

The material balance equation however remains unchanged.

2.7 Classification of Oils

Generally, oil occurs in the form of insoluble droplets within the plant cells. They occur in endocarp mainly in the cotyledons and endosperm, in the pericarp and mesocarp. However, the obtained oil differ from one another physically and chemically. In order to identify a specific one, oils are classified under three main groups based on their ability to absorb oxygen from the atmosphere. These groups include drying, semi-drying and non-drying oils.

i. Drying oils

These are oils that absorb oxygen on exposure to air. Oils of these types are usually rich in glycosides of unsaturated fatty acid, particularly linoleic acid with few oleic compounds. They are therefore useful as solvent for pigments in the paint and varnish industries. These oils are characterized by iodine values of more than 130 and form a tough elastic but resistant film.

ii Semi-drying oils

These are oils characterized by the presence of large amount of linoleic acid and saturated acids. These oils absorb oxygen slowly and produce a soft film after prolonged exposure to the atmosphere. This film is not elastic. The iodine ranges from 100 to 130 thereby including cotton oil in its class. Corn seed oil is another example

iii Non- drying oils

These oils remain inert to atmospheric oxygen even after prolonged exposure and hence, remain liquid at room temperature. No film is formed. They are largely glycerides of saturated acids and oleic acids. The iodine value is less than 100. Examples include castor oil and olive oil.

2.7.1 Analysis and testing of oils

The analysis and testing of oils are needed for an assessment of quality and purity as well as for their identification. A number of physical and chemical properties have been established for these purposes. Although many of them are empirical, others are quite specific measurements of the characteristics of the oil. Those most commonly used to establish identity are: saponification value, iodine value, refractive index and the Reichert-Polenske-Kirscher values. Colour reactions can also give useful guidance in identifying an essential oil. Measurements of melting point are of debatable value.

Oils are natural products and are therefore subject to some variation in composition so that the properties fall within a range. Even, so from time to time, oils become available which have properties outside the normal range. They may vary according to the botanical variety, climatic conditions, soil composition, rainfall and temperature.

Other data are determined on the oils in order to assess quality. Of these, the free fatty acid content, peroxide value, benzidine or anisidine value, moisture, impurities and unsaponifiable matter all help to give an adequate measure of the quality of the oil for which it serves to achieve.

If the oil is required for other special applications additional tests may be demanded in order to check suitability.

2.7.2 Methods and significance of tests

Each of the test used in examining the avocado oil is chosen to measure a specific characteristics of the oil and hence, the determination of the composition and property or identity of the oil under investigation.

i. Saponification value

This is a measure of the mean molecular weight of the fatty acids present in the oil. The process of saponification is the hydrolysis of triglycerides into glycerol and the potassium salt of the fatty acids, using a solution of potassium hydroxide in alcohol. This process measures the amount of alkali which is required to combine with the free acids liberated and from this the equivalent weight and molecular weight of the fatty acid can be determined.

ii. Iodine value

This is a measure of the proportion of unsaturated acids present. The test measures the amount of iodine which can be absorbed by the unsaturated acids. The iodine value will give a figure for the total degree of unsaturation, expressed as the percentage of iodine absorbed by the oil.

iii. Reichert, Poleuskew and Kirschner Value

These values are all determined at the same time. They measure the amount of steam-volatile fatty acids which can be recovered under standard conditions. Low molecular weight acids are steam volatile under the test conditions and are separated from the higher acids. The acids are further subdivided into those which are water-soluble and the water-insoluble ones.

The Reichert value measures the water-soluble acids, Polenske value, the insoluble acids in addition to the Kirschner value. The test is empirical, and the procedure must be followed meticulously if the results are to have any value. The greatest significance of these constants is in checking for adulteration, or in examining mixtures containing oils.

iv. Unsaponifiable matter

This is a measurement of the water-insoluble components produced after heating the oils with potassium hydroxide (caustic refining). During this process, all the glycerides (aldehydes) are broken down to form glycerol and potassium salts of the fatty acids. Both of these products are water soluble and the insoluble hydrocarbons, sterols and fatty alcohols can be extracted from the aqueous solution and their proportion measured. The amount of unsaponifiable matter found in avocado oil is about 1.5% which by itself is a very valuable component useful in many creams and medical ointments.

v. Refractive index

This is a physical attribute of triglyceride, measured by the angle through which a beam of height is bent when passed through a thin film of the avocado oil. The index of each oil falls within a range and can be used as characteristics of the oil in checking purity or searching for components of a mixture. It is temperature dependent.

vi. Specific gravity

The specific gravity is the ratio of the weight of the substance to the weight of an equal volume of water at specific temperature. The specific gravity of oil is always less then one.

vii. Free fatty acid

Free fatty acid results from chemical or enzymatic hydrolysis of the fatty acid glycerides. Its presence is a measure of the quality of the unrefined and refined oils.

viii. Peroxide value

This is a measure of primary oxidation of the oil whose product is hydrocarbons. These hydrocarbons are further oxidized to water which causes rancidity of the oil on storage. Thus the peroxide value is an indication of the presence or absence of rancidity in the oil which defines the degree to which oxidation has occurred before examination.

CHAPTER THREE

3.0 METHODOLOGY

The experimental procedures involved the procurement of materials and equipment; pretreatment of avocado apple and peels (pericarp); steam distillation and extraction of the oil from the avocado's pericarp; characterization of both the crude and refined oil and the process and equipment design of the unit operations.

The procedure followed in conducting the extraction experiment, the method used in evaluating the compositions and material analysis and the instrumentation used in the course of the experiment provided the basis of the factorial design method. A step-by-step procedure was followed in the preparation of the material (avocado apples pericarp/peels) so as to provide for the desired particle size free of moisture and foreign particles.

In this work, only the unconventional materials and major equipment set-up used in the experiment are given in Tables 3.1 and 3.2 respectively.

Table 3.1: List of Materials Used for the Experiment

Materials	Source	Research code name	Comments
Avocado Apple	Obollo-Afor Market, Nsukka, Enugu – Nigeria	AVOA	Fresh greenish to very dark green in colour
Avocado Peel/Pericarp	Avocado Apple	AVOP	Amber, dark brown to appear black in colour
N-Hexane	WR international Ltd Poole, BH15 1TD, England	UN 1208 (N – H)	Bp 60°C n-hexane >>99% BDH (ANALAR) Highly flammable volatile liquid
Anhydrous Ethanol	BDH Chemicals Poole, England	UN 1170 (ABS)	99.7–100% Ethanol, bp 78°C analytical grade

Table 3.2: List of Equipment Used for the Study

Materials	**Source**	Model	Comment
Steam Extraction Still	Awal industries Kaduna	NA	Stainless steel material
Soxhlet Extractor	Pyrex, England	BDB 24	Glass apparatus
Separating Funnels and Settling Tanks	AG, Borosilicate, England	BS2021	Glass apparatus
Connecting Slits	Quickfit, England	DA23	Glass apparatus
Weighing Balance	Ohaus, USA	B300D	Digital display
Measuring Cylinder	Technico, England	BS604	Glass apparatus
Condensers	AG, Borosilicate, England	BS1848	Glass apparatus
Oven	Gallenkamp, England	CE94	Vacuum drier
Stack of Sieves	Chemical Engineering Laboratory, FUT, Minna	SOS241	Stainless steel
Heating mantle	Electrothermal Britain Everest, China Local, Minna	B300 ANI020 LM	Stainless case Ceramic-stainless case Galvanized steel case

3.1 Procurement and Pretreatment of Avocado Apple and Peels (Pericarp)

The avocado apple was sourced locally form Obollo-Afor market, Nsukka, in Enugu state of Nigeria. The fruits were thoroughly washed and screened to remove foreign bodies. The pericarp/peel was removed from the mesocarp and the endocarp (seed). The pericarp/peel was dried at ambient temperature (25°C) for five days to eliminate moisture. The dried pericarp was thereafter reduced to particle sizes ranging from 0.75 mm to 2.00 mm

3.2 Experimental Procedure

3.2.1 Steam distillation of oil from AVOP

Ten (10) grams of AVOP and varying volumes of 1000 ml, 1250 ml and 1500 ml of water for steam generation were used alternatively at slow or low, moderate or medium and fast or high heating rates respectively. 1000 ml of water was introduced into the bottom chamber of the still. The chamber was covered with a perforated metal plate in which a white filter cloth was placed. 10 gram of AVOP was then placed on the filter cloth. This was further covered with white filter cloth. The last perforated metal plate was placed on the top compartment. Finally, the still was made air tight with the last covering to prevent the escape of the steam – oil mixture during heating. The set-up was then connected to a condenser via a pipe fixed at the top of the extraction still where an opening had been made. The delivery tube from the condenser was connected to the separating funnel to receive the mixture of steam and oil on condensation. The set–up was then mounted and connected to three heating sources alternatively for extraction times of 30, 60, 90, 120, 150, and 180 mins. respectively. At the end of the time intervals, the set-up was switched-off and allowed to cool. The water-oil mixture was decanted to separate the oil from the water at the water-oil interface. Thereafter the mass of the AVOP after extraction and drying in an electric oven was recorded. The mass of oil extracted was also recorded.

This procedure was repeated for 1250 ml and 1500 ml of water with 10 gram of AVOP at extraction times of 30, 60, 90, 120, 150 and 180 mins. at the three heating rates respectively. The steam-sample ratio, steam and sample utilization and steam supply rate for each extraction times were recorded. The laboratory set up of the steam distillation apparatus is shown in Figure 3.1 below while the sketch of the process is given in Figure 3.1a.

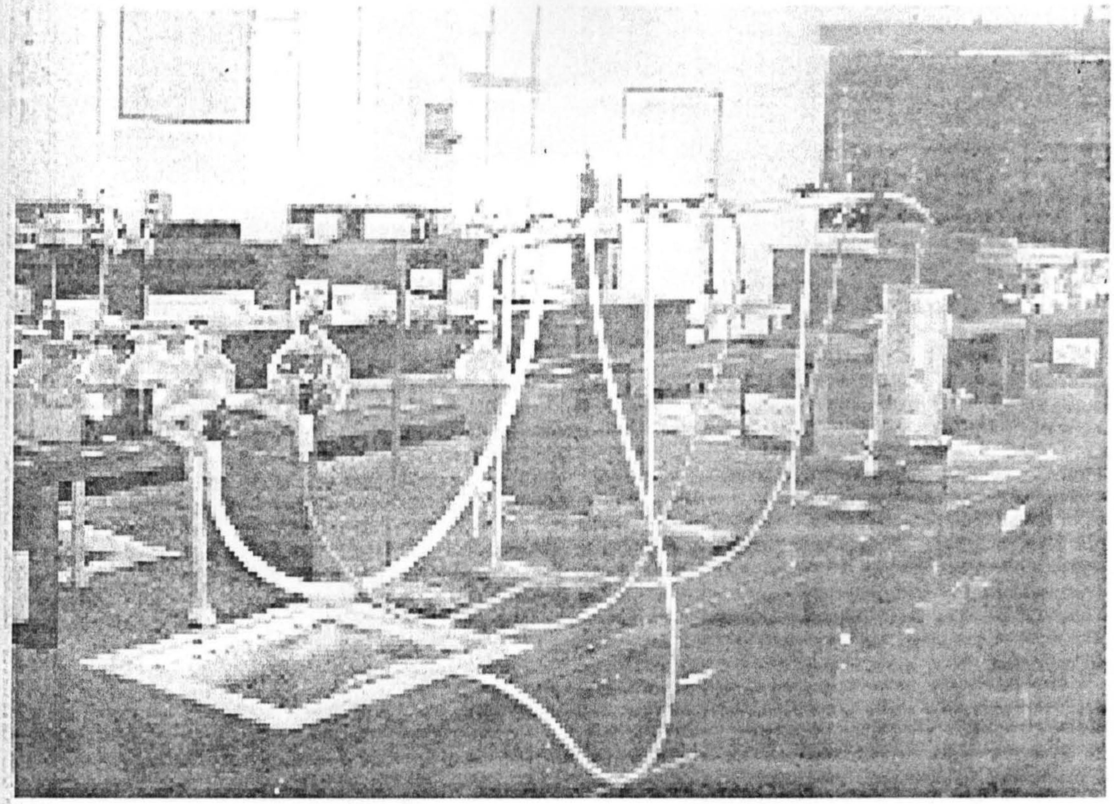


Figure 3.1 Laboratory Set-Up of the Steam Distillation Apparatus/Equipment

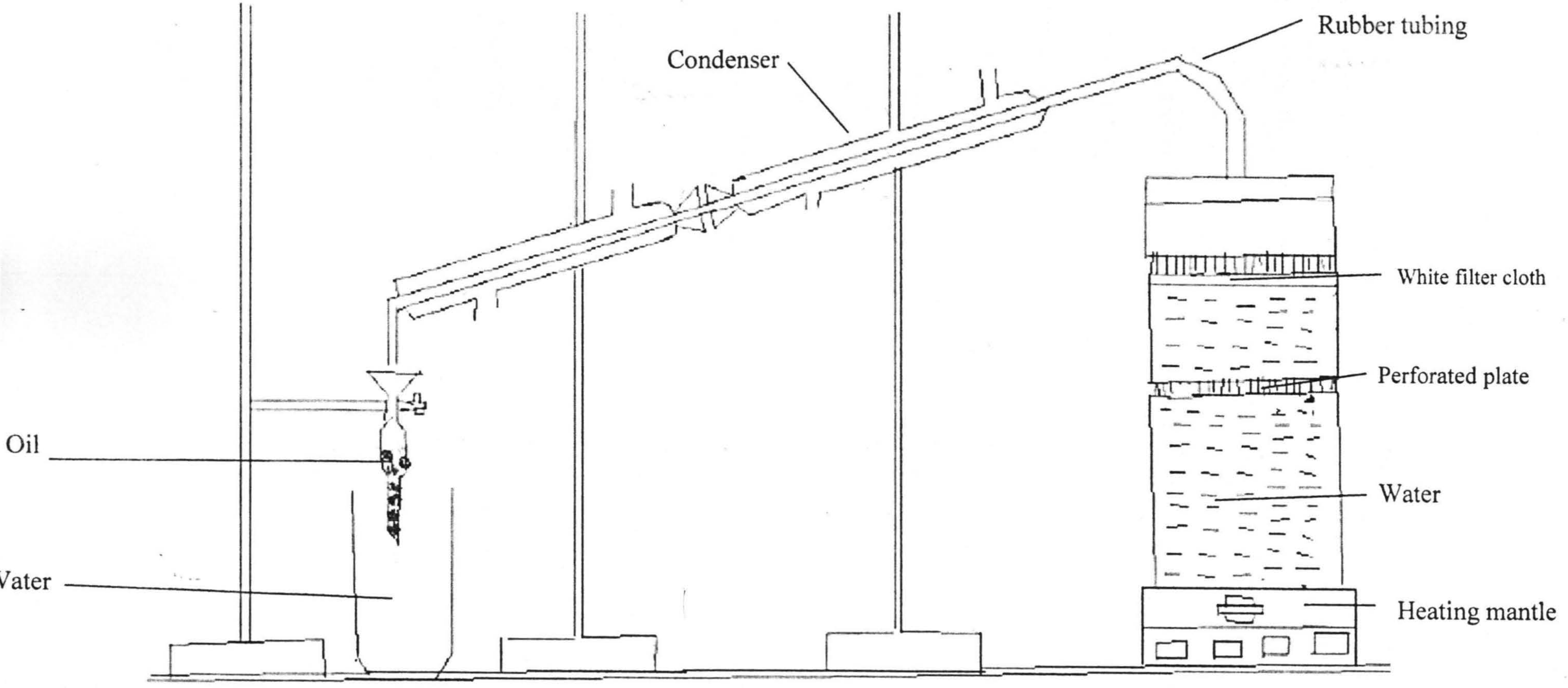


Figure 3.1a Sketch of the laboratory set-up of the Steam Distillation Apparatus/Equipment

3.2.2 Indirect (Soxhlet) extraction of oil from AVOP

In this process, 10 gram of AVOP each of particle sizes 0.75 mm, 1.00 mm, 1.59 mm and 2.00 mm and two solvents namely n-hexane and anhydrous ethanol were used alternatively in the indirect extraction (leaching) using the soxhlet extractor. 10 gram of AVOP, 0.75 mm particle size, was placed inside a thimble and inserted into the inner tube of the soxhlet extractor. This apparatus was then fitted to a round bottom flask, which contained 200 ml of the solvent (n-hexane or anhydrous ethanol). A reflux condenser was also mounted and fitted on the apparatus. The set-up was held tight with a retort stand and then placed on a heating mantle that was switched-on for extraction times of 30, 60, 90, 120, 150 and 180 min at the boiling point of the solvent (n-hexane, 60^0C or anhydrous ethanol, 78^0C).The vapour passed up through the tube, condensed by the condenser and the condensed solvent falls into the thimble and slowly fills the body of the soxhlet. When the solvent reached the top of the tube, it siphoned over into the flask and thus removed the portion of the sample that has been extracted in the thimble. The process repeated itself automatically for the extraction times and the apparatus was dismantled. The mass of AVOP after extraction and drying in an electric oven was weighed and the weight recorded.

These procedures were repeated for particle sizes 1.00 mm, 1.59 mm and 2.00 mm using fresh samples of the same mass (10 gram) and the same solvent (n-hexane or anhydrous ethanol) at the boiling point of the solvent, (60^0C for n-hexane, or 78^0C for anhydrous ethanol) for each samples at extraction times of 30, 60, 90, 120, 150 and 180 mins. respectively for each particle size and solvent.

The solvent recovery process involved using the same soxhlet extractor. The mixture of solvent and oil (also called miscella) was heated in the flask. On constant heating, the solvent evaporated and thereafter condensed in the thimble chamber. The solvent was collected before it siphoned back into the flask. The extracted oil was then recovered and the mass recorded.

The set-up of the soxhlet extraction apparatus is shown in Figure 3.2 while the sketch of the process is shown in Figure 3.2a.

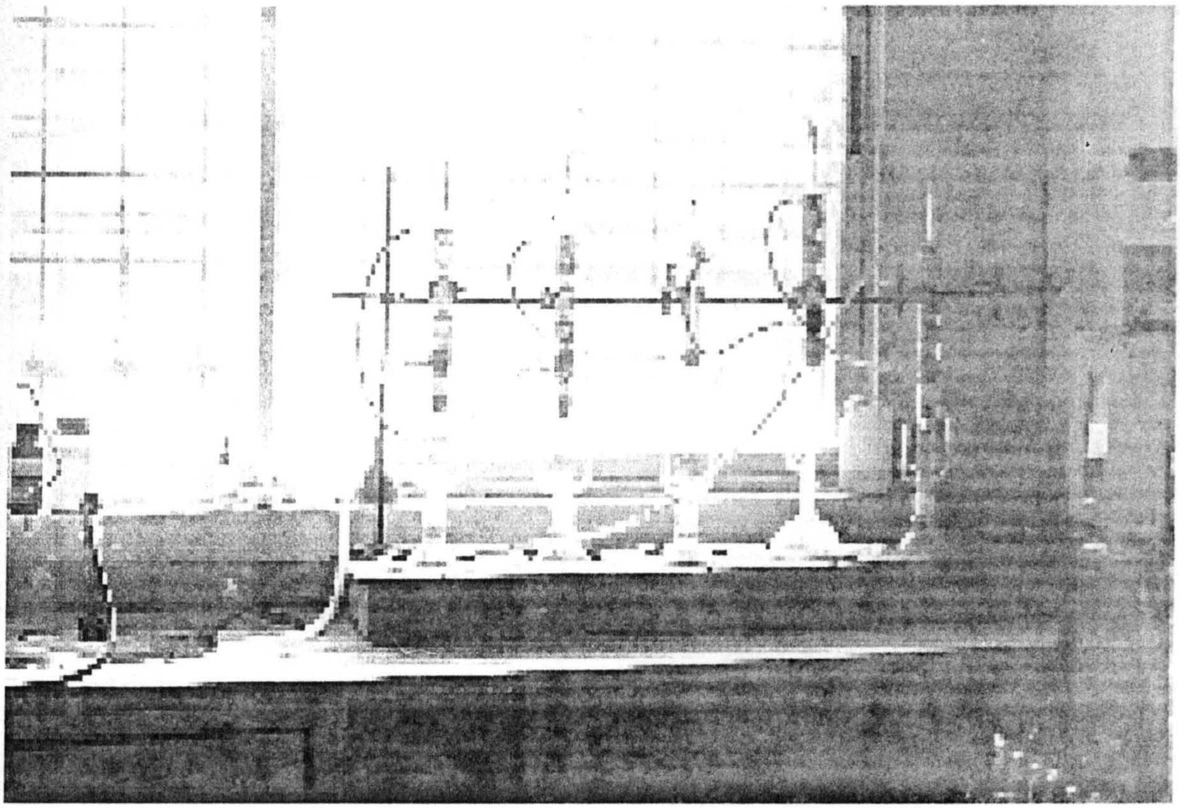


Figure 3.2: Laboratory Set-Up of the Indirect Leaching Process (Soxhlet Extraction Apparatus)

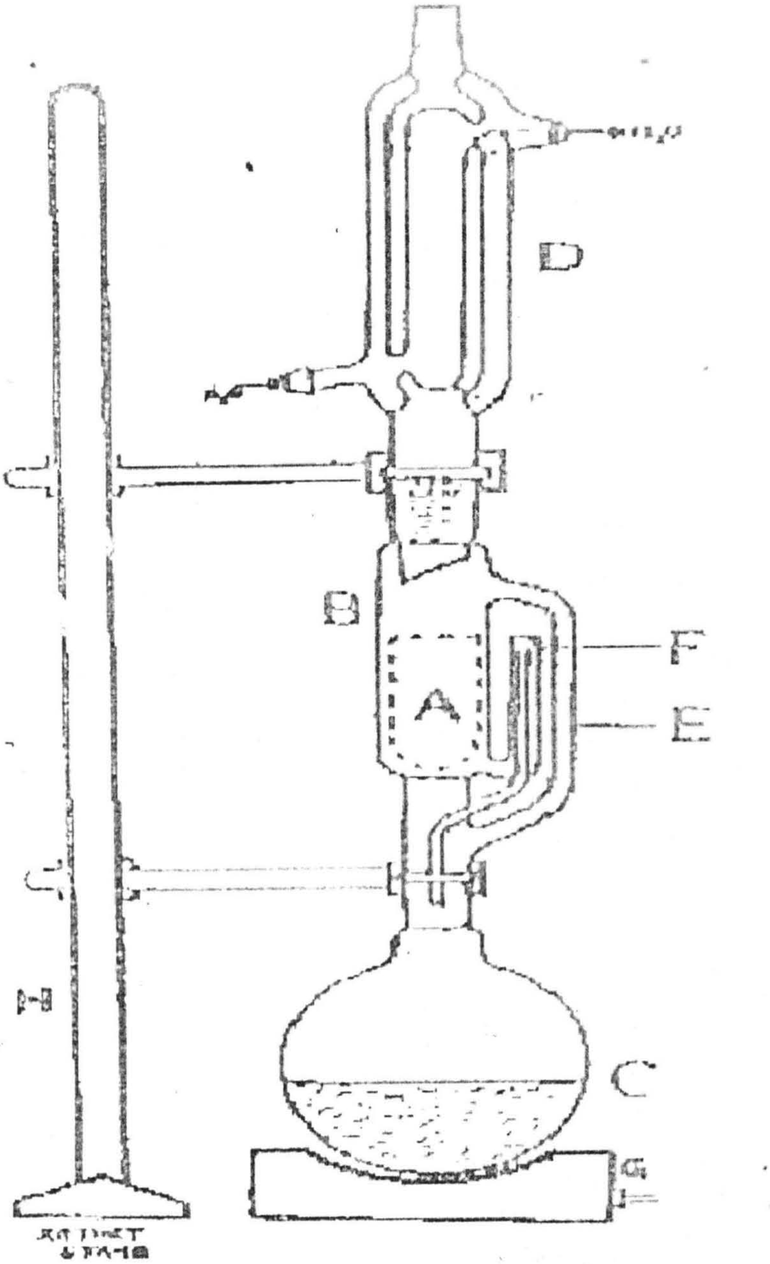


Figure 3.2a Indirect Leaching (Soxhlet) Experimental Set-up

A	Thimble
B	Soxhlet unit
C	Round bottom flask
D	Reflux Condenser
E	Vapour duct
F	Siphon tube
E	Electric heater

3.2.3 Direct extraction of oil from AVOP

In this process, 10 gram of AVOP each of particle size 0.75 mm, 1.00 mm 1.59 mm and 2.00 mm and two solvents namely n-hexane and anhydrous ethanol were used alternatively in the direct extraction (leaching) of oil from AVOP. 10 gram of AVOP; 0.75 mm particle size, was measured into a round bottom flask which contained 200 ml of the solvent (n-hexane or anhydrous ethanol). The mixture was vigorously agitated by swirling the flask. A reflux condenser was mounted and fitted onto the conical flask. The condenser was then connected to a tap water source. The vent of the flask was made air-tight to prevent the escape of the evaporating solvent. The set-up was held tight with a retort stand and the mixture placed on an electric heater and the thermostat adjusted to maintain a constant heating rate at the boiling point of the solvent (n-hexane, 60^0C and anhydrous ethanol, 78^0C). The mixture was allowed to boil for the extraction times of 30, 60, 90, 120, 150 and 180 mins. respectively. The vapour from the boiling solvent was made to condense and return to the mixture by means of a reflux condenser which was mounted on the flask through which water was constantly flowing. After the extraction had been completed, the heater was switched-off and the solvent decanted and filtered. The raffinate was weighed and its weight recorded after drying in an electric oven.

These procedures were repeated for particles sizes 1.00 mm, 1.59 mm and 2.00 mm using fresh sample of the same mass (10 gram) and the same solvent (n-hexane or anhydrous ethanol) at the boiling point of the solvent (60^0C for n-hexane or 78^{0}C) for each samples at extraction times of 30, 60, 90, and 120, 150 and 180 mins. respectively for each particle size and solvent.

Solvent recovery process was further carried-out on the extract phase to obtain the oil extract (active principle). This involved the use of the soxhlet extractor. On constant heating, the solvent evaporated and thereafter condensed in the thimble chamber. The solvent was collected before it siphoned back into the flask. The extracted oil was then recovered and its mass recorded. The laboratory set-up of the direct extraction apparatus is shown in Figure 3.3 while the sketch of the process is given in Figure 3.3a.

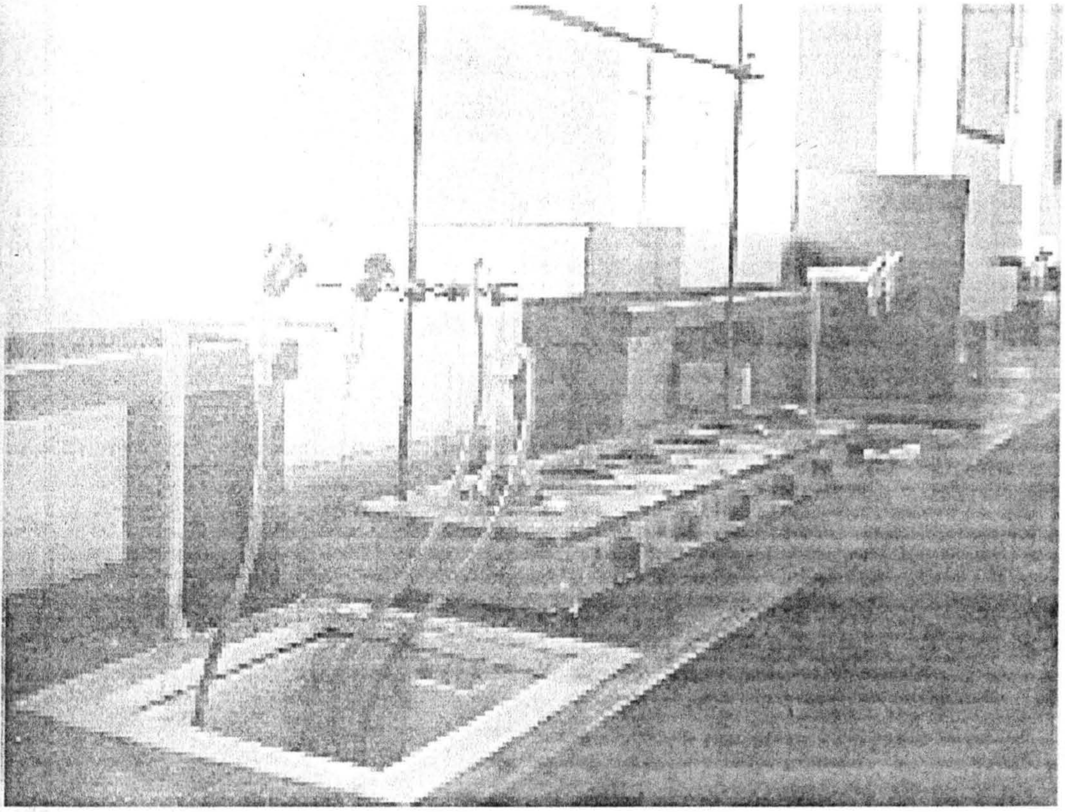


Figure 3.3: Laboratory Set-Up of the Direct Leaching Process Apparatus/Equipment

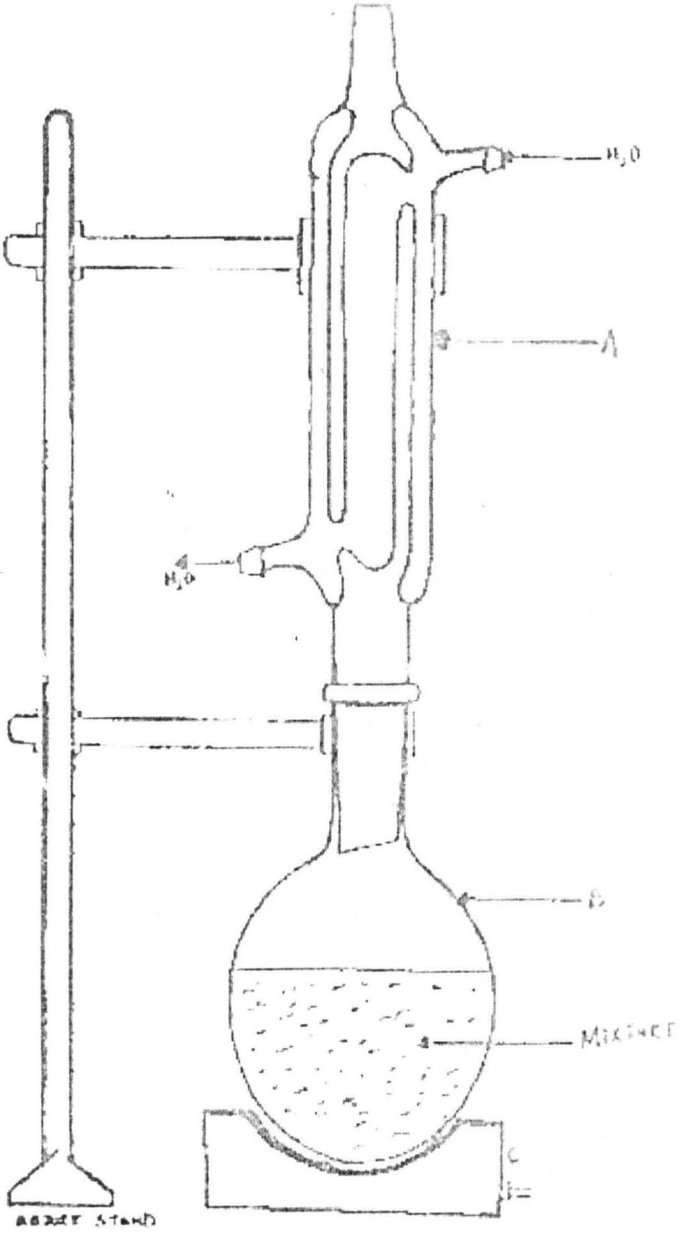


Figure 3.3a Direct Leaching Experimental Set-up

A	Reflux condenser
B	Round bottom flask
C	Electric heater

Subsequently, the crude AVOP oil was refined with activated clay (bleaching earth) locally sourced from chemical Engineering laboratory, FUT, Minna which was previously sieved to 70-5 microns. 0.1% by weight of the clay was added to 10 gram of the oil sample. The mixture was heated to a constant temperature of 100^0C with stirring for 30 min. The oil was then filtered at the same temperature and the filtrate characterized, in order to determine its physiochemical properties and hence its suitability for industrial applications.

3.3 Characterization of the Crude and Refined Oils

The crude and refined oil samples were subjected to various physicochemical analysis (ISO/TR 21092, IS0 212, IS0/TR 210, AOCS) to determine its properties such as saponification value, iodine value, peroxide value, free fatty acid value, acid value, specific gravity, refractive index and viscosity.

3.3.1 Determination of free fatty acid

Twenty five (25) milliliters of diethylether was mixed with 25 ml of ethanol in a beaker containing 20 ml of the crude or refined oil sample. Phenolphthalein was added as the indicator. The mixture was titrated against 0.1 mole of sodium hydroxide with continuous agitation of vessel until an emerald green colour was observed and the result recorded.

3.3.2 Determination of peroxide value

Five (5) milliliters of the crude or refined oils samples was mixed with 10 ml of chloroform and stirred immediately. 1,5 ml of acetic acid was added followed by the addition of 1.0 ml of freshly prepared potassium hydroxide. 75 ml of water was added to the mixture and was vigorously shaken using the hand. Few drops of starch solution were discharged into the mixture as indicator. The resulting solution was titrated with 0.01 mole of sodium thiosulphate solution and the value was recorded.

3.3.3 Determination of acid value

Twenty five (25) milliliters of diethylether was mixed with 25 ml of ethanol using phenolphthalein as indicator. The solution was carefully neutralized with 0.1 mole sodium hydroxide or carbon tetrachloride. 10 ml of the crude or refined oil was then added to neutralize the solvent and then titrated with 0.1 ml of aqueous solution of sodium hydroxide.

3.3.4 Determination of saponification value

Ten (10) grams of the crude or refined oil was put into a conical flask and 2.5 cm^3 of ethanol potassium hydroxide was added. The flask was connected to a reflux condenser and heated for 30 min. in boiling water under constant agitation. Similarly, 2.5 cm^3 of ethanol and 0.5 mole potassium hydroxide in ethanol was treated together. 1.0 ml of phenolphthalein was then added to the mixture as indicator. After cooling, each flask was titrated against 0.5 mole hydrochloric acid to an end point. The second flask served as the blank solution that was without the oil.

3.3.5 Determination of iodine value

Dam's reagent was prepared using 25 ml pyridine, 0.25 mole hydrogen sulphate, 1.5 ml glacial acetic acid and 10 ml of bromine. About 0.3 gram or 5 ml of the crude or refined oil sample was mixed with 20 ml of chloroform and 25 ml of Dam's reagent. The mixture was subjected to vigorous mixing using a stirring rod and left in the dark for 90 min. after which 20 ml of potassium iodide and 150 ml of water were added until the mixture was homogenous. Few drops of prepared starch was added as indicator. The mixture was titrated with 0.1 mole of sodium thiosulphate solution.

3.3.6 Determination of specific gravity

A clean dry 50 cm^3 specific gravity bottle was accurately weighed and the weight recorded. The bottle was then filled to the mark with crude or refined oil samples and reweighed. The difference between these weights gave the mass of the oil sample and this was recorded. The oil sample was then removed and the bottle cleaned, dried and weighed again.

3.3.7 Determination of Refractive Index

The instrument for the determination of refractive index is the refractometer. The surfaces of the prisms was cleaned with ethanol and allowed to dry before use due to the fact that index of refraction is sensitive to even the smallest amount of contaminant. The crude or refined oil samples was placed on the lower prism and ensured that it covered the entire width of the prism plate using a dropper. The upper prism was brought into contact with the lower prism so that the oil sample formed an unbroken layer between the two prisms. The controls were manipulated to bring the light and dark fields into focus with the cross hairs. The readings were then made at a temperature of about 25°C.

3.3.8 Determination of viscosity

The measurement of viscosity of the crude or refined oil sample was carried out using viscometer bath with an inserted U – tube capillary. 15 ml of the oil sample was poured into the U – tube capillary and corked. The U - tube was suspended into the viscometer bath containing water maintained at a temperature of 30°C. The cork was removed and the oil sample begun to flow through the capillary. The time taken for the oil to flow through the capillary of the fixed length was recorded and used for the calculation of the viscosity of the oils samples. This was recorded.

3.4 Experimental Design: Factorial Explanation and Statistical Analysis

3.4.1 Factional explanation

This experiment was designed to take into consideration the factors which are believed to influence the rate of extraction process. From the literature survey, physical characteristics of solids i.e particle size, weight of sample, solvent selection, temperature of extraction, degree of agitation, production method and time of extraction are the basic factors that affect the extraction rate. Therefore, in the course of this research work, weight of sample, temperature and degree of agitation were fixed whereas other factors such as particle size, extraction time and production method was varied as shown in Table 3.3.

Table 3.3; Systematic Variation of Key Factors in the Experiment

Production method X_3	Particle size (mm) x_2	Extraction time (min) x_1	Mass of AVOP	Temp.	Agitation
Steam distribution	0.75	30	10	-	-
	1.00	30	-	-	-
	1.59	30	-	-	-
	2.00	30	-	-	-
Direct leaching	0.75	30	-	-	-
	1.00	30	-	-	-
	1.59	30	-	-	-
	2.00	30	-	-	-
Indirect leaching	0.75	30	-	-	-
	1.00	30	-	-	-
	1.59	30	-	-	-
	2.00	30	-	-	-
Steam distillation	0.75	60	-	-	-
	1.00	60	-	-	-
	1.59	60	-	-	-
	2.00	60	-	-	-
Direct leaching	0.75	60	-	-	-
	1.00	60	-	-	-
	1.59	60	-	-	-
	2.00	60	-	-	-
Indirect leaching	0.75	60	-	-	-
	1.00	60	-	-	-
	1.59	60	-	-	-
	2.00	60	-	-	-
Steam distillation	" 2.00	" 180	-	-	-

3.4.2 Statistical analysis used

The statistical functions such as mean, standard deviation, variance etc were analyzed so as to derive maximum information from the given set of data (analysis) and conversely minimizing the amount of the data (experimental design) to derive specific information. The concept utilized included standard deviation, true value, degrees of freedom, normal frequency distribution and theoretical model distribution variabilities.

3.5 Design of an Improvised Pilot Plant for the Production of Oil from Avocado Apple (*Persea americana*)

3.5.1: Process design

The process consisted of a packed bed steam extraction vessel (still or distillation apparatus) equipped with two condensers, a settling tank and/or separating funnel to collect and separate the mixture of water and oil. The steam extraction vessel was divided into two compartments; the bottom compartment was for water used to generate steam on heating to percolate the sample (AVOP); the top compartment for the actual percolation and diffusion of steam through the sample with the consequent movement of steam-oil vapour to the condenser. The two compartments were separated by a perforated metal plate made of the same material as the still and finally, a covering that provided an air-tight arrangement.

3.5.2: Equipment design

The equipment for the process consisted of the still (distillation apparatus), condenser and separator. The steam required for the process was obtained from water heated directly in the bottom compartment of the still. The schematic diagram for the production of oil from AVOP is shown in Figure 3.4

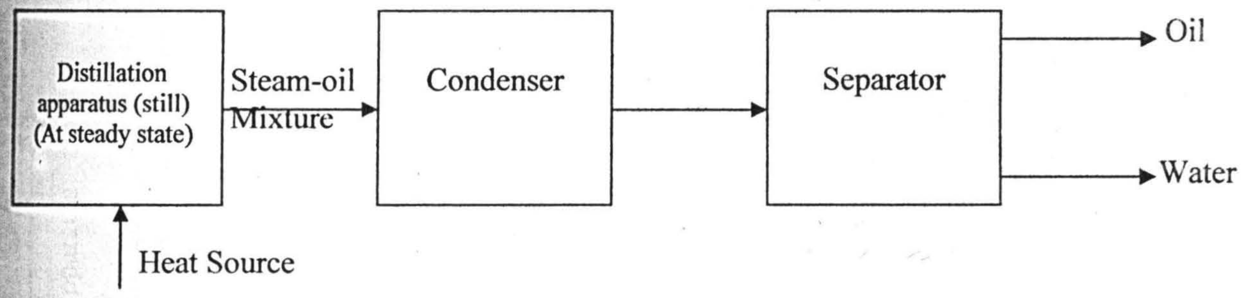


Figure 3.4: Schematic Diagram for the Production of Oil from AVOP

Similarly, the process flow sheet for the unit operation is shown in Figure 3.5.

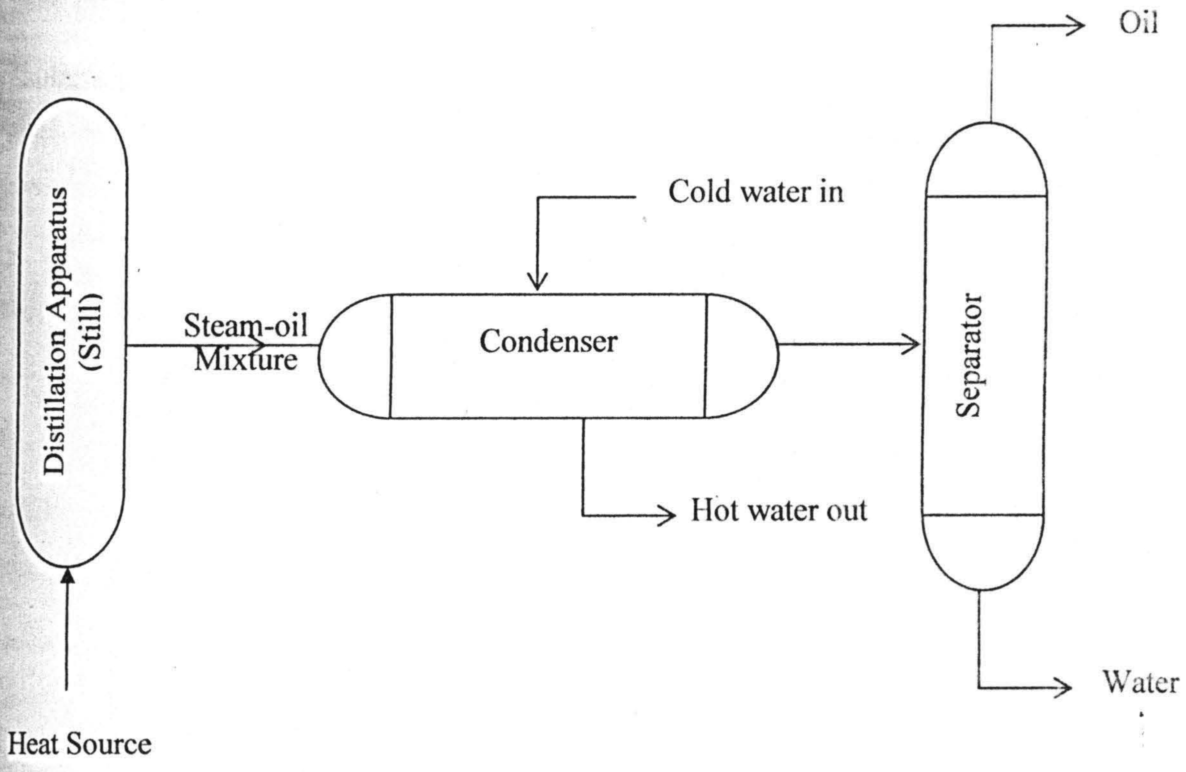


Figure 3.5: Process Flowsheet for the Production of Oil from AVOP

Design condition

In this design, most parameters required were experimentally determined. Comprehensive experiments on the determination of the technological parameters and crude AVOP obtained were performed in laboratory condition. The results are summarized as follows:

Table 3.3: **Summary of Parameters Required for the Design**

Operating conditions	**Values**
Optimum steam supply rate per gram sample	($2.654 \times 10^{-7} m^3/s$)
Density of steam, ρ	995 kg/m^3
Viscosity of water, μ_W	8.0×10^{-4} Ns/m^2
Cold water inlet temperature	25°C
Hot water outlet temperature	50°C
Conductivity coefficient of stainless steel	4.608W/mK
Condensate temperature	45°C
Density of oil, ρ	900.6 kg/m^3
Density of steam-oil mixture, ρ	900.76 kg/m^3
Viscosity of steam-oil mixture, μ	$6.98 \times 10^{-4} Ns/m^2$
Heat capacity of mixture, C	4,100 kJ/kg°C
Thermal conductivity of mixture, k_f	0.87 W/m°C
Residence time after first water-oil mixture drops	45-55min
Steam temperature	100°C
Density of water	$1000 kg/m^3$
Operating pressure	1 atmosphere
Density of sample	$440. kg/m^3$
Productivity	9.574 m^3/hr

3.6 Scale – Up Method: Chemical Similitude

The basis of this method are several dimensionless groups derived from the application of the laws of conservation of mass, momentum and energy to the chemical reaction system. Basically, the rate of a given chemical process is independent of the size and structure of the reactor though the chemical reaction rate is influence by physical processes such as mass and heat transfer which are usually controlled by the size and structure of the reactor.

The following assumption is made to simplify the principle upon which chemical similitude is used.

i. the system under consideration is solid-liquid first order reaction.

ii. the analysis of the laws of conservation is based on a unit volume of a circular cylindrical reactor of radius R and length L

iii. Steady state condition prevails which implies that no accumulation of mass, momentum and energy with respect to time.

Now, let the model reactor of radius R be compared with a prototype reactor of radius R^1 such that

$$R^1 = \lambda R \quad \text{......................3.1}$$

Where λ is the scale factor

Introducing the above assumption into the partial differential equation describing the conservation of mass, momentum and energy given as

$$\Phi = -y \frac{2\,\Phi}{2x} \quad \text{................3.2}$$

Where Φ = flux of property being transferred

Y = diffusivity (thermal, mass or momentum)

$\varnothing$ = concentration of property being transferred

Seven dimensionless groups are derived. The values of these groups for a model reactor and a prototype reactor must be the same to satisfy the similitude conditions.

Now , from the reactor still developed, the following nomenclature holds: C is concentration in moles/cu.ft, L is reactor length in feet, u is velocity in ft/sec, r is the rate of reaction in moles/cu.ft(sec), R is the reactor radius in ft, D is diffusivity in sq.ff/sec, Δv is volume change per unit disappearance of the reactant for which r is written in cu.ff/mole, ρ is the density in lb/cu.ft, μ is the viscosity in lb/(ft)(sec), Cp is the heat capacity in Btu/(lb)(^{o}f), K is the thermal conductivity in Btu/(sec) (sq.ft) (^{o}f/ft), ΔT is the temperature in excess of the wall temperature in ^{o}F, Q is the heat generated by the system in Btu/mole reacted, D_p is the particle diameter in feet, S is the space velocity with units of sec^{-1}, 'a' is the wall surface area per unit of reactor volume in ft^{-1}, σ is a proportionality constant of 0.484 Btu/sec) (sq.ft) ^{o}f)^4, ξ is the emissivity or absorptivity of the reactor wall and is dimensionless.

The dimensionless groups derived for this heterogeneous system includes

$$\text{i.}\quad \frac{r}{Sc} \qquad \text{ii.}\quad \frac{rDp^2}{DC} \qquad \text{Iii.}\quad \frac{S}{r\,\Delta v} \qquad \text{Iv}\quad \frac{DpSL_e}{\mu} \qquad \text{..............3.3}$$

$$\text{v.}\quad \frac{C_pS_eR^2}{k + 4/3\ Dp^6T^3} \qquad \text{vi.}\quad \frac{QrR^2}{(K + 4/3\ Dp^6T^3)\ \Delta T} \qquad \text{vii.}\quad \frac{Qr}{Cper\ \Delta v\ \Delta T}$$

From the expression that model reactor of radius R is to be compared with a prototype reactor of radius R^1, such that

$R^1 = \lambda R$ 3.4

Where λ is the scale factor. Further assuming that all the physical properties such as Cp, ρ, μ, D, etc can be maintained the same in both reactor. Then, by introducing equation 3.4 into the dimensionless groups for the heterogeneous reactions and remembering that the values of these groups must be the same for reactors of both scales, the following relations are obtained. The set of relations applicable in this case since heat transfer is mainly by conduction. Thus:

$R^1 = \lambda R$ 3.5

$L^1 = \lambda L$ 3.6

$D_p{}^1 = \lambda D_p$ 3.7

$S^1 = \lambda^{-2} S$ 3.8

$r^1 = \lambda^{-2} r$ 3.9

$\Delta T^1 = \lambda \Delta T$ 3.10

Also, the temperatures for both scales must be the same, equation 3.5 through 3.7 reveal that in comparison with the vessel diameter, the length of the prototype as well as the size of the particle in it should be of smaller proportion than those for the model.

The analysis of the chemical similitude carried out above implied that the overall rate is controlled by the physical rate processes such as mass transfer, heat transfer, etc and thus, the significance of chemical similitude of dimensionless groups containing reaction rate parameters diminishes, and dynamic and/or thermodynamic similitude of groups containing force and/or thermal properties played the key role.

Of course, complete dimensional similitude is nearly impossible hence, the net result of chemical similitude is to automatically fix the reactor geometry and other physical parameter without complete satisfaction of other aspects of dimensional similitude.

3.7: Material Balance Calculations

Based on the targeted process production rate, the material balance was performed starting from the separator unit. For steady state operation,

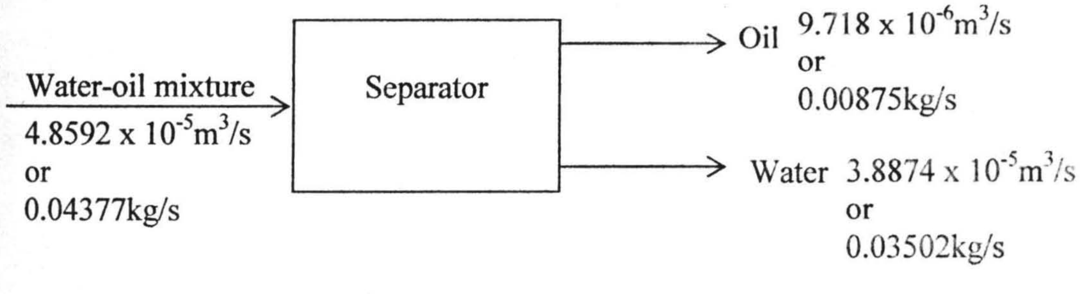
INPUT = OUTPUT

Basis: 9.574m^3/hr of AVOP oil

Unit: Separator

The material balance over the separator unit is presented as follows.

Average volumetric flow rate for the moderate steam heating rate into the separator unit of the water-oil mixture was 174.93 ml/hr (4.8592 x $10^{-5}m^3/s$).



The average composition of the water-oil mixture in the separator unit was 20% oil and 80% water.

The volumetric flow rate of the water-oil mixture was $4.8592 \times 10^{-5} m^3/s$.

The mass flow rate of the water-oil mixture was obtained as follows:

Mass flow rate = Volumetric flow rate x density of mixture

$$\begin{array}{c|c} 4.8592 \times 10^{-5} m^3 & 900.76 kg \\ \hline s & m^3 \end{array}$$

$= 0.04377 kg/s$

Therefore mass flow rate of the water-oil mixture =0.04377kg/s.

From the average value obtained, the flow rate of the oil is computed as follows:

Oil flow rate $= 20\%$ of $4.8592 \times 10^{-5} = 9.718 \times 10^{-6} m^3/s$

or $= 20\%$ of $0.04377 = 0.00875$ kg/s

Hence, flow rate of water is given as:

Water flow rate $= 4.8592 \times 10^{-5} - 9.718 \times 10^{-6}$

$= 3.8874 \times 10^{-5} m^3/s$

or $= 0.04377 - 0.008754$

$= 0.03502$ kg/s

Hence, flow rates of oil and water out of the separator unit was $9.718 \times 10^{-6} m^3/s$ (0.00875 kg/s) and $3.887 \times 10^{-5} m^3/s$ (0.003502 kg/s) respectively.

Table 3.4: Summary of Material Balance Calculations on Separator Unit

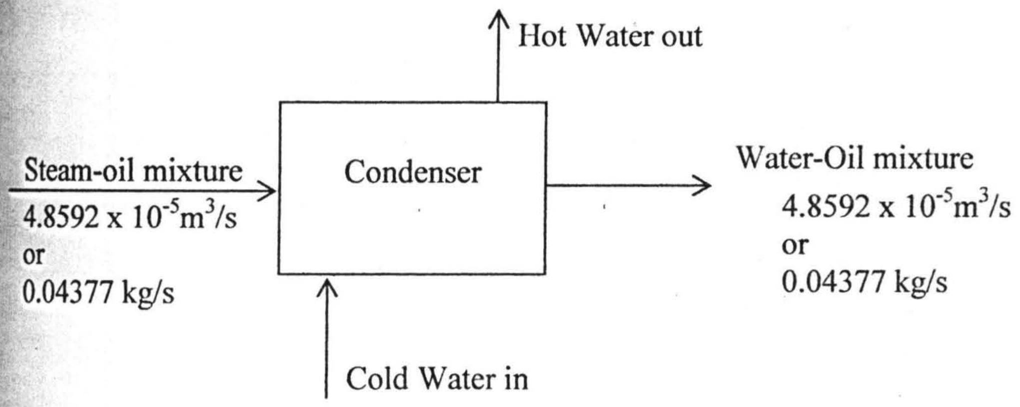
	INPUT		OUTPUT		
Components	**Volumetric flow rate (m^3/s)**	**Mass flow rate (kg/s)**	**Volumetric flow rate (m^3s)**	**Mass flow rate (kg/s)**	**% composition**
Water-oil mixture	4.8592×10^{-5}	0.04377	0.00	0.00	0.00
Oil	0.00	0.00	9.718×10^{-6}	0.00875	20.00
Water	0.00	0.00	3.8874×10^{-5}	0.03502	80.00
Total	4.8592×10^{-5}	0.04377	4.8592×10^{-5}	0.04377	100

Unit: Condenser

In the condenser unit, the steam-oil mixture from the still was liquefied using cold water sourced from a running tap.

In the condenser unit, since steady state condition prevailed,

INPUT = OUTPUT



The volumetric and mass flow rates of the water (hot and cold streams) would be obtained during the condenser design calculations which would be presented in the course of the design.

Table 3.5: Summary of Material Balance Calculations on Condenser Unit

	INPUT		OUTPUT	
Components	**Volumetric flow rate (m^3/s)**	**Mass flow rate (kg/s)**	**Volumetric flow rate (m^3s)**	**Mass flow rate (kg/s)**
Steam-oil mixture	$4.8592x10^{-5}$	0.04377	$4.8592x10^{-5}$	0.04377
Total	$4.8592x10^{-5}$	0.04377	$4.8592x10^{-5}$	0.04377

Unit: Distillation Apparatus (Still)

Based on the present experiment, the process in the packed bed still consisted of two stages.

The first involved the induction period in which the sample was heated to the steam temperature. This stage represented the unsteady state condition.

The second stage was the period the oil was deposited on the sample surface and was transferred by the moving steam to the condenser. This represented the steady state.

From the experimental results obtained, the maximum oil output due to moderate heating that could be removed from the sample per gram was 4.787 x $10^{-3}m^3/g$ (59.8%) per hour. This was expected to be at a production rate of 9.574 m^3/hr per batch.

Basis: $9.574m^3/hr$ per batch

Calculation of the quantity of sample required per batch of production:

Mass of sample required to produce $9.574m^3$ oil is as follows:

$$X = \begin{array}{c|c|c} 9.574m^3 & 1.0g & \\ \hline & & 4.787\times10^{-3}m^3 \end{array}$$

$\therefore x = 2000g$ sample = 2kg sample

Thus, mass of sample required to produce $9.574m^3$ oil is 2000g (2kg).

Quantity of steam required = Mass flow rate of steam x induction time

From the experiment conducted, the maximum induction period for the moderate or medium heating rate lied between 12 – 18 min.. However, 15 min. was considered for this design work.

Hence,

$$\text{Quantity of steam required} = \begin{array}{c|c} 0.03502\text{kg} & (15 \times 60)\text{ s} \\ \hline \text{s} & \end{array}$$

$$= 31.52 \text{ kg}$$

Theoretically, the condensate flow rate was the same as that of steam. Thus, allowing for film condensate on the sample surface of 10%, the condensate flow rate is obtained as follows;

$$\text{Condensate flow rate} = 0.03502 \frac{\text{kg}}{\text{s}} - (10\% \text{ of } 0.03502 \frac{\text{kg}}{\text{s}})$$

$$= (0.03502 - 0.003502)$$

$$= 0.03182 \text{ kg/s}$$

Hence, the condensate flow rate was taken to be 0.03182 kg/s

3.7.1: Material balance for the unsteady state condition

For a loss of 0.1% in the sample due to unsteady state process, then

$$\text{Used sample} = \text{sample mass} - 0.1\% \text{ of sample mass}$$

$$= 2.0\text{kg} - (0.1\% \text{ of } 2.0\text{kg})$$

$$= (2.0 - 0.002)\text{kg}$$

$$= 1.998 \text{ kg}$$

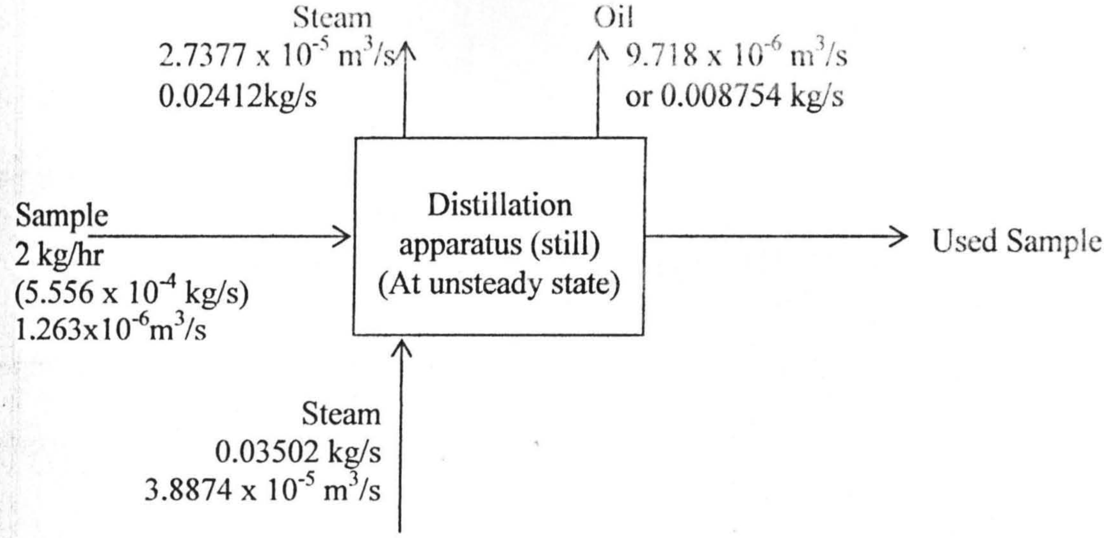
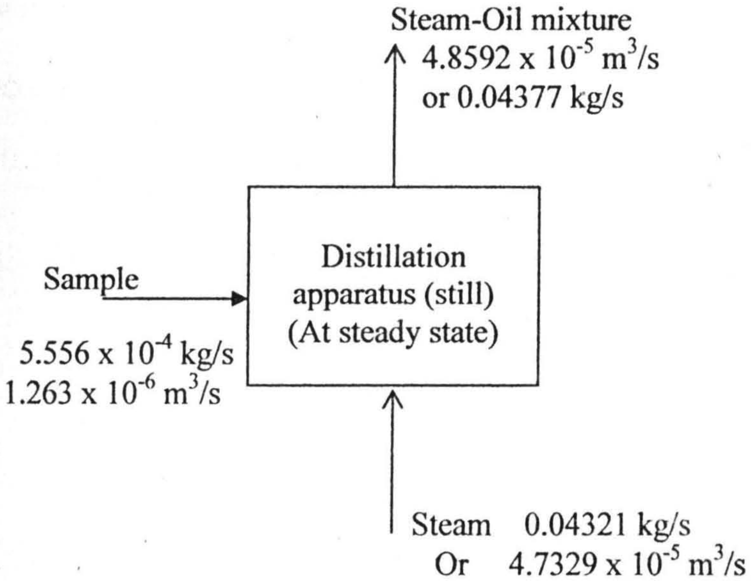

Steam
2.7377 x 10⁻⁵ m³/s
0.02412kg/s
Oil
9.718 x 10⁻⁶ m³/s
or 0.008754 kg/s
Sample
2 kg/hr
(5.556 x 10⁻⁴ kg/s)
1.263x10⁻⁶m³/s
Distillation
apparatus (still)
(At unsteady state)
Used Sample
Steam
0.03502 kg/s
3.8874 x 10⁻⁵ m³/s

Table 3.6: Summary of Unsteady State Material Balance Calculations on the Still

Components	INPUT		OUTPUT	
	Volumetric flow rate (m^3/s)	Mass flow rate (kg/s)	Volumetric flow rate (m^3/s)	Mass flow rate (kg/s)
Steam	3.8874×10^{-5}	0.03502	2.7377×10^{-5}	0.02412
Sample	1.263×10^{-6}	5.556×10^{-4}	0.00	0.00
Oil	0.00	0.00	9.718×10^{-6}	0.008754
Total	4.0137×10^{-5}	0.03558	3.7095×10^{-5}	0.03287

3.7.2: Material balance for the steady state condition

At steady-state, steam-oil mixture was formed with the out-going steam from 1000 ml of water in the still chamber.



Volume of the packed bed on the perforated metal sheet was obtained as follows:

Mass of sample used in the experiment, m = 10 g = 0.01 kg

Density of sample = 440 kg/m^3

:- Volume of packed bed;

$$V = \frac{mass}{density} = \frac{0.01}{440} = 2.273 \times 10^{-5} m^3$$

For the cylindrical shaped vessel

$V = \frac{\pi}{4} D^2 H$ (Volume of a cylinder)

:- V = 0.785 D^{2}H

For the design,

Height, H = 0.464 m and,

Diameter D = 0.231 m

:- $V = 0.785\ D^2H$

$= 0.785\ (0.231)2\ (0.464)$

: $V = 0.1944\ m^3$

Hence, volume of cylindrical shaped still

$V = 0.1944\ m^3$

Table 3.7: Summary of Steady State Material Balance Calculations on the Still

	INPUT			OUTPUT
Components	**Volumetric flow rate m^3/s**	**Mass flow rate (kg/s)**	**Volumetric flow rate m^3/s**	**Mass flow rate kg/s**
Steam	4.7329×10^{-5}	0.04321	0.00	0.00
Sample	1.263×10^{-6}	5.556×10^{-4}	0.00	0.00
Steam-Oil mixture	0.00	0.00	4.859×10^{-5}	0.04377
Total	4.8592×10^{-5}	0.04377	4.8592×10^{-5}	0.04377

3.8: Energy Balance Calculations

At unsteady state, the sample packed bed was likened to a plug flow reactor system in which the steam at 100°C flowed into and lost some of the heat to the sample bed. The heat lost by the steam continued until a steady state was reached at which the temperature of the bed was equal to that of the steam. At that point, equilibrium was said to exist.

The condensation of steam on the sample surface was a complicated heat transfer process that involved three fluid flowing phases. These were; the vapour phase, the condensate phase and the oil phase.

At a steady steam supply rate in a reactor of height, H, the induction period was dependent on the steam-sample ratio or packing density.

The steam lost its heat content and condensed on the sample surface. There was no temperature drop.

Thus, the heat supplied to the system, given as, $Q_{\sup plied}$, was

$$Q_{\sup plied} = Q_{in} = M_{steam} L$$

Where M_{steam} = mass flow rate of steam = 0.03502 kg/s

L = Latent heat of vaporization of water

$$= 22{,}600\, kJ/kg$$

Therefore,

$$Q_{\sup plied} = Q_{in} = \begin{array}{r|r|} \text{0.03502kg} & \text{22,600KJ} \\ \hline \text{s} & \text{kg} \end{array}$$

$$= 791.452\, kJ/s$$

Assuming a steam transfer efficiency of 90%, additional heat of 79.1452 kJ/s was required from the heating source to the still.

3.8.1: Heat received by the still and the contents

As the steam condensed, it lost its heat content and was removed from the system at the boiling point of water (100°C).

Heat absorbed by the system,

$Q_{received} = \text{Heat Content}$ of the oil + heat content of used samples + heat content of condensate removed from the reactor with marginal fall in temperature + heat content of condensate on sample surface + heat of vaporization of condensate and construction material.

This is mathematically expressed as follows:

$$Q_{received} = Q_{oil} + Q_{ss} + Q_{c} + Q_{css} + Q_{cm}$$

Thus:

i. Heat content of the oil or heat acquired by oil is given as follows

$$Q_{oil} = M_{oil}.C.\Delta T_{oil}$$

Where M_{oil} = mass flow rate of oil = 0.008754 kg/s

C = specific heat capacity of oil = 2.6 x 10^{-3} kJ/kg.k

ΔT_{oil} = Change in temperature = 100 – 25 = 75°C

$Q_{oil} =$

.008754kg	2.6x10^{-3}KJ	75^0C
s	Kg^{0}C	

$$= 1.707 \times 10^{-3}\ kJ/s$$

ii. Heat content of used sample or heat acquired by used sample as follows:

$$Q_{ss} = M_{ss}.C.\Delta T_{ss}$$

Where M_{ss} = mass flow rate of used sample = 1.998 kg/hr (5.55×10^{-4} kg/s)

C = specific heat capacity of used sample, which can be approximated to

half that of wood = 0.9414 k^3/kg.^{0}C

ΔT_{ss} = temperature change = 100 – 25 = 75°C

$$\therefore Q_{ss} = \begin{array}{c|c|c} 5.55\times10^{-4}\text{kg} & 0.9414\text{KJ} & 75^0\text{C} \\ \hline \text{s} & \text{Kg}^0\text{C} & \end{array}$$

$$= 3.919 \times 10^{-2}\ kJ/s$$

iii. Heat content of condensate removed from the reactor with marginal fall in temperature = Heat lost by out-going condensate (steam-oil mixture). This is as follows:

$$Q_c = M_c.C.\Delta T_c$$

Where M_c = mass flow rate of condensate = 0.03182 kg/s

C = Specific heat capacity of condensate = 4200 kJ/kg. °C

ΔT_c = Marginal temperature change = 100 – 96 = 4°C

$$Q_c = \begin{array}{c|c|c} 0.03182\text{kg} & 4200\text{KJ} & 4^0\text{C} \\ \hline \text{s} & \text{Kg}^0\text{C} & \end{array}$$

$$534.576\, kJ/s$$

iv. Heat content of condensate on sample surface = Heat acquired by condensate on sample surface to heat it up to 100°C. This is given by:

$$Q_{css} = M_{css}.C.\Delta T_{css}$$

Where M_{ess} = mass flow rate of condensation sample surface = 0.003502 kg/s

C = Specific heat capacity of water = 4200 kJ/kg.^{0}C

ΔT_{css} = Marginal temperature change = 4^{o}C

$$Q_{css} = \begin{array}{r|l|l} 0.003502\text{kg} & 4200\text{KJ} & 4^0\text{C} \\ \hline \text{s} & \text{Kg}^0\text{C} & \end{array}$$

$58.834\,kJ/s$

v. Heat of vaporization of condensate material and construction material = Heat acquired by condensate on sample surface to vaporize it.

$$Q_{css} = M_{cv}.L$$

Where M_{cv} = mass flow rate of condensate on sample = 0.003502 kg/s

L = Latent heat of vaporization of water = 22,600 kJ/kg

$$Q_{cv} = \begin{array}{r|l} 0.003502\text{kg} & 22{,}600\text{KJ} \\ \hline \text{s} & \text{Kg} \end{array}$$

= 79.145kJ/s

vi. Heat transfer loses via leakages from various connection

= 5% of total heat supplied.

= 5% of $791.452\,kJ/s$

$= 0.05 \times 791.452\,kJ/s$

$= 39.573\,kJ/s$

Hence, total heat absorbed by system was given as $Q_{received}$

$$Q_{received} = \left(1.707\times10^{-3} + 3.919\times10^{-2} + 534.576 + 58.835 + 79.145 + 39.573\right)kJ/s$$

$$= 712.169\, kJ/s$$

vii. Heat due to construction material is given by the expression:

$$Q_{cm} = KF\Delta T_{cm}$$

Where k = overall heat transfer coefficient. It is a function of the still construction material – Stainless steel.

F = surface area of the Still (reactor)

ΔT_{cm} = mean temperature difference

However, at equilibrium, and where steady state condition prevails,

$$Q_{\sup plied} = Q_{recieved} - KF\Delta T_{cm}$$

$$\therefore KF\Delta T_{cm} = Q_{\sup plied} - Q_{received}$$

$$= 791.452 - 712.169$$

$$= 79.283\, kJ/s$$

Therefore, $Q_{cm} = 79.28\, kJ/s$

Table 3.8: **Summary of Energy Balance Calculations**

Components	Energy Supplied				Energy Received				
	Latent heat (KJ/kg)	Mass flowrate (Kg/s)	Q supplied (KJ/s)	Loss	Mass flowrate (Kg/s)	Latent heat (KJ/kg)	Specific heat capacity (KJ/kg°C)	Temperature change ΔT (°C)	Q received (KJ/S)
Steam (water)	22,600	0.03502	791.542						
Oil	-	-	-		0.008754	-	$2.6x10^{-3}$	75	$1.707x10^{-3}$
Used samples Condensate	-	-	-		$5.55x10^{-4}$	-	0.9414	75	$3.919x10^{-2}$
Sample surface	-	-	-		0.03182	-	4200	4	534.576
Condensate	-	-	-		0.003502	-	4200	4	58.834
Condensate on sample					0.003502	22,600	-	-	79.145
Losses via leakage from connection	-	-	-	39.573					
Construction material component	-	-	-			0.00	0.00	0.00	79.283
TOTAL			791.452	39.573					751.88

3.9: Equipment Design

3.9.1: Design of the still (Distillation apparatus)

Conductive heat transfer, Q_{cm},

Q_{cm}, $= KF\Delta T_{LM}$ = 79.28 KJ/s

k = overall heat transfer coefficient which is a function of the still construction material; in this case, stainless steel and insulator (fiber with binder and baked)

$k = 1/(\delta/\lambda + 1/d_1)$

where δ – insulator thickness 20 mm, λ – heat transfer coefficient of insulator 5.5 kw/mk, α – heat transfer coefficient of stainless steel 0.2059 KW/MK

F = surface area of the still.

$K = 1/(0.005/5.5 + 1/275.9 \times 10^{-3}) = 0.275$ w/mk

But, Q_{cm}, $= KF\Delta T_{LM} = 79.28$ KJ/s

$$\text{Therefore} \quad \Delta_{Lm} = \frac{(100-25)-(50-25)}{\ln[(100-25)/(50-25)]} = 45.5^{o}C$$

$$\therefore F = \frac{Q_{cm}}{K\Delta T_{cm}}$$

$$\therefore F = \begin{array}{r|c|r} 79.28\text{x}10^{3}J & 1 \quad sm^{20}C & 1 \\ \hline s & 0.275J & 45^{0}C \end{array}$$

$$= 64.06 \text{ m}^2$$

Pressure-Drop across the packed bed

The pressure drop across the packed bed was dependent on the packing density (steam-sample ratio) of the sample. Pressure-drop data on the flow of fluids through beds of granular solids are not readily correlated because of the variety of granular materials and their packing density or arrangement.

For the flow of a single incompressible fluid through a bed of granular solids, the pressure-drop or other flow characteristics can be predicted from the correlation given by the formula (Perry and Green, 1984; Perry and Chilton, 1973):

$$\Delta P = \frac{2 f_m G^2 L (1-\in)^{3-n}}{D_p g_c \ell \phi_s^{3-n} \in^3}$$

Where ΔP – Pressure drop, P_a

L – Depth of packed bed, = 10mm = 0.1m

g_c – dimensionless constant

D_p – average particle diameter, defined as the diameter of a sphere of the same volume as the particle.

Average volume of AVOP = 8.00mm^3 = 8.00 x 10^{-9}m^3

Volume of sphere $= \frac{4\pi r^3}{3}$

Hence, the average particle diameter, dp, is:

$$8.00 \times 10^{-9} = \frac{4\pi r^3}{3} \Rightarrow r^3 = \frac{8.00 \times 10^{-9} \times 3}{4\pi} = 1.9099 \times 10^{-9}$$

r = 1.241 x 10^{-3}m and Dp = r x 2 = 1.241 x 2 = 2.482 x 10^{-3}m

Average particle diameter, $Dp = 2.482 \times 10^{-3} m = 0.00248m$

$\in$ – voidage (fractional free volume)

= volume of sample/volume of bed zone = (mass of sample/sample density)/volume of bed zone

$$\left(0.11 kg / 440.40 kg/m^3\right) / 2.73 \times 10^{-5} m^3 = 0.999$$

:- 1 - ε = 0.999 :- 1 - 0.999 = 0001

n – exponents, a function of the modified Reynolds number

$$N_{\mathrm{Re}}^{1}.N_{\mathrm{Re}} = \frac{DpG}{\mu}$$

$$= 0.002482\mathrm{m}^2 \cdot \frac{0.03502\mathrm{kg}}{\mathrm{s}} \cdot \frac{10^4}{8.0\mathrm{kgm}^2} = 0.1086$$

From the chart, n = 1 (Discharge coefficient C versus Reynolds number chart).

ϕ_s – Shape factor of the solid; defined as the quotient of the area of a sphere equivalent to the volume of the particle divided by the actual surface of the particle.

$$\phi_s = 8.00 \times 10^{-9}\mathrm{m}^3 \cdot \frac{1}{0.785\mathrm{m}} \cdot \frac{1}{1.935 \times 10^{-5}\mathrm{m}^2}$$

$$= 0.000527$$

G – fluid mass velocity based on chamber cross section = 0.03502kg/s

ℓ – fluid density = 995kg/m^3

f$_m$ – Frictional factor, a function of $N_{\mathrm{Re}}^{1} = 100/0.10861$

$$= 920.81$$

Hence, the pressure was computed as follows:

$$\therefore \Delta P = \frac{2}{0.002482\mathrm{m}} \cdot \frac{920.81}{gc} \cdot \frac{(0.03502)^2\mathrm{kg}^2}{\mathrm{s}^2} \cdot \frac{0.1\mathrm{m}}{(0.000527)^{3-1}} \cdot \frac{(0.999)^{3-1}}{0.001^3} \cdot \frac{1\mathrm{m}^3}{995\mathrm{kg}}$$

$$\therefore \Delta P = 3.2864/gc$$

Using the velocity-head concept, an approximate velocity drop was computed as follows:

$$\Delta P = \Delta h \approx 50\left(V^2/2g_c\right)$$

$$\therefore V^2 = \frac{(\Delta P)(2gc)}{50}$$

$\therefore V^2$ =	3.2864	2gc	2 1	= 0.13146
	gc		50	

V = 0.3626kg/s – mass velocity.

Thus, this simply correlates that the mass velocity of steam in the still should be maximum at $0.1788 + 0.03502 = 0.39762 kg/s$

3.9.2: Condenser design

In selecting the flow path for two fluids flowing through the condenser several general approaches was considered. The tube-side fluid is more corrosive or dirtier or at a higher pressure and the shell-side fluid was a liquid of high viscosity or a gas. Generally, oils are corrosive and therefore always assigned to the tube-side of the condenser.

Calculation of the heat duty or load or heat transfer

The heat duty was calculated from the expression:

$$q = UA\Delta T_{LM}$$

Where U = overall heat transfer coefficient

A = surface area for heat transfer consistent with definition of U

ΔT_{LM} = log mean temperature difference.

Heat capacity of the steam-oil mixture, = 4,100 J/kg^{0}C

Hence heat capacity or load is given by:

$q = M_m.C.\Delta T_{Lm}$

0.04377kg	4,100J	38.05^{0}C
S	kg^{0}C	

$$= 6828.339 J/s$$

The cold water mass flow rate was obtained as follows:

Heat capacity of water = 4,200 J/kg°C

Coolant water mass flow rate, $G_w = Q/C\Delta T$

Where Q = heat duty or load

C = heat capacity of water

ΔT = temperature change

$$\therefore G_w = \begin{array}{c|c|c} 6828.339\text{ J} & \text{kg}^0\text{C} & \\ \hline \text{s} & 4200\text{J} & 75^0\text{C} \end{array}$$

$$= 0.02168 kg/s$$

Mean log temperature difference

It was observed that the temperature difference between the hot and cold water varies between inlet and outlet for which an average was used.

Hence, the mean log temperature change was obtained as follows:

$$\Delta T_m = \frac{[(100-45)-(50-25)]}{In[(100-45)/(50-25)]} = 38.050^0 C$$

Calculating the dimensionless temperature ratio for a one-shell pass and two-tube passes was as follows:

$$R = (100-45)/(50-25) = 55/25 = 2.2$$

and

$$S = (50-25)/100-45) = 25/55 = 0.45$$

In actual case, the temperature differences between the two fluids in the condenser vary from point to point. Thus, the Mean Temperature Difference (LMTD) was calculated from the terminal temperatures as was done above for the two streams on the assumptions that the following statements are valid.

i. All elements of the fluid stream have the same thermal history in passing through the condenser.

ii. The condenser operated at steady state

iii. The specific heat was constant for each stream (or either stream undergoes an isothermal phase transition).

iv. The over-all heat-transfer coefficient was constant

v. Heat losses were negligible.

From the temperature correction factor chart, F_t was obtained as 0.68

$\therefore$ Log Mean temperature difference $= \Delta T_{LM} = 0.68 \times 38.05 = 25.87^o C$

However, since the condenser is assumed to operate at steady state and isothermal condensation prevailed, ΔT_{LM} was not necessary.

From the correction table of hot fluid versus cold fluid, the overall heat transfer coefficient, U_0, was taken as 300w/m^2 ^{0}C. Hence, the provisional area, Ap, was calculated as follows:

$$Ap = Q / U_0 \Delta T_m$$

$$= \begin{array}{r|c|l} 6828.339J & 1\ s\ m^2\ {}^0C & \\ \hline s & 300J & 38.05^0C \end{array}$$

$$= 0.5982\ m^2$$

$\therefore$ Provisional area, Ap = 0.5982 m^2

From standard data for condensers and heat exchangers tube (Pallen and Taboreck, 1969), a 16 mm internal diameter (di), 20 mm outer diameter (do) and 0.80 m tube length (L), made from super-cooled glass was chosen.

Area of 1 tube, A = Circumference x L

$$= 2\pi r L = \pi D L$$

$$\therefore A = (\pi)(0.02m)(0.80m) = 0.0503m^2$$

Hence, number of tubes was obtained thus:

$$N_t = A_p / A = 0.5982/0.0503 = 11.89$$

The estimated shell diameter was obtained from the expression, (Perry and green, 1984),

$$D_h = d_o (N_t / K_1)^{0.453}$$

Where K_1 is a constant = 0.0193

$$\therefore Dh = \begin{array}{c|c} 0.02m & (11.89/0.0193)^{0.453} \\ \hline & \end{array}$$

= 0.367 m

= 367 mm

Tube-Side heat-transfer coefficient calculations

Mean water-temperature

$$= \frac{\text{Cold water temperature} + \text{Hot water temperature}}{2}$$

$$= \frac{25 + 50}{2} = 75/2 = 37.5^{\circ} C$$

Tube-side cross sectional area $= \dfrac{\pi D^2}{4} = 0.786D^2$

$$= (0.786)(16mm)^2 = 201.22mm^2 = 2.0122\times 10^{-4} m^2$$

Tube per pass $= 17.20/2 = 8.6 \approx 9$

Total flow area, A_{TW} = Tube cross-sectional area x tube per pass

$$= (8.6 \times 2.0122 \times 10^{-4})\ m^2$$

$$= 1.73\times 10^{-3} m^2$$

Water mass velocity, $G_S = G_W/A_{TW}$ =

$$\begin{array}{c|c} 0.03133kgs & 10^3 \\ \hline s & 1.73\ m^2 \end{array}$$

= 18.11 kg/s.m^2

Density of steam = 995 kg/m^3

Viscosity of water, $\mu_w = 8.0\times 10^{-4}\ Ns/m^2$

Thus, linear velocity of water $= \dfrac{\text{Mass velocity of water}}{\text{Density of water (steam)}}$

$$\therefore U \quad \begin{array}{c|c} 18.11kg & 1\ \ m^3 \\ \hline sm^2 & 995kg \end{array}$$

= 0.0182 m/s

The heat transfer coefficient for the tube-side is obtained from the expression. (Perry and Green, 1984).

$$h_i = [4200(1.35 + 0.02t) \times U^{0.8}]/di^{0.2}$$

$$h_i = \frac{\left[4200(1.35 + 0.02\times 24)\times (0.0182)^{0.8}\right]}{(0.016)^{0.2}}$$

$$= \frac{4200 \times 1.83 \times 0.04056}{0.43735}$$

$$= 712.80\, w/m^{2\,o}\, C$$

Hence, the Reynolds number for the tube-side was computed from the relation given by:

$$NR_e = \frac{G_s \times d_e}{\mu_w}$$

Where G_s = water mass velocity

d_e = equivalent diameter

μ_w = viscosity of water

$$\therefore NR_e = \begin{array}{l|c|c} 18.11\text{kg} & 0.016\text{m} & 10^4\text{sm} \\ \hline \text{sm}^2 & & 8.0\text{kg} \end{array}$$

$= 362.2$

Shell-Side heat-transfer coefficient calculations

Calculation of shell-side geometrical parameters

Assuming a bundle clearance allowance of 10 mm, the actual shell diameter, ds, becomes 367 mm + 10 mm = 377 mm

Hence, baffle spacing, $I_B = \frac{\text{Shell diameter}}{5} = \frac{377}{5} = 75.4mm$

Square tube pitch, $p_t = 1.25 d_o$

Where d_o = outer diameter = 20 mm

$\therefore p_t = 1.25 \times 0.02 = 0.025m = 25m$

The fraction of crossflow area available for bypass flow can be determined by obtaining the cross flow area, A_S.

Cross flow area, $A_S = \left[\frac{(p_t - d_o)}{p_t}\right] dsI_B$

$$A_S = \begin{array}{c|c|c|c} (0.025-0.02)\text{ m} & 0.377\text{ m} & 1 & 0.0754\text{ m} \\ \hline & & 0.025\text{ m} & \end{array}$$

$$=0.005685\text{ m}^2 = 5.685 \times 10^{-3}\text{ m}^2$$

The mass velocity was calculated as follows:

Mass velocity, $G_{SI} = G_w / A_s$

$$G_{si} = \begin{array}{c|c} 0.04377\text{ kg} & 10^3 \\ \hline \text{s} & 5.685\text{ m}^2 \end{array}$$

$$=7.699\text{ kg/s.m}^2$$

The equivalent diameter, de, was obtained from the correlation. (Perry and Green, 1984).

$$d_e = 1.27/d_o\left(p_t^2 - 0.785 d_o^2\right)$$

$$\therefore d_e = \frac{1.27}{20}\left(25^2 - 0.785(20)^2\right) = 0.0635(625 - 314)$$

$$\therefore d_e = 19.75mm$$

Hence, linear velocity, $U = \frac{\text{mass velocity}}{\text{density of steam - oil mixture}}$

$$\therefore U = \begin{array}{c|c} 7.699\text{kg} & \text{m}^3 \\ \hline \text{sm}^2 & 900.76\text{kg} \end{array}$$

$$= 8.5472 \times 10^{-3}\text{ m/s}$$

The mean shell temperature = (100 + 50)/2 = 75°C.

Viscosity, µ, of steam-oil mixture = 6.98×10^{-4} Ns/m^2

Estimation of wall temperature

The mean temperature difference was obtained as follows:

$$75 - 38 = 37^{o}C$$

Thus, mean temperature difference across the oil film

$$= \left(\frac{U}{h_s}\right)\Delta T = \left(\frac{300}{1892.45}\right)(37) = 5.87^{o}C$$

Hence, mean wall temperature = 75 – 5.87 = 69.13°C

The over-all heat transfer coefficient was calculated from the correlation expression as: (Perry and Green, 1984);

$$\frac{1}{U} = \frac{1}{h_s} + \frac{1}{h_{od}} + \left[d_o l_n (d_o/d_i)\right] \Big/ 2k_w + (d_o/d_i) \times 1/h_{id} + (d_o/d_i)/1/h$$

Where h_{id} and h_{od} are the inside ant outside fluid film coefficient, $w/m^{2\,o}\,C = 6000$

k_w = thermal conductivity of the tube wall material = 205.9 $w/m^{2\,0}C$

$$\frac{1}{U} = \frac{1}{234.785} + \frac{1}{6000} + \left[0.020 l_n \left(\frac{0.020}{0.016}\right)\right] / 2 \times 205.9 + \frac{0.02}{0.016}$$

$$\times \frac{1}{6000} + \frac{0.02}{0.016} \times \frac{1}{299.85}$$

$$= 0.004259 + 0.0001667 + 0.00001084 + 0.0002083 + 0.004169 = 0.00881384$$

$$\therefore \frac{1}{U} = 0.00881384$$

$$U = 113.46\, w/m^{2\,o}\,C$$

Hence, the calculated over-all heat transfer coefficient,

$$U_o = 113.46\, w/m^{2\,o}C$$

This value for the over-all heat transfer coefficient was well below the assumed values and thus, showed the low heat load of the condenser and potential for process expansion.

Calculation of pressure drop

At the tube-side

From the Grimison correlation chart, at $NR_e = 362.2$

$$j_k = 0.164 \times 10^{-1} = 0.0164$$

$$\Delta P = \left[8 j_k (L/d_i)(\mu/\mu_w)^{0.14} + 2.5\right] \ell U_t^2 / 2$$

$$= \left[(8 \times 0.0164)(0.55/0.016)(6.98/8)^{0.14} + 2.5\right] \frac{(995)(0.0182)^2}{2}$$

$$= 1.14\, N/m^2$$

At the shell-side

From the Grimison correlation chart, at $NR_e = 1272$

$$j_k = 0.04$$

$$\therefore \Delta P = \left[8 j_k (d_s/d_e)(L/I_B)\right] \ell U_s^2 / 2$$

$$= (8 \times 0.04)(377/19.75)(0.80/0.0754)(900.76)(0.0499)^2 / 2$$

$$= 7.268 \text{ N/m}^2$$

Thus, the calculated pressure drop in the condenser indicated that the baffle pitch was adequate.

3.9.3: Separator design

The separator unit undertakes the separation of the water-oil mixture based on the criteria of immiscibility of the two liquids. The oil and water layer was allowed to accumulate in the separating vessel to be drawn off periodically. Separation was expected to be effected after every hour.

Thus, the volume of the vessel =

$$\begin{array}{r|l} 3.8874\text{x}10^{-5}\text{m}^3 & 3600\text{sec} \\ \hline \text{sec} & \end{array}$$

$$= 0.13995m^3$$

Allowing 20% extra volume for safety, the actual volume was calculated as follows:

20% of $0.13995\text{m}^3 = 0.20 \times 0.13995\ \text{m}^3$

$$= 0.02799\text{m}^3$$

$\therefore$ Actual volume of vessel $= (0.13995 + 0.02799)\ \text{m}^3$

$$= 0.16794\ \text{m}^3$$

Since $V = 0.785D^2h$

Taking the internal diameter as 0.2m, the vessel height was computed as follows:

Vessel height, $h = \dfrac{V}{0.785D^2}$

$$\begin{array}{r|c} 0.16794\text{m}^3 & 1 \\ \hline & 0.785(0.2\text{m})^2 \end{array}$$

$$= 0.535m$$

3.9.4: Design of the piping system

Design condition:

The practical influences applicable to the design of the piping system included internal and external pressures and design material temperature.

The pipe sizing for this design was performed in accordance with Darcy Rational relation for compressible fluids. Calculated value of flow rate of the system at steady state was 0.03502kg/s or $3.8874\text{x}10^{-5}m^3/s$

For a schedule 40 standard nominal pipe (A.N.S.I. B31, 1972) of size 1.0in, of internal diameter 1.049in (0.02665m), the Reynolds number for flow configuration was computed thus:

$$NR_e = 50.6Q/d\mu_w$$

$$\begin{array}{r|c|c} 50.6\text{x}0.38874m^3 & 1 & s \\ \hline s & 0.02665m & 0.8m^2 \end{array}$$

$$= \quad 922.62$$

This represents a laminar flow regime.

From the Moody chart, the friction factor, f, was calculated as follows:

Friction factor, $f = 64/NR_e = 64/922.62 = 0.0694$

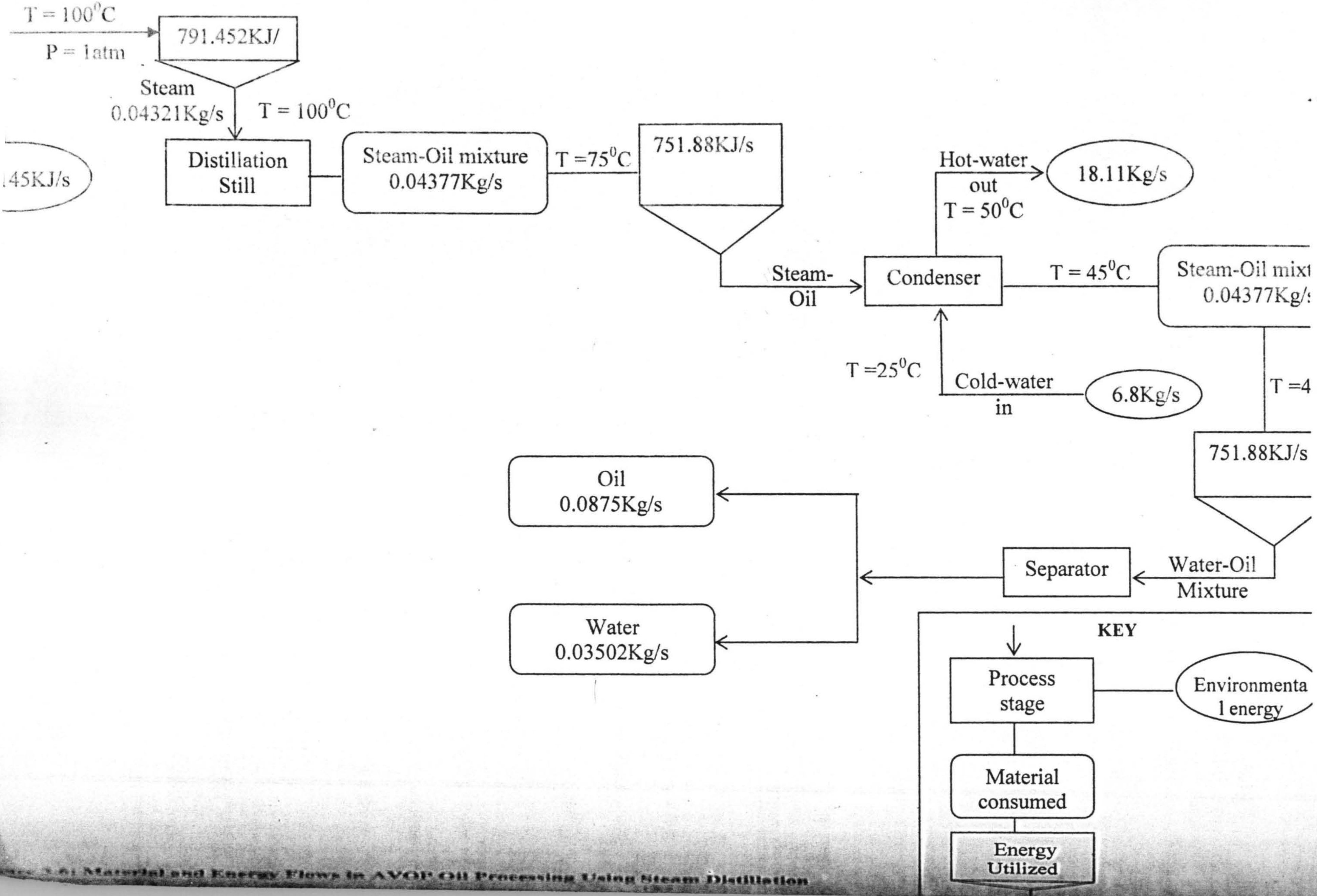
The length of pipe was assumed to be 1.0m based on the expected distance between the still and the condenser. The rate of discharge, size of channel, pressure head drop or head loss for the pipe, h, was calculated as follows:

$$h = fLQ^2/2g$$

$$= \begin{array}{r|c|c|c} 6.94\text{x}10^{-2} & 1.0m & (3.8874\text{x}10^{-5})^2m & s^2 \\ \hline & & s^2 & 2 \text{ x } 9.8m \end{array}$$

$$= 5.357 \times 10^{-12} m$$

From the value for head loss, it was seen that the loss was quite insignificant and thus, could be neglected. This implies that the size chosen was adequate



3.6: Material and Energy Flows in AVOP Oil Processing Using Steam Distillation

3.10: Economic Analysis

3.10.1: Economic analysis on steam distillation process

Douglass documents cost correlation for the capital cost also referred to as the purchase cost was given by the relation thus:

$$PC\$ = \left(\frac{M\$S}{280}\right)\left(101.9D^{1.066}H^{0.802}(2.18+F_e)\right)$$

Where D = diameter (ft), H = height (ft), F_c = Cost factor, F_p = Pressure factor F_m = Material factor.

Now, M$S = 1100, D = 0.231m = 0.758 ft

H = 0.464m = 1.522 ft

Pressure factor, F_p = 3.86

Exchange rate, $1.00 = N130.00

Material factor for stainless steel, F_m = 1.7

$F_c = 1.00 + F_m + F_p = 1.00 + 1.7 + 3.86 = 6.56$

Hence,

i. Purchase cost of the still (distillation apparatus)

$$:- PC\ \$ = \frac{1100}{280}\left(101.9(0.7580)^{1.066}(1.522)^{0.802}(2.18+6.65)\right)$$

$$PC\ \$ = [(3.9286)(101.9(0.7443)(1.40054)(8.74))]$$

$$= (3.9286)(928.387)$$

$$= \$3,647.261$$

= ₦474,143.93

Richardson and Coulson established a cost model for the capital cost of any equipment. This was given by the relation.

$$C_e = CS^n$$

Where C_e = purchase equipment cost, S = size range of equipment

C= cost constant, n = equipment index.

However, the present cost of equipment as at 1992 with appropriate allowances for inflation, insurance, piping, insulation, painting and transportation was given by the relation.

$$\text{Present cost of equipment} = \text{cost at 1992}\frac{(\text{Cost index in 1992})}{(\text{Cost index in the required period})}$$

$$= \text{cost at 1992}\left(\frac{155.52}{108}\right)$$

$$= \text{cost at } 1992 \times 1.44$$

∴ Present cost of equipment = cost at 1992 x 1.44

Where 1.44 is a factor for relating cost at specified periods

Cost values of the equipment used for steam distillation were obtained from Richardson and Coulson.

ii. Cost estimate for condenser unit

S = size of equipment, Ap = $0.865m^2$

C = Cost constant = \$1107.2

n = 0.7

∴ Purchase cost, $C_e = 1107.2\ (0.865)^{0.7}$

= \$ 1000.32

Present cost of condenser = \$1000.32 x 1.44

= \$1,440.46

= N187,259.80

iii. Cost estimate for separator unit

S = size of separator unit = $0.16794 m^3$

C = Cost constant = \$778.00

n = 0.6

$\therefore$ Purchase cost, C_e = \$778.00 $(0.16794)^{0.6}$

= \$384.09

= N49,931.70

Thus, the total physical plant cost was obtained as follows:

Total physical plant cost

PPC = PCE = $(1+F_1 + F_2 + \ldots + F_n)$

WherePCE = Physical Cost of Equipment

= PCE = \$ (3,647.261 + 1,440.46 + 384.09

= \$ 5,471.811

= N711,335.43

$\therefore$ Total Plant Physical Cost

PPC = PCE x 3.15

Where 3.15 is a factor for relating the physical equipment cost to the total plant physical cost.

:- PPC = \$ 5,471.81 x 3.15

= \$ 17,236.20

= N2,240,706.00

iv. The Total Fixed cost was obtained as follows:

Total Fixed Cost = PPC x 1.40

Where 1.40 is a factor for relating PPC to the total fixed cost

= $ 17,236.20 x 1.40

= $ 24,130.68

= N3,136,988.40

v. Operating or Working capital

Raw materials	$	
Avocado apples		
360 pieces of avocado apple at $ 0.29/apple	104.40	(N13,572.00)

Utilities		
Electricity (550 kW installed capacity, consumption rate 250 kw)		
294 kwhcl at 0.05 $/Kwhcl	14.70	
Fresh water (4.54 L/hr) 80L at $ 0.12/L	9.60	
Steam (2.25 L/hr) obtained from heating water in the still using electric stove)	14.81	
	39.11	(N5,084.30)

Hence, total operating or working capital

= $ (104.40 + 39.11) = $ 143.51

= N18,656.30

vi. Total investment = fixed capital + working capital

= $ (24,130.68 + 143.51)

= $ 24,274.19

= N3,155,644.70

vii. Contingency = 5% of working capital

= 0.05 x \$ 143.51

= \$ 7.18

= N933.40

Conclusions

Initial capital outlay = total investment

= \$ 24,274.19

= N3,155,644.70

Annual revenue

Refined AVOP oil 57,444ml at \$ 20/60ml bottle \$19,148.00

= N2,489,240.00

viii. Direct production cost (DPC)

= Total investment = \$ 24,274.19 = N3,155,644.70

Applying the time value of money using net present value (NPV) profitability measure, the payback period (PBP) or payout time is obtained thus:

Pay Back Period (PBP)

$$\text{PBP} = \frac{\text{Direct Production cost}}{\text{Annual revenue}}$$

$$= \frac{24{,}274.19}{19{,}148.00}$$

$= 1.27$ year ≈ 1.3 year

The rate of return on investment (ROI) is given thus

$$\text{ROI} = \frac{\text{Annual return on investment}}{\text{Total capital investment}} \times 100\%$$

$$= \frac{\text{present value of project earnings}}{\text{original fixed investment + working capital}} \times 100\%$$

$$= \frac{19{,}148.00}{24{,}274.19} \times 100\%$$

$= 78.88\%$

3.10.2 Economic analysis on indirect leaching process

Douglas, 1988 developed cost model depending on the extractor type, trays and/or packing internals to be used. The cost model for the purchase and installation of these materials based upon correction factors is given thus;

i. Capital cost of extractor, $\$ = \left(\frac{M\,\&\,S}{280}\right)\left(101.9D^{1.066}H^{0.802}\right)$

ii. Capital cost of internals, $\$ = \left(\frac{M\,\&\,S}{280}\right)\left(4.7D^{1.55}H.Fc\right)$

The correction factor is the sum of the correction factor for the spacing internal type and internal material (Douglas, 1988).

Extraction column dimensions;

Diameter, D, ft = 0.758 ft

Height, H, ft = 1.522 ft

Using the Douglass, 1988 correlation for capital costs;

Extractor column cost, $\$ = \left(\frac{1061}{280}\right)\left(101.9 \bullet 0.758^{1.066} \bullet 1.522^{0.802}\right)$

$= \$\ 402.49$

Cost of internals, $\$ = \left(\frac{1061}{280}\right)\left(4.7.0.758^{1.55}.1.522\right)$

$= \$17.64 = N2{,}293.20$

Cost of condenser unit;

S = size of equipment, Ap = $0.865m^2$

C = cost constant = \$ 1107.2, N = 0.7

Purchase cost, Ce = 1107.2 $(0.865)^{0.7}$ = \$1000.32 = N130,041.60

Present cost of condenser = \$1,000.32x1.44 = \$1,440.46 = N187,259.80

Total capital cost = capital cost of column + capital cost of internals + cost of condenser

iii. Total capital cost = \$ (402.49 + 17.64 + 1,440.46) = \$1,860.59 = N241,876.70

iv. Operating cost

Raw materials

		\$	
1.	Avocado apples		
	360 pieces of avocado apples at \$0.29/apple	104.40	(N13,572.00)

2. Chemicals.

Solvents.

i.	n – hexane	(2.5L) at \$58.40	58.40	
ii.	Anhydrous ethanol	(2.5L) at \$58.40	58.40	
iii.	Caustic soda	10g at \$2.00	2.00	
iv.	Bleaching earth	10g at \$2.90	2.90	
			\$121.70	(N15,821.00)

Utilities

Electricity (550Kw installed capacity, consumption rate 450Kw)

294 Kwhcl at 0.05$/Kwhcl	14.70	
Fresh water (4.5 L/hr) 80L at $0.15/L	12.00	
	$26.70	(N3471.00)

v Solvent and product recovery cost

Utilities.	$
Electricity cost for solvent $ product recovery = $\left(\frac{0.04\$}{kWhr}\right)\left(\frac{2000}{600}\right)$	3.20
Water (4.5 L/hr) 80 L at $ 0.15 /L	12.00
	$ 15.20 (N1,976.00)

vi. Total investment = Total capital cost + operating cost + solvent and product recovery cost.

= $ (1,860.59 + 252.80 + 15.20) = $ 2,128.59

= $ 2,128.59 = N276,716.70

vii. Contingency = 5% of operating cost

= 0.05 x 252.80

= $ 12.64 = N1643.20

Conclusions;

Initial capital outlay = Total investment

= $ 2,128.59 = N276,716.70

Annual revenue

Refined AVOP oil 2,541 ml at $ 20/60ml bottle $ 847.0 N110,110.00

vii. Direct production cost (DPC)

= Total investment = $ 2,128.59 = N276,716.70

Payback period (PBP)

$$\text{PBP} = \frac{\text{Direct production cost}}{\text{Annual revenue}}$$

$$= \frac{2,128.59}{847.40}$$

$$= 2.51 \text{ years} \approx 2.5 \text{ years}$$

Rate of return on investment

$$\text{ROI} = \frac{\text{Annual revenue}}{\text{Total capital investment}} \times 100\%$$

$$= \frac{847.40}{2,128.59} \times 100$$

$$= 39.81\%$$

3.10.3 Economic analysis on direct leaching process

Using the Douglass 1988 correlation document for capital cost of an extractor column given thus;

Extraction column cost, \$ $\$ = \left(\frac{M \& S}{280}\right)(101.9D^{1.066}H_{0.802})$

Where D = diameter, ft = 0.758 ft

H = height, ft = 1.522 ft

The cost of the extractor for the direct leaching process is obtained as follows:

i. Extractor column cost, $\$ = \left(\frac{1061}{280}\right)(101.90.758^{1.066}1.522_{0.802})$

$= \$\ 402.49$

ii. Cost of condenser unit;

S = size of equipment, Ap = 0.865m^2

C = cost constant

:- Purchase cost, Ce = \$1107.2 (0.865)07 = \$ 1000.32 = N130,041.60

Present cost of condenser = \$ 1000.32 x 1.44 = \$ 1,440.46 = N187,259.80

Hence, total capital cost = capital cost of column + cost of condenser

iii. Total capital cost = \$ (402.49 + 1,440.46)

= \$ 1,842.95 = N239,583.50

iv. Operating cost

Raw materials		\$	
1. Avocado apples			
360 pieces of avocado apples at \$ 0.29/apple		104.40	
		\$104.40	N13,572.00
2. Chemicals			
Solvents			
i. n-hexane	(2.5 litres) at \$ 58.40	58.40	
ii. Anhydrous ethanol	(2.5 litres) at \$ 58.40	58.40	
iii. Caustic soda	10g at \$ 2.00	2.00	
iv. Bleaching agent	10g at \$ 2.90	2.90	
		\$ 121.70	N15,821.00
Utilities			
Electricity (550Kw installed capacity, consumption rate 450 Kw)			
294 kwhcl at 0.05 \$/kwhl		14.70	
Fresh water (4.5L/hr)	80 litres at \$ 0.15 Litre	12.00	
		\$ 26.70	N3471.00

vi. Solvent and product recovery cost

Utilities		$
Electricity cost for solvent and product recovery = $\left(\frac{0.04\$}{kWhr}\right)\left(\frac{2000}{600}\right)$		3.20
Water (4.5L/hr)	80 litres at $ 0.15 /litre	12.00
		$ 15.20 N1,976.00

vii. Total investment = total capital cost + operating cost + solvent and product recovery cost

= $ (1,842.95 + 252.80 + 15.20)

= $ 2,110.95 = N274,423.50

vii. Contingency = 5% of operating cost

= 0.05 x 252.80

= $ 12.64 = N1643.20

Conclusions

Initial capital outlay = total investment

= $ 2,110.95 = N274,423.50

Annual revenue

Refined AVOP oil 3,865.80 ml at $ 20/60ml bottle $ 1,288.60 = N167,518.00

viii. Direct production cost (DPC)

= Total investment = $ 2.110.95 = N274,423.50

Payback period (PBP)

$$PBP = \frac{\text{Direct production cost}}{\text{Annual revenue}}$$

$$= \frac{2{,}110.95}{1{,}288.60}$$

$$= 1.64 \text{ year} \approx 1.6 \text{ year}$$

$$\text{ROI} = \frac{\text{annual revenue}}{\text{Total capital investment}} \times 100\%$$

$$= \frac{1{,}288.60}{2{,}110.95} \times 100\ \%$$

$$= 61.04\%$$

The following assumptions had been made in the compilation of the cash flow development.

I The plant has a 20 year life, though in practice it might continue operating for an even longer period provided that the standard of maintenance has been good.

ii It is assumed that months would be required until start-up and that the plant would assume full capacity from initial operation.

Iii No depreciation had been allowed for as the pre-tax position was examined. Depreciation was not regarded as a working cost but was an allowance against tax and would therefore not affect the liquidity position.

CHAPTER FOUR

4.0 RESULTS

4.1 Results of Steam Distillation

The yields of steam distillation of avocado apple's pericarp at various thermodynamic condition and particle sizes of the avocado's pericarp are given in Tables 4.1 – 4.10.

Table 4.1: Yields of Oils at Varying Steam Heating Rates and 1000 ml of Water

Volume of water (ml)	Mass of AVOP	Time (mins)	Mass of AVOP after extraction and drying (g)	Volume of steam (ml)	Steam rate (ml/min)	Steam-sample ratio (ml/g)	Mass of oil (g)	% yield
1000	10	30	7.78	20.54	0.69	2.64	2.22	22.2
1000	10	60	7.25	59.60	0.99	8.22	2.75	27.5
1000	10	90	6.86	181.65	2.02	26.48	3.14	31.4
1000	10	120	6.47	247.15	2.06	38.20	3.53	35.3
1000	10	150	6.48	369.20	2.46	56.98	3.52	35.2
1000	10	180	6.47	434.70	2.42	67.19	3.53	35.3

Table 4.2: Yields of Oils at Varying Steam Heating Rates and 1250 ml of Water

Volume of water (ml)	Mass of AVOP (g)	Time (mins)	Mass of AVOP after extraction and drying (g)	Volume of steam (ml)	Steam-sample ratio (ml/g)	Steam rate (ml/min)	Mass of oil (g)	% yield
1250	10	30	7.68	39.94	5.20	1.44	2.32	23.2
1250	10	60	7.37	94.48	12.82	1.57	2.63	26.3
1250	10	90	6.84	195.62	28.60	2.17	3.16	31.6
1250	10	120	6.26	268.43	42.88	2.24	3.74	37.4
1250	10	150	6.26	369.57	59.04	2.46	3.74	37.4
1250	10	180	6.26	565.19	90.29	3.14	3.74	37.4

Table 4.3: Yields of Oil at Varying Steam Heating Rates and 1500 ml of Water

olume f water (ml)	Mass of AVOP (g)	Time (mins)	Mass of AVOP after extraction and drying (g)	Volume of steam (ml)	Steam rate (ml/min)	Steam-sample ratio (ml/g)	Mass of oil (g)	% yield
1500	10	30	7.62	76.35	2.55	10.02	2.38	23.8
1500	10	60	7.12	214.45	3.57	30.12	2.88	28.8
1500	10	90	6.56	407.25	4.53	62.08	3.44	34.4
1500	10	120	6.13	566.04	4.72	92.34	3.87	38.7
1500	10	150	6.13	749.83	4.99	122.32	3.87	38.7
1500	10	180	6.13	826.18	4.59	134.78	3.87	38.7

Table 4.4: Yields of Oils at Varying Steam Heating Rates and 1000 ml of Water

Volume of water (ml)	Mass of sample (g)	Time (mins)	Mass of sample after extraction and drying (g)	Volume of steam (ml)	Steam rate (ml/min)	Steam-sample ratio (ml/g)	Mass of oil (g)	% yield
1000	10	30	7.47	103.76	3.46	13.89	2.53	25.3
1000	10	60	7.14	301.59	5.03	42.24	2.86	28.6
1000	10	90	6.82	463.62	5.15	67.98	3.15	31.8
1000	10	120	6.20	597.87	4.98	96.43	3.80	38.0
1000	10	150	6.20	732.12	4.89	118.08	3.80	3.80
1000	10	180	6.20	821.24	4.56	132.46	3.80	3.80

Table 4.5: Yields of Oils at Varying Steam Heating Rate and 1250 ml of Water

Volume of water (ml)	Mass of AVOP (g)	Time (mins)	Mass of AVOP after extraction and drying (g)	Volume of steam (ml)	Steam rate (ml/min)	Steam-sample ratio (ml/g)	Mass of oil (g)	% yield
1250	10	30	7.56	122.77	4.09	16.24	2.44	24.4
1250	10	60	7.11	332.18	5.54	46.72	2.89	28.9
1250	10	90	6.52	458.23	5.09	70.28	3.48	34.8
1250	10	120	6.04	592.77	4.94	98.14	3.96	39.6
1250	10	150	6.04	715.54	4.77	118.47	3.96	39.6
1250	10	180	6.04	865.77	4.81	143.34	3.96	39.6

Table 4.6: Yields of Oils at Varying Steam Heating Rate and 1500 ml of Water

olume of water (ml)	Mass of AVOP (g)	Time (mins)	Mass of AVOP after extraction and drying (g)	Volume of steam (ml)	Steam rate (ml/min)	Steam-sample ratio (ml/g)	Mass of oil (g)	% yield
1500	10	30	7.64	153.03	5.10	20.03	2.36	23.6
1500	10	60	7.27	365.54	6.09	50.28	2.73	27.3
1500	10	90	6.62	519.47	5.77	78.47	3.38	33.8
1500	10	120	6.25	616.75	5.14	98.68	3.75	37.5
1500	10	150	6.25	766.82	5.11	122.69	3.75	37.5
1500	10	180	6.25	915.05	5.08	146.41	3.75	37.5

Table 4.7: Yields of Oil at Varying Steam Heating Rates and 1000 ml of Water

Volume of water (ml)	Mass of AVOP (g)	Time (mins)	Mass of AVOP after extraction and drying (g)	Volume of steam (ml)	Steam rate (ml/min)	Steam-sample ratio (ml/g)	Mass of oil (g)	% yield
1000	10	30	7.20	63.36	2.11	8.80	2.80	28.0
1000	10	60	6.20	173.60	2.89	28.00	3.80	38.0
1000	10	90	5.82	302.64	3.36	52.80	4.18	41.8
1000	10	120	4.02	308.98	2.57	76.86	5.98	59.8
1000	10	150	4.02	372.34	2.48	92.62	5.98	59.8
1000	10	180	4.02	545.94	3.03	135.81	5.98	59.8

Table 4.8: Yields of Oils at Varying Steam Heating Rates and 1250 ml of Water

Volume of water (ml)	Mass of AVOP (g)	Time (mins)	Mass of AVOP after extraction and drying (g)	Volume of steam (ml)	Steam rate (ml/min)	Steam-sample ratio (ml/g)	Mass of oil (g)	% yield
1250	10	30	7.33	71.83	2.39	9.80	2.67	26.7
1250	10	60	6.38	183.74	3.06	28.80	3.62	36.2
1250	10	90	5.88	366.91	4.08	62.40	4.12	41.2
1250	10	120	5.28	463.58	3.86	87.80	4.72	47.2
1250	10	150	5.28	555.03	3.70	105.12	4.72	47.2
1250	10	180	5.28	738.45	4.10	139.86	4.72	47.2

Table 4.9: Yields of Oils at Varying Steam Heating Rates and 1500 ml of Water.

Volume of water used (ml)	Mass of AVOP (g)	Time (mins)	Mass of AVOP after extraction and drying (g)	Volume of steam (ml)	Steam rate (ml/min)	Steam-sample ratio (ml/g)	Mass of oil (g)	% yield
1500	10	30	7.20	86.40	2.8s8	12.00	2.80	28.0
1500	10	60	6.43	24.34	4.07	38.00	3.57	35.7
1500	10	90	4.98	358.56	3.98	71.99	5.02	50.2
1500	10	120	4.12	403.76	3.36	98.00	5.88	58.8
1500	10	150	4.12	493.27	3.29	119.73	5.88	58.8
1500	10	180	4.12	693.39	3.85	168.30	5.88	58.8

Table 4.10: Summary of Yields of Oils at Varying Steam Heating Rates, Varying Volumes of Water and Optimum Extraction Time.

Volume of water (ml)	Mass of AVOP (g)	Time (mins)	Mass of AVOP after extraction and drying (g)	Volume of steam (ml)	Steam rate (ml/min)	Steam-sample ratio (ml/g)	Mass of oil (g)	% yield
1000	10	120	6.47	247.15	2.06	38.20	3.53	35.3
1250	10	120	6.26	268.43	2.24	42.88	3.74	37.4
1500	10	120	6.13	566.04	4.72	92.34	3.87	38.7
1000	10	120	6.20	597.87	4.98	96.43	3.80	38.0
1250	10	120	6.04	592.77	4.94	98.14	3.96	39.6
1500	10	120	6.25	616.75	5.14	98.68	3.75	37.5
1000	10	120	4.02	308.98	2.57	76.86	5.98	59.8
1250	10	120	5.28	463.58	3.86	87.80	4.72	47.2
1500	10	120	4.12	403.76	3.36	98.00	5.88	58.8

4.2 Results of Extraction (Indirect and Direct Leaching)

The yields of extraction (indirect and direct leaching) of avocado apple's pericarp at various thermodynamic condition and particle sizes of the avocado apple's pericarp using n–hexane and anhydrous ethanol are given in Tables 4.11 – 4.28

4.2.1 Yields of indirect extraction (leaching) of avocado apple's pericarp

The yields of indirect extraction (leaching) of avocado apple's pericarp at various thermodynamic conditions (temperature and time) and particle sizes of the avocado's pericarp using n–hexane (bp 60^0C) and anhydrous ethanol (bp 78^0C) are given in Tables 4.11 – 4.23.

Table 4.11: Yields of Oils from Indirect Leaching Using N–Hexane at Varying Particle Size and 30 Minutes Extraction Time

Mass of AVOP (g)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Y
10	0.75	30	1	8.53	1.47		
		30	2	8.55	1.45	1.46	14
10	1.00	30	1	8.56	1.44		
		30	2	8.61	1.39	1.42	14
10	1.59	30	1	8.73	1.27		
		30	2	8.75	1.25	1.26	12
10	2.00	30	1	8.76	1.24		
		30	2	8.82	1.18	1.21	12

Table 4.12: Yields of Oils from Indirect Leaching Using N – Hexane at Varying Particle Size and 60 Minutes Extraction Time.

Mass Of AVOP (g)	Particle Size (mm)	Time (mins)	No. of runs	Mass Of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Yield
10	0.75	60	1	8.11	1.89		
		60	2	8.18	1.82	1.86	18.6
10	1.00	60	1	8.38	1.62		
		60	2	8.41	1.59	1.61	16.1
10	1.59	60	1	8.42	1.58		
		60	2	8.36	1.64	1.59	15.9
10	2.00	60	1	8.53	1.53		
		60	2	8.47	1.62	1.50	15.0

Table 4.13: Yields of Oils from Indirect Leaching Using N–Hexane at Varying Particle Size and 90 Minutes Extraction Time

Mass of AVOP (g)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Yield
10	0.75	90	1	7.96	2.04		
		90	2	8.01	1.99	2.02	20.2
10	1.00	90	1	8.16	1.84		
		90	2	8.21	1.79	1.82	18.2
10	1.59	90	1	8.26	1.74		
		90	2	8.31	1.69	1.72	17.2
10	2.00	90	1	8.38	1.62		
		90	2	8.42	1.58	1.60	16.0

Table 4.14: Yields of Oils from Indirect Leaching Using N–Hexane at Varying Particle Size and 120 Minutes Extraction Time

Mass Of AVOP (g)	Particle Size (mm)	Time (mins)	No. Of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Yield
10	0.75	120	1	7.90	2.10		
		120	2	7.86	2.14	2.12	21.2
10	1.00	120	1	8.09	1.91		
		120	2	8.10	1.90	1.91	19.1
10	1.59	120	1	8.20	1.80		
		120	2	8.22	1.78	1.79	17.9
10	2.00	120	1	8.29	1.71		
		120	2	8.30	1.70	1.71	17.1

Table 4.15: Yields of Oils from Indirect Leaching Using N--Hexane at Varying Particle Size and 150 Minutes Extraction Time

Mass Of AVOP (g)	Particle Size (mm)	Time (mins)	No. Of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Yield
10	0.75	150	1	7.90	2.10		
		150	2	7.86	2.14	2.12	21.2
10	1.00	150	1	8.09	1.91		
		150	2	8.10	1.90	1.91	19.1
10	1.59	150	1	8.20	1.80		
		150	2	8.22	1.78	1.79	17.9
10	2.00	150	1	8.29	1.71		
		150	2	8.30	1.70	1.71	17.1

Table 4.16: Yields of Oils from Indirect Leaching Using N–Hexane at Varying Particle Size and 180 Minutes Extraction Time

Mass Of AVOP (g)	Particle Size (mm)	Time (mins)	No. Of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Yield
10	0.75	180	1	7.90	2.10		
		180	2	7.86	2.14	2.12	21.2
10	1.00	180	1	8.09	1.91		
		180	2	8.10	1.90	1.91	19.1
10	1.59	180	1	8.20	1.80		
		180	2	8.22	1.78	1.79	17.9
10	2.00	180	1	8.29	1.71		
		180	2	8.30	1.70	1.71	17.1

Table 4.17: Yields of Oils from Indirect Leaching Using Anhydrous Ethanol at Varying Particle Size and 30 Minutes Extraction Time

Mass Of AVOP (g)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Yield
10	0.75	30	1	6.82	3.18		
		30	2	6.76	3.24	3.21	32.1
10	1.00	30	1	7.02	2.98		
		30	2	6.97	3.03	3.01	30.1
10	1.59	30	1	7.32	2.68		
		30	2	7.28	2.72	2.70	27.0
10	2.00	30	1	7.53	2.47		
		30	2	7.32	2.68	2.58	25.8

Table 4.18: Yields of Oils from Indirect Leaching Using Anhydrous Ethanol at Varying Particle Size and 60 Minutes Extraction Time

Mass of AVOP (g)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Yield
10	0.75	60	1	6.58	3.42		
		60	2	6.62	3.38	3.40	34.0
10	1.00	60	1	6.82	3.18		
		60	2	6.77	3.23	3.21	32.1
10	1.59	60	1	7.05	2.95		
		60	2	6.98	3.02	2.99	29.9
10	2.00	60	1	7.06	2.94		
		60	2	7.03	2.97	2.96	29.6

Table 4.19: Yields of Oils from Indirect Leaching Using Anhydrous Ethanol at Varying Particle Size and 90 Minutes Extraction Time

Mass of AVOP (g)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Yield
10	0.75	90	1	6.49	3.51		
		90	2	6.51	3.49	3.50	35.0
10	1.00	90	1	6.67	3.33		
		90	2	6.65	3.35	3.34	33.4
10	1.59	90	1	6.87	3.13		
		90	2	6.90	3.11	3.12	31.2
10	2.00	90	1	6.88	3.12		
		90	2	6.89	3.11	3.12	31.2

Table 4.20: Yields of Oils from Indirect Leaching Using Anhydrous Ethanol at Varying Particle Size and 120 Minutes Extraction Time

Mass of AVOP (g)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Yield
10	0.75	120	1	6.40	3.60		
		120	2	6.43	3.57	3.59	35.9
10	1.00	120	1	6.60	3.40		
		120	2	6.60	3.40	3.40	34.0
10	1.59	120	1	6.80	3.20		
		120	2	6.83	3.17	3.19	31.9
10	2.00	120	1	6.83	3.17		
		120	2	6.88	3.12	3.15	31.5

Table 4.21: Yields of Oils from Indirect Leaching Using Anhydrous Ethanol at Varying Particle Size and 150 Minutes Extraction Time

Mass of AVOP (g)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Yiel
10	0.75	150	1	6.40	3.60		
		150	2	6.43	3.57	3.59	35.
10	1.00	150	1	6.60	3.40		
		150	2	6.60	3.40	3.40	34.
10	1.59	150	1	6.80	3.20		
		150	2	6.83	3.17	3.19	31.
10	2.00	150	1	6.83	3.17		
		150	2	6.88	3.12	3.15	31.

Table 4.22: Yields of Oils from Indirect Leaching Using Anhydrous Ethanol at Varying Particle Size and 180 Minutes Extraction Time

Mass of AVOP (g)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Yield
10	0.75	180	1	6.40	3.60		
		180	2	6.43	3.57	3.59	35.9
10	1.00	180	1	6.60	3.40		
		180	2	6.60	3.40	3.40	34.0
10	1.59	180	1	6.80	3.20		
		180	2	6.83	3.17	3.19	31.9
10	2.00	180	1	6.83	3.17		
		180	2	6.88	3.12	3.15	31.5

Table 4.23: Summary of Yields of Oils from Indirect Leaching at Varying Extraction Time, 0.75 mm Particle Size (Optimum Particle Size) and Two Solvents

Mass of AVOP (g)	Solvent	Boiling Point (^{0}C)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Yield
10	n-hexane	60	0.75	30	1	8.53	1.47		
				30	2	8.55	1.45	1.46	14.6
10	n-hexane	60	0.75	60	1	8.11	1.89		
				60	2	8.18	1.82	1.86	18.6
10	n-hexane	60	0.75	90	1	7.96	2.04		
				90	2	8.01	1.99	2.02	20.2
10	n-hexane	60	0.75	120	1	7.90	2.10		
				120	2	7.86	2.14	2.12	21.2
10	Anhydrous ethanol	78	0.75	30	1	6.82	3.18		
				30	2	6.76	3.24	3.21	32.1
10	Anhydrous ethanol	78	0.75	60	1	6.58	3.42		
				60	2	6.62	3.38	3.40	34.0
10	Anhydrous ethanol	78	0.75	90	1	6.49	3.51		
				90	2	6.51	3.49	3.50	35.0
10	Anhydrous ethanol	78	0.75	120	1	6.40	3.60		
				120	2	6.43	3.57	3.59	35.9

4.2.2 Yields of direct extraction (leaching) of avocado apple's pericarp

The yields of direct extraction (leaching) of avocado apple's pericarp at various thermodynamic conditions (temperature and time) and varying particle size of the avocado's pericarp using n–hexane (bp 60^0C) and anhydrous ethanol (bp 78^0C)are given in Table 4.24 – 4.36.

Table 4.24: Yields of Oils from Direct Leaching Using N–Hexane at Varying Particle Size and 30 Minutes Extraction Time

Mass of AVOP (g)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Yield
10	0.75	30	1	8.06	1.94		
		30	2	7.98	2.02	1.98	19.8
10	1.00	30	1	8.46	1.54		
		30	2	8.42	1.58	1.56	15.6
10	1.59	30	1	8.43	1.57		
		30	2	8.48	1.52	1.55	15.5
10	2.00	30	1	8.52	1.48		
		30	2	8.48	1.52	1.50	15.0

Table 4.25: Yields of Oils from Direct Leaching Using N-Hexane at Varying Particle Size and 60 Minutes Extraction Time

Mass of AVOP (g)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Yield
10	0.75	60	1	7.82	2.18		
		60	2	7.92	2.08	2.13	21.3
10	1.00	60	1	8.26	1.74		
		60	2	8.32	1.68	1.71	17.1
10	1.59	60	1	8.29	1.71		
		60	2	8.31	1.69	1.70	17.0
10	2.00	60	1	8.41	1.59		
		60	2	8.37	1.63	1.61	16.1

Table 4.26: Yields of Oils from Indirect Leaching Using N–Hexane at Varying Particle Size and 90 Minutes Extraction Time

Mass of AVOP (g)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted	% Yie
10	0.75	90	1	7.96	2.31		
		90	2	7.72	2.28	2.29	22
10	1.00	90	1	8.07	1.93		
		90	2	8.10	1.90	1.92	19
10	1.59	90	1	8.11	1.89		
		90	2	8.14	1.86	1.88	18
10	2.00	90	1	8.22	1.76		
		90	2	8.45	1.75	1.77	17

Table 4.27: Yields of Oils from Direct Leaching Using N-Hexane at Varying Particle Size and 120 Minutes Extraction Time.

Mass of AVOP (g)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass Of Oil Extracted (g)	% Yield
10	0.75	120	1	7.60	2.40		
		120	2	7.62	2.38	2.39	23.9
10	1.00	120	1	7.80	2.20		
		120	2	7.78	2.22	2.21	22.1
10	1.59	120	1	8.00	2.00		
		120	2	8.00	2.00	2.00	20.0
10	2.00	120	1	8.04	1.96		
		120	2	8.02	1.98	1.97	19.7

Table 4.28: Yields of Oils from Direct Leaching Using N-Hexane at Varying Particle Size and 150 Minutes Extraction Time.

Mass of AVOP (g)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass Of Oil Extracted (g)	% Yield
10	0.75	150	1	7.60	2.40		
		150	2	7.62	2.38	2.39	23.9
10	1.00	150	1	7.80	2.20		
		150	2	7.78	2.22	2.21	22.1
10	1.59	150	1	8.00	2.00		
		150	2	8.00	2.00	2.00	20.0
10	2.00	150	1	8.04	1.96		
		150	2	8.02	1.98	1.97	19.7

Table 4.29: Yields of Oils from Direct Leaching Using N-Hexane at Varying Particle Size and 180 Minutes Extraction Time.

Mass of AVOP (g)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass Of Oil Extracted (g)	% Yield
10	0.75	180	1	7.60	2.40		
		180	2	7.62	2.38	2.39	23.9
10	1.00	180	1	7.80	2.20		
		180	2	7.78	2.22	2.21	22.1
10	1.59	180	1	8.00	2.00		
		180	2	8.00	2.00	2.00	20.0
10	2.00	180	1	8.04	1.96		
		180	2	8.02	1.98	1.97	19.7

Table 4.30: Yields of Oils from Direct Leaching Using Anhydrous Ethanol at Varying Particle Size and 30 Minutes Extraction Time

Mass of AVOP (g)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass Of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Yi
10	0.75	30	1	6.24	3.76		
		30	2	6.21	3.79	3.78	37
10	1.00	30	1	6.73	3.27		
		30	2	6.77	3.23	3.25	32
10	1.59	30	1	6.82	3.18		
		30	2	6.78	3.22	3.20	32
10	2.00	30	1	6.91	3.09		
		30	2	7.03	2.97	3.03	30

Table 4.31: Yields of Oils from Direct Leaching Using Anhydrous Ethanol at Varying Particle Size and 60 Minute Extraction Time.

Mass of AVOP (g)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Yield
10	0.75	60	1	6.03	3.97		
		60	2	5.98	4.02	4.00	40.0
10	1.00	60	1	6.53	3.47		
		60	2	6.57	3.43	3.45	34.5
10	1.59	60	1	6.71	3.29		
		60	2	6.68	3.32	3.31	33.1
10	2.00	60	1	6.82	3.18		
		60	2	6.76	3.24	3.21	32.1

Table 4.32: Yields of Oils from Direct Leaching Using Anhydrous Ethanol at Varying Particle Size and 90 Minutes Extraction Time

Mass of AVOP (g)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Yield
10	0.75	90	1	5.83	4.17		
		90	2	5.89	4.11	4.14	41.4
10	1.00	90	1	6.36	3.64		
		90	2	6.41	6.59	3.62	36.2
10	1.59	90	1	6.63	3.37		
		90	2	6.58	3.42	3.40	34.0
10	2.00	90	1	6.64	3.36		
		90	2	6.61	3.39	3.38	33.8

Table 4.33: Yields of Oils from Direct Leaching Using Anhydrous Ethanol at Varying Particle Size and 120 Minutes Extraction Time

Mass of AVOP (g)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Yield
10	0.75	120	1	5.78	4.22		
		120	2	5.82	4.18	4.20	42.0
10	1.00	120	1	6.30	3.70		
		120	2	6.28	3.72	3.71	37.1
10	1.59	120	1	6.42	3.58		
		120	2	6.42	3.58	3.58	35.8
10	2.00	120	1	6.53	3.47		
		120	2	6.51	3.49	3.48	34.8

Table 4.34: Yields of Oils from Direct Leaching Using Anhydrous Ethanol at Varying Particle Size and 150 Minutes Extraction Time

Mass of AVOP (g)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Yield
10	0.75	150	1	5.78	4.22		
		150	2	5.82	4.18	4.20	42.0
10	1.00	150	1	6.30	3.70		
		150	2	6.28	3.72	3.71	37.1
10	1.59	150	1	6.42	3.58		
		150	2	6.42	3.58	3.58	35.8
10	2.00	150	1	6.53	3.47		
		150	2	6.51	3.49	3.48	34.8

Table 4.35: Yields of Oils from Direct Leaching Using Anhydrous Ethanol at Varying Particle Size and 180 Minutes Extraction Time

Mass of AVOP (g)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Yield
10	0.75	180	1	5.78	4.22		
		180	2	5.82	4.18	4.20	42.0
10	1.00	180	1	6.30	3.70		
		180	2	6.28	3.72	3.71	37.1
10	1.59	180	1	6.42	3.58		
		180	2	6.42	3.58	3.58	35.8
10	2.00	180	1	6.53	3.47		
		180	2	6.51	3.49	3.48	34.8

Table 4.36: Summary of Yields of Oils from Direct Leaching at Varying Extraction Time, 0.75 mm Particle Size (Optimum Particle Size) and Two Solvents

Mass of AVOP (g)	Solvent	Boiling Point (^0C)	Particle Size (mm)	Time (mins)	No. of runs	Mass of AVOP after Extraction and Drying (g)	Mass of Oil Extracted (g)	Average Mass of Oil Extracted (g)	% Yield
10	n-hexane	60	0.75	30	1	8.06	1.94		
				30	2	7.98	2.02	1.98	19.8
10	n-hexane	60	0.75	60	1	7.82	2.1		
				60	2	7.92	2.08	2.13	21.3
10	n-hexane	60	0.75	90	1	7.69	2.31		
				90	2	7.72	2.28	2.29	22.9
10	n-hexane	60	0.75	120	1	7.60	2.40		
				120	2	7.62	2.38	2.39	23.9
10	Anhydrous ethanol	78	0.75	30	1	6.24	3.76		
				30	2	6.21	3.79	3.78	37.8
10	Anhydrous ethanol	78	0.75	60	1	6.03	3.97		
				60	2	5.98	4.02	4.00	40.0
10	Anhydrous ethanol	78	0.75	90	1	5.83	4.17		
				90	2	5.89	4.11	4.14	41.4
10	Anhydrous ethanol	78	0.75	120	1	5.78	4.22		
				120	2	5.82	4.18	4.20	42.0

4.3 Results of Physicochemical Properties of Crude and Refined AVOP Oils

The physicochemical property of both crude and refined oils is given in Table 4.37.

Table 4.37: Physicochemical Properties of the Crude and Refined AVOP Oils.

Properties	Crude	Refined
Saponification value	198	196.4
Iodine value	72.4	67.7
Peroxide value	3.07	1.27
Free fatty acid value	0.08729	0.0872
Acid value	6.8943	5.653
Specific gravity	0.9162	0.90261
Refractive index	1,465	1,600
Viscosity (cp)	180.43	181.180
Boiling point (^{0}C)	89 – 90	89

Table 4.38 gives the physicochemical properties of similar oils such as Olive oil used in similar applications with reference to organizations such as the American Oil Chemists' Society (AOCS) and the International Organization for Standardization (ISO).

Table 4.38: Physicochemical Properties of Refined Olive Oil

Properties	Value
Saponification value	177 – 198
Iodine value	75 – 82
Peroxide value	1.0 – 2.0
Free fatty acid value	0.15 – 0.25
Acid value	1 – 7
Specific gravity	0.914 – 0.918
Refractive index	1.465

Source: ISO/TR 21092, 210, ISO 212 and AOCS.

4.4 Results of Experimental Design: Factorial Design

The results of the factional design performed on the experimental data is given in Tables 4.39 and 4.40 for the factors varied.

Table 4.39: Factorial Analysis Table for Yield Description

No. of Run	X_1	X_2	X_3	Mean Value of Yield (g)	Percentage Yield %	Rate of Yield/Extraction (%/min)
1	30	0.75	SD	2.80	28.0	0.93
2	30	0.75	DL	3.78	37.8	1.26
3	30	0.75	IL	1.46	14.6	0.49
4	60	1.00	SD	3.80	38.0	0.63
5	60	1.00	DL	3.45	34.5	0.58
6	60	1.00	IL	1.61	16.1	0.27
7	90	1.59	SD	4.18	41.8	0.46
8	90	1.59	DL	3.40	34.0	0.38
9	90	1.59	IL	1.72	17.2	0.19
10	120	2.00	SD	5.98	59.8	0.49
11	120	2.00	DL	3.48	34.8	0.29
12	120	2.00	IL	1.71	17.1	0.14
13	150	0.75	SD	5.98	59.8	0.39
14	150	0.75	DL	3.48	34.8	0.23
15	150	0.75	IL	1.71	17.1	0.11
16	180	1.00	SD	5.98	59.8	0.33
17	180	1.00	DL	3.48	34.8	0.19
18	180	1.00	IL	1.71	17.1	0.10

Table 4.40: Results Interpretation

S/N	Factors fixed/value	Factors varied	Percentage yield (%)
1	Particle size/2.00 mm	Production method (SD)	59.8
2	Particle size/0.75 mm Production/S.D Method/IL	Extraction time	37.8
3	Extraction time/120 mm Production/S.D Method/D.L	Particle size	17.2

CHAPTER FIVE

5.0 DISCUSSION OF RESULTS

5.1 Steam Distillation of Avocado Apple's Pericarp

In Table 4.1, the yields of oils at varying steam heating rates are given. At the extraction time of 120 min. and 1000 ml of water, the maximum oil yield was 35.3%. From the results in Table 4.1, at a higher time, there was a corresponding increase in yields of oils until a particular extraction time when there was no significant increase in the yield of oil extracted. From the result in the table, it was seen that at the temperature of production and with higher extortion time there was increase in the yield of oils obtained.

At 1250 ml of water for steam generation, the steam heating rate increased at the corresponding time interval and hence the volume of steam. However, the yield of oil (37.4%) was still maximum at 120 min. extraction time and therefore not necessary to increase the extraction time beyond 120 min. which was in accordance with literature (Table 4.2).

Table 4.3 gives the yields of oils at varying steam heating rates and 1500 ml of water. From the table, maximum oil yield was achieved at 120 min. extraction time and this was 38.7%. The volume of steam used over the time increased progressively due to the volume of water used. Similarly, there was a corresponding increase in oil yield with time (Table 4.3).

Figure 4.1 in Appendix B1 shows the steam-sample ratio per time with steam generated from various volumes of water at varying steam heating rates. From the figure, it could be seen that there are time lags. The time lags represented the period the steam generated from the various volumes of water in the still was utilized to heat up the still and the sample. The time lag was dependent on the rate of steam supply to the sample, the weight of sample and the sample particle size. It was observed to be between 12-18 min.

Table 4.4 shows the optimal oil yields of 38.0% from 10 grams of avocado's pericarp at 120 min. extraction time and 1000 ml of water for steam generation. The optimal steam heating rate to the sample was 4.98 ml/min. The result from the table showed that the percentage oil yields were time dependent which was in accordance with literature value (Table 4.4).

In Table 4.5, at 1250 ml of water used for steam generation, the highest yield of oils was 39.6% at a steam rate of 4.94 ml/min. This was at the extraction time of 120 min. From the result in the table, increase in extraction time beyond 120 min. to 180 min. had no effect on the oil yield (Table 4.5)

From Table 4.6, oil yield of 37.5% was obtained from 10g of avocado's pericarp. This was at 120 min. and 1500 ml of water for steam generation at a rate of 5.14 ml/min. This was the optimal oil yield at the highest time for the experiment. This was in accordance with literature (Table 4.6).

Similarly, Figure 4.2 in Appendix B2 shows the steam-sample ratio per time with steam generated from various volumes of water at varying steam heating rates. From the figure, it was evident that time lags exist. This was between 12-18 min. and it was dependent on the rate of steam supply to the sample, the weight of sample and sample particle size. The time lags represented the period the steam generated in the still was used to heat up the still and the sample.

In Table 4.7, the yields of oils at varying steam heating rates are presented. At the extraction time of 120 min. and 308.9 ml of steam, the maximum oil yields was 59.8%. From the table, it could be seen that yields of oils increased with time to the maximum depending on the steam supply rate.

Table 4.8 gives oil yields from 1250 ml of water at varying steam heating rates. From the table, the steam rate of 3.86 ml/min gave the optimal oil yield of 47.2% and at the extraction time of 120 min. which conforms to literature (Table 4.8)

In Table 4.9, maximum oil yield was achieved at 120 min. extraction time and this was 58.8%. The volume of steam used over the time increased progressively due to the volume of water used. Similarly, there was a corresponding increased in oil yields with time up till the maximum oil yield at 120 min. This conforms to literature value on the influence of time on extraction.

Figure 4.3 in Appendix B3 shows the steam-sample ratio per time with steam generated from various volumes of water at varying steam heating rates. The figure showed that time lags existed for the varying steam heating rates. This was between 10-16 min. and the time lags were dependent on the rate of steam supply to the sample, sample weight and sample particle size.

From Figures 4.1, 4.2, and 4.3, it could be seen that the rate at which steam passed through the sample though approximately linear with time to a particular extent, may deviate from linearity to curve depending on the packing density (loading capacity) of the sample on the plate.

In Table 4.10, the yields of oils at the optimum extraction time (120 min.) varying steam heating rates and varying volumes of water are presented. From the table, the maximum oil yields of 59.8% was achieved from 1000 ml of water at a steam rate of 2.57 ml/min which represented the moderate heating rate; 2.06 ml/min and 4.94 ml/min representing low and high heating rates respectively with oil yields of 35.3% and 39.6%(Table 4.10).

5.2 Indirect Leaching of Avocados Apple's Pericarp

In Table 4.11, the yields of oils at varying particle sizes and extraction time of 30 min. is given. At the extraction time, the maximum oil yield was 14.6% which was from particle size of 0.75 mm. From the results in Table 4.11, the yields of oils decreased with increasing particle size at the prevailing extraction time (30 min) and temperature (60^0C).

At extraction time of 60 min, the yields of oils decreased with increasing particles size. However, the yield of oil (18.6%) was still maximum at the lowest particle size (0.75 mm) which was in accordance with literature value (Table 4.12).

Table 4.13 gives the yields of oils at varying particle size at 60^0C, boiling point of the solvent and extraction time of 90 min. From the table, maximum oil yield was achieved from 0.75 mm particle size and this was 20.2%. The yields of oils over the extraction time and particle size decreased with increase in particle size at a constant time of 90 min. for each extraction (Table 4.13).

Table 4.14 gives the optimal oil yield of 21.2% from 10 gram of avocado's pericarp size of 0.75 mm at the 120 min. extraction time. The result from the table showed that the yield of oils was time and particle size dependent which was in accordance with literature value (Table 4.14).

In Table 4.15, the yields of oils at varying particle sizes and extraction time of 150 min. is given. From the results in the table, it was seen that increasing the extraction time beyond 120 min. had no increase in the yield on oil extracted since the same quantity or oil yields was obtained at both extraction times of 120 min. and 150 min. Hence, it was therefore not necessary to increase the extraction time of beyond 120 min. The optimal oil yield for the extraction time of 150 min. was 21.2% and this was from 0.75 mm particle size.

Similarly, Table 4.16 gives the yields of oils at varying particle size at 60^0C, boiling point of n-hexane and extraction time of 180 min. From the table, maximum oil yield was achieved from 0.75 mm particle size and this was 20.2%. The result from the table showed that the yield of oils was time and particle size dependent which was in accordance with literature value. However, from the results in Tables 4.14 and 4.15 it was seen that increasing the extraction time beyond 120 min. had no effect on the yield of oil extracted since the same oil yields were obtained at all extraction times of 120 min., 150 min. and 180 min. Hence, it was unnecessary to increase the extraction time beyond 120 min.

In Table 4.17, the yields of oils at varying particle sizes and extraction time of 30 min. is given. An optimal oil yield of 32.1% was achieved from 0.75 mm particle size using anhydrous ethanol (bp 78^0C) at the extraction time of 30 min. The table revealed that oil yield decreased with increased particle size at the prevailing temperature of extraction and time.

Table 4.18 gives the yields of oils from 10 gram of AVOP at 78^{0}C; boiling point of solvent at varying particle size and extraction time of 60 min. From the result in the table, it was revealed that the particle size of 0.75 mm gave the highest oil output (34.0%) at the extraction time. More so, the table revealed that oil yields were dependent on the size of the AVOP particles at the thermodynamic conditions employed.

In Table 4.19, the yields of oils at varying particle size and 90 min. extraction time from 10 gram of AVOP are given. At the extraction time and 0.75 mm particle size of AVOP, the maximum oil yield was 35.0%. From the result in Table 4.19, at a lower particle size, the yields of oils increased at the time of extraction.

Table 4.20 gives the yields of oils at varying particle size, boiling point of solvent, 78^{0}C, and 120 min. extraction time. From the table, the highest yield of oil was achieved from 0.75 mm particle size and this was 35.9%. The oil yields decreased with increase in the size of the particle at the extraction time. This was in accordance with literature.

In Table 4.21, the yields of oils at varying particle sizes and extraction time of 150 min. is given. From the results in the table, it was seen that increasing the extraction time beyond 120 min. had no increase in the yield on oil extracted since the same yields of oils was obtained at both extraction times of 120 min. and 150 min. Thus, it was not necessary to increase the extraction time beyond 120 min. The optimal oil yield for the extraction time of 150 min. was 35.9% and this was from particle size of 0.75 mm.

Table 4.22 gives the yields of oils at varying particle size, boiling point of anhydrous ethanol (bp 78^{0}C) and extraction time of 180 min. From the table, the maximum oil yield was achieved from 0.75 mm particle size and this was 35.9%. The result showed that the yields of oils were time and particle size dependent which was in accordance with literature value. However, the results in Tables 4.20, 4.21 and 4.22 showed that increasing the extraction time beyond 120 min. had no effect on the yields of oils extracted since the same oil yields were obtained at all extraction times. Hence, it was unnecessary to increase the extraction time beyond 120 min.

In Table 4.23, the yields of oils at varying extraction time, 0.75 mm particle size (optimum particle size) and two solvents are presented. From the table, the maximum oil yield for the indirect leaching process was achieved at 120 min. extraction time using anhydrous ethanol at its boiling point of 78^{0}C. This was 35.9%. However, at a similar extraction time (120 min.) using n–hexane (bp 60^{0}C) the oil yield was 21.2% (Table 4.23).

5.3 Direct Leaching of Avocado Apple's Pericarp

In Table 4.24, the yields of oils at varying particle sizes and extraction time of 30 min. is given. At the extraction time, the maximum oil yield was 19.8%. This was from 0.75 mm particle size. From the results in the table, it was seen that the smaller the particle size, the higher the yield of oil obtained which was in accordance with literature.

At the extraction time of 60 min., the yields of oils decreased with increased particle size. 21.3% yield of oil was the maximum at the smallest particle size (0.75

mm) which was in accordance with literature. The oil yields decreased with increasing particle size (Table 4.25).

Table 4.26 gives the yields of oils at varying particle size, boiling point of solvent (60^0C) and extraction time of 90 min. From the table, the maximum oil yield was achieved from 0.75 mm particle size and this was 22.9%. The yields of oils over the extraction time and particle size decreased with increased in particle size at the constant time of 90 min. for each extraction (Table 4.26).

Table 4.27 gives the optimal oil yield of 23.9% from 10g of avocado's pericarp size of 0.75 mm. This was at 120 min. extraction time. The results from the table revealed that the yield of oils was time and particle size dependent which agreed with literature values (Table 4.27).

Table 4.28 gives the optimal oil yield of 23.9% from 10 gram of avocado's apple pericarps at particle size of 0.75 mm. The extraction time was 150 min. The results from the table revealed that the yield of oils was time and particle size dependent which agreed with literature values (Table 4.28). However, from the results in Tables 4.27 and 4.28, it was seen that increasing the extraction time beyond 120 min. had no effect on the yields of oils extracted since almost the same oil yields was obtained at both extraction times of 120 min. and 150 min. Thus, it was unnecessary to increase the extraction time beyond 120 min.

In Table 4.29, the yields of oils at varying particle sizes and extraction time of 180 min. is presented. From the table, the maximum oil yield was achieved from 0.75 mm particle size and this was 23.9%. The result showed that the yields of oils were time and particle size dependent which was in accordance with literature value. However, the results in Tables 4.27, 4.28 and 4.29 showed that increasing the extraction time beyond 120 min. had no effect on the yields of oils extracted since the same quantity of oil were obtained at all extraction times of 120, 150 and 180 min. Hence, the optimum extraction time was 120 min.

In Table 4.30, the yield of oils at varying particle size and extraction time of 30 min. is given. An optimal oil yield of 37.8% was achieved from 0.75 mm avocado's pericarp using anhydrous ethanol (bp 78^0C) at the extraction time of 30 min. The table revealed that oil yield was a function of particle size at the temperature of extraction and time (Table 4.30).

Table 4.31 gives the yields of oils from 10 gram of AVOP at 78^0C; boiling point of solvent at varying particle sizes and extraction time of 60 min. From the results in the table, it was revealed that the maximum oil yield was 40.0% and this was obtained from 0.75 mm particle size of AVOP at the extraction time of 60 min. More so, the table revealed that oil yields were dependent on the size of the particle at the thermodynamic condition employed.

In Table 4.32, the yields of oils at varying particle size and 90 min. extraction time is given. At the extraction time and 0.75 mm particle size, the maximum oil yield was 41.4%. From the results in Table 4.32, at a smaller particle size, the yields of oils increased and decreased with larger particle size at the extraction time which was 90 min. (Table 4.32).

Table 4.33 gives the yields of oils at varying particle size, boiling point of solvent; 78^0C, and 120 min. extraction time. From the table, the highest yield of oil was obtained from 0.75 mm particle size and this was 42.0%. The oil yields decreased with increase in the size of the particle at the time period for extraction. This was in accordance with literature value.

In Table 4.34, the yield of oils at varying particle size and 150 min. extraction time is presented. At the extraction time and 0.75 mm particle size, the maximum oil yield was 42.0%. From the results in the table, at a smaller particle size, the yields of oils increased and decreased with larger particle size at the extraction time of 150 min. However, Tables 4.33 and 4.34 showed that increasing the extraction times beyond 120 min. had no effect on the oil yields since almost the same amount of oil was obtained at both extraction times. Hence, it was unnecessary to increase the extraction time beyond 120 min.

Table 4.35 gives the optimal oil yield of 42.0% from 10 gram of avocado's pericarps of 0.75 mm particle size. The extraction time was 180 min. The results from the table revealed that the yields were time and particle size dependent which agreed with literature values (Table 4.35). However, the results in Tables 4.33, 4.34 and 4.35 showed that increasing the extraction time beyond 120 min. had no effect on the yields of oils extracted.

In Table 4.36, the yields of oils at varying extraction time, 0.75 mm particle size (optimal particle size) and two solvents are presented. From the table, the maximum oil yield for the direct leaching process was achieved at 120 min. extraction time using anhydrous ethanol at its boiling point of 78^0C. This was 42.0%. However, at a similar extraction time (120 min.), using n-hexane (bp 60^0C), the oil yield was 23.9% (Table 4.36).

5.4 Comparison of Results of Steam Distillation and Leaching (Direct and Indirect Extraction)

From the table of results on steam distillation and leaching (direct and indirect extraction) at varying particle sizes and 30 min. extraction time, the maximum oil yield was 37.8% and this was from particle size of 0.75 mm and direct leaching using anhydrous ethanol (bp 78^{0}C) as solvent. From the results in all the tables, it was revealed that the optimal oil yield of 28.0% (moderate) was achieved from steam distillation at 30 min. extraction time. Similarly, indirect leaching using n-hexane and anhydrous ethanol gave optimal oil yields of 14.6% and 32.1% respectively while the direct leaching using the solvents gave optimal oil yields of 19.8% and 37.8% respectively at the extraction time of 30 min. Hence, from the results in the tables, the maximum oil yield of 37.8% was obtained for the extraction time (30 min.), particle size (0.75 mm) and production method of direct leaching.

At extraction time of 60 min., a comparison of the yields of oils from steam distillation and leaching (direct and indirect extraction) methods showed that the yield of oil (40.0%) was maximum at the lowest particle size (0.75 mm) using direct leaching and anhydrous ethanol as the solvent. This was at the solvent's boiling point (78^{0}C). From the results in the tables, the optimal oil yields for steam distillation; moderate steam heating rate was 38.0% at particle size of 2.00 mm. This was the optimal particle size for the steam distillation method. The tables also revealed that, in the indirect extraction method, the highest oil yield of 34.0% and 18.6% were achieved using anhydrous ethanol and n-hexane as solvents respectively. This trend was also prevalent with the direct leaching method where the oil yields were 40.0% and 21.3% for anhydrous ethanol and n-hexane respectively. This was from particle size of 0.75 mm and extraction time of 60 min. Thus, from the results in the tables, the maximum oil yield at the extraction time was 40.0%. This was from 0.75 mm particle size and production method of direct leaching (extraction) using anhydrous ethanol.

Similarly, at extraction time of 90 min. and varying particle size, the results in all the tables showed that the maximum oil yield of 50.2% was obtained from steam distillation method of production. This was from particle size of 2.00 mm and moderate steam heating rate. From the results in the tables, a comparison of the yields of oils from steam distillation and leaching (direct and indirect extraction) revealed that optimal oil yields of 20.2% and 35.0% was achieved from indirect extraction (leaching) using n-hexane and anhydrous ethanol respectively while 22.9% and 41.4% was achieved from direct extraction (leaching) using n-hexane and anhydrous ethanol respectively. These were at particle sizes of 0.75 mm. Hence, the maximum oil yield for 90 min. extraction time was 50.2%, from particle size of 2.00 mm. The production method was steam distillation.

At the optimal extraction time for research (120 min.), comparison of oil yields of AVOP from steam distillation and leaching (direct and indirect extraction) methods at varying particle sizes showed that the maximum oil yield was 59.8%. This was from particle size of 2.00 mm and the production method for this oil yield was steam distillation at moderate steam heating rate. From the results in all the tables, at 120 min. extraction time, it was revealed that the optimal oil yields of 42.0% and 23.9% was achieved from direct leaching (extraction) at the boiling points of anhydrous ethanol (78^0C) and n-hexane (60^0C) respectively. Similarly, indirect extraction (leaching) gave optimal oil yields of 35.9% and 21.2% for anhydrous ethanol and n-hexane at their boiling points respectively. The yields of oils for both direct and indirect extraction (leaching) were achieved from particle size of 0.75 mm at the extraction time of 120 min.

5.5 Discussion of Results of Physicochemical Properties of Crude and Refined AVOP Oils

Table 4.37 gives the results of the analysis on the crude and refined AVOP oil samples, that is, the physicochemical properties. From the table, the values of properties fall within the range of values for oils used in similar applications with reference to organizations such as the American Oil Chemists' Society (AOCS) and the International Organization for Standardization (ISO).

The table also gives the values of the physicochemical properties of the refined AVOP oil which are most commonly used to establish the identity of oils. Each of the property was chosen to measure a specific characteristic of the oil.

From Tables 4.37, the properties of the oil such as saponification value, iodine value, peroxide value, free fatty value and acid value are mostly used to specify the characteristic of the oil. The others are empirical in nature though they also give useful guidance in identifying the oil.

Comparison of results of crude and refined AVOP oil revealed that the difference in saponification value between the crude and refined oils was less than

1.0 (0.81%). This is attributable to the fact that the oil's many natural constituents are still present and hence, little lipase activity.

Also, from the table, a 65% level decrease in iodine value between the crude and refined oils implied that less amount of hydrogen would be required in converting the unsaturated components of the oil into saturated oil for industrial use.

Similarly, the lower peroxide value (58.63%) of the refined oil implied that the oil cannot be easily decomposed and neither can it become rancid as a result of the presence of triglyceride esters of the oil to form peroxide when compared to the crude oil.

An acid value of less than 10 enhances the stability of most oils and both values fall within this range. Thus, the refining quality of the oil is enhanced.

The low value of the FFA for both the crude and refined oils implied that the oil contains acid that are uncombined with glycerol and thus, do not easily decompose nor become rancid, indicating little lipase activity.

Similarly, Table 4.38 gives the physicochemical properties of olive oil which is used in similar industrial application as the refined AVOP oil with reference to the relevant standards organizations (AOCS and ISO).

Comparison of Tables 4.37 and 4.38 showed that the physicochemical properties of the AVOP oil tested fall within the range of oil properties for olive oil as obtained by standard organizations (ISO and AOCS) and hence, could be used for similar industrial applications in cosmetics and pharmaceutical drugs production.

5.6 Discussion of Results of Experimental Design by Factorial Design

The results obtained from the experiments provided the basis for factorial analysis for the selective comparison of the three factors varied. The results provides for the effective recovery of oil from the pericarp of the avocado apple. The result showed that the production of oil from the pericarp of the avocado apple is feasible for as high as 60% oil recovery. It thus showed that the production method had more influence followed by the extraction time and the particle size of the AVOP used.

The factional design method provided a qualitative determination of the relationship between the variables that describes the response of one factor to the yield when the other factors are fixed. It provided the efficient variable conditions for maximum oil recovery for the system under study.

CHAPTER SIX

6.0 CONCLUSIONS AND RECOMMENDATIONS

6.1 Conclusions

The conclusions emerging from this work include the following:

1. That high quality oil could be obtained from the pericarp of the avocado apple which hitherto was discarded as waste.
2. A steam distillation process at steam rate of 2.57 ml/min (moderate) gave the highest oil yield of 59.8% from the pericarp at extraction time of 120 min. The effect of particle size variation was not significant on the steam distillation process as smaller particle sizes (<2.00 mm) formed lumps on percolation of the steam through them making extraction difficult.
3. The rate of extraction was known to proceed favorably with increasing time and decreasing particle size at the temperature of extraction. The results of this work using indirect and direct extraction (leaching) where supportive of that fact.
4. The yields of oils from indirect and direct leaching using n-hexane and anhydrous ethanol increased progressively with decreased particles size and increasing extraction time at the boiling points of the solvents. Also, the affinity of the sample materials toward either of the solvent used showed that anhydrous ethanol was more useful as the leaching solvent based on the yields of oils obtained than n-hexane for this particular work
5. Comparison of the results of indirect and direct leaching showed that, at a similar extraction time (120 min.), particle size (0.75 mm) and boiling points of

the solvents (n-hexane 60^0C, anhydrous ethanol 78^0C), the optimum yields of oils of 21.2% and 23.9% for indirect and direct leaching using n-hexane and 35.9% and 42.0% for indirect and direct leaching using anhydrous ethanol were obtained. The physicochemical properties of both the crude and refined oils were found comparable with oils of similar structural constituents used for industrial applications.

6. Additionally, the design of a steam distillation apparatus or equipment for the production of the oil on a pilot-scale basis showed that such a venture would be economically profitable, with adequate return on investment of 78.88%.

6.2 Recommendations

Based on the results obtained and analysis carried out, it was obvious that the objective of the research thesis was achieved. However, other areas needing further research include:

- Investigating the potential for introducing the supercritical fluid extraction technology into the production of oil from the pericarp of the avocado and other fruits while still comparing the process with steam distillation and extraction to ascertain the best alternative method.
- Investigating the effect of the quality of the fruit on the oil quality and yield.
- Investigating the oxidative stability of the oil obtained and the kinetics of photo-oxidation of the oil.
- Investigating the antiviral and insecticidal properties (Active Ingredient) of the fruit pericarp with reference to the oil produced and finally,
- Investigating into the comparative production of oil from the seed of the avocado apple using extraction, steam distillation and supercritical fluid extraction methods.

Finally, research should be undertaking to use as much as possible waste product of agricultural origins for other beneficial purposes of which this research thesis adequately utilized thereby converting waste to wealth; that is, creating higher earnings from land use, as well as creating job opportunities both on the land and in manufacturing.

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APPENDIX A

i. Steam-Sample ratio

$$\left({ml}/{g} \right) = \frac{\text{Volume of steam used}}{\text{Mass of sample after extraction and drying}}$$

ii. Optimum steam supply rate per gram sample or steam required to transport the oil (steam utilization) is obtained from calculation of the gradient in Figure 4.1-4.3 thus:

$$\text{Slope or gradient} = \frac{y_2 - y_1}{x_2 - x_1} = \frac{98 - 12}{120 - 30} = \frac{86}{90}$$

$$= 0.9556 ml/\min \left(2.654 \times 10^{-7} \, {m^3}/{s} \right)$$

iii Steam heating rate (ml/min) = $\frac{\text{Volume of steam used}}{\text{Extraction Time}}$

iv % Yield $= \frac{\text{Mass of oil extracted}}{\text{Total initial mass of sample}} \times 100\%$

v Mass of oil extracted = Initial mass of sample – Mass of sample after Extraction and drying.

vi Saponification value

$= \frac{\text{Number of KOH in 1ml of 0.5KOH x Blanck titration sample}}{\text{Weight of sample}}$

vii Iodine value $= \frac{(B - S) \times N \times 126.9}{\text{Weight of sample}}$

Where B – Blank titration; S – sample titration

N – Normality of thiosulphate solution

126.9 – Atomic weight of iodine

viii Free fatty acid value % = $(VxMxN)/100W$

Where N – Normality of NaOH; V – Volume of NaOH; and W – Weight of Oil

ix Peroxide value, PV = $(VxNx100)/G$

where V – Volume of thiosulphate used (ml); N – Normality of thiosulphate solution, and G – Volume of sample (ml).

APPENDIX B1

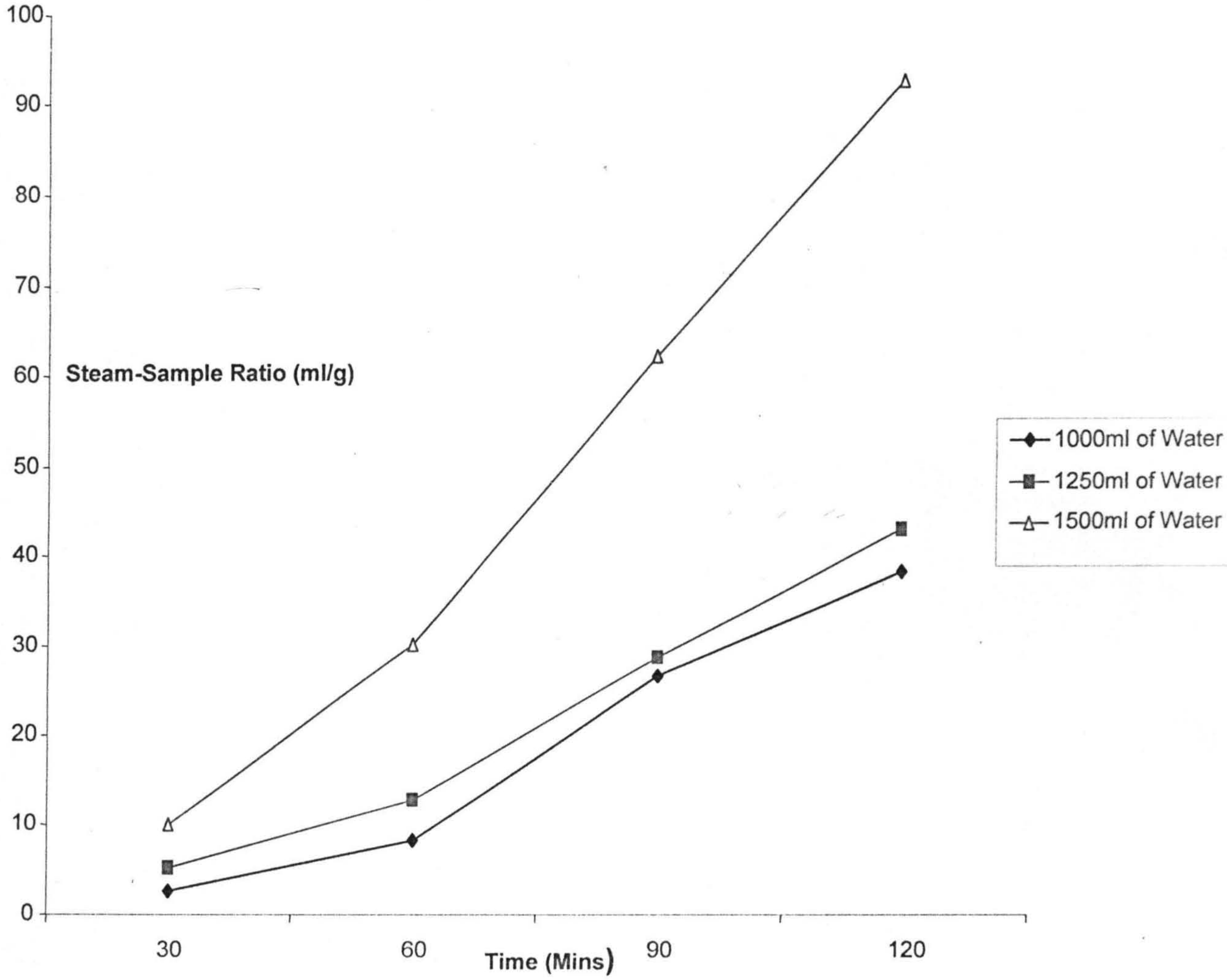

Figure 4.1: Steam-Sample Ratio per Time with Steam Generated from Various Volumes of Water at Varying Steam Heating Rates

APPENDIX B2

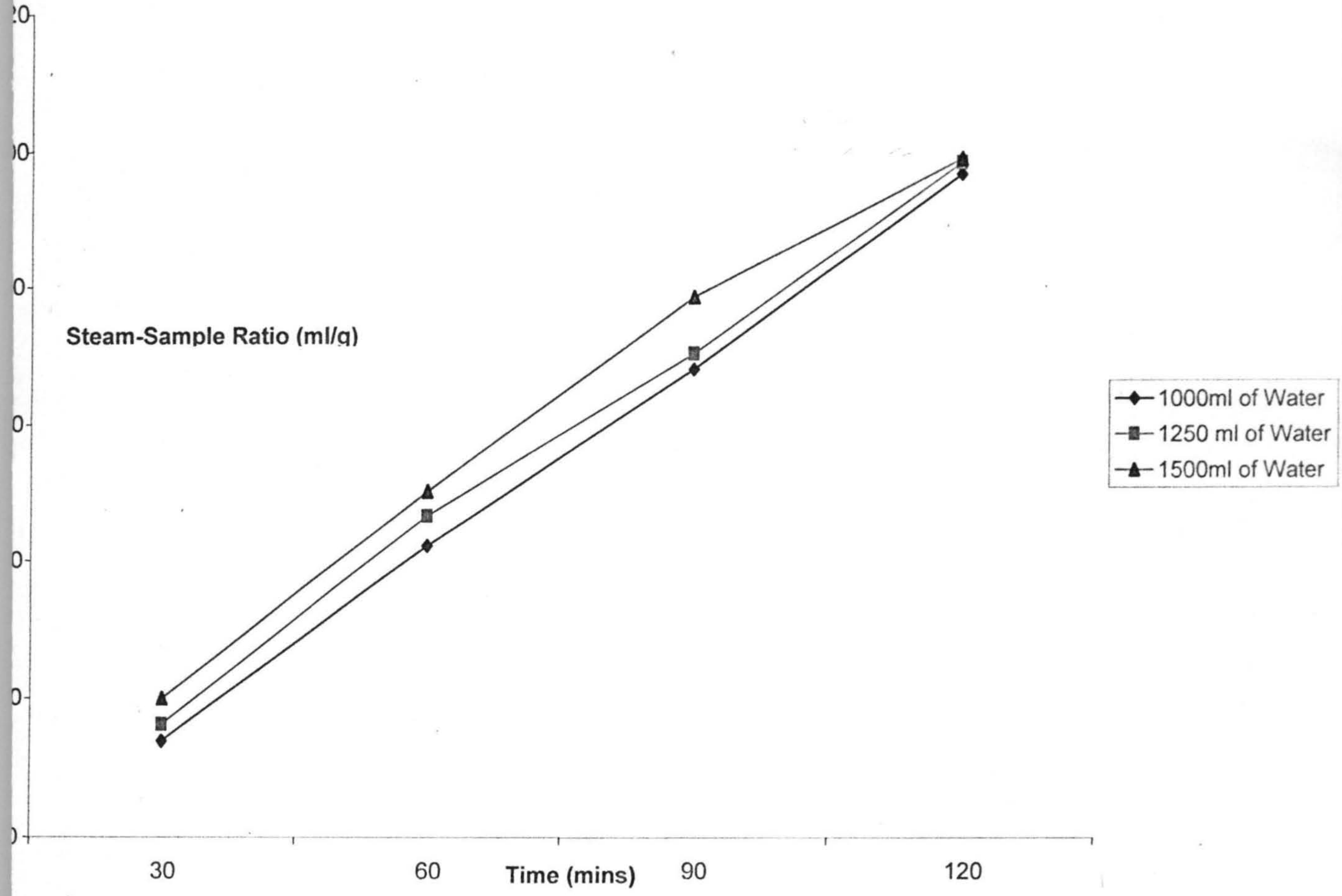

Figure 4.2: Steam-Sample Ratio per Time with Steam Generated from Various Volumes of Water at Varying Steam Heating Rates

APPENDIX B3

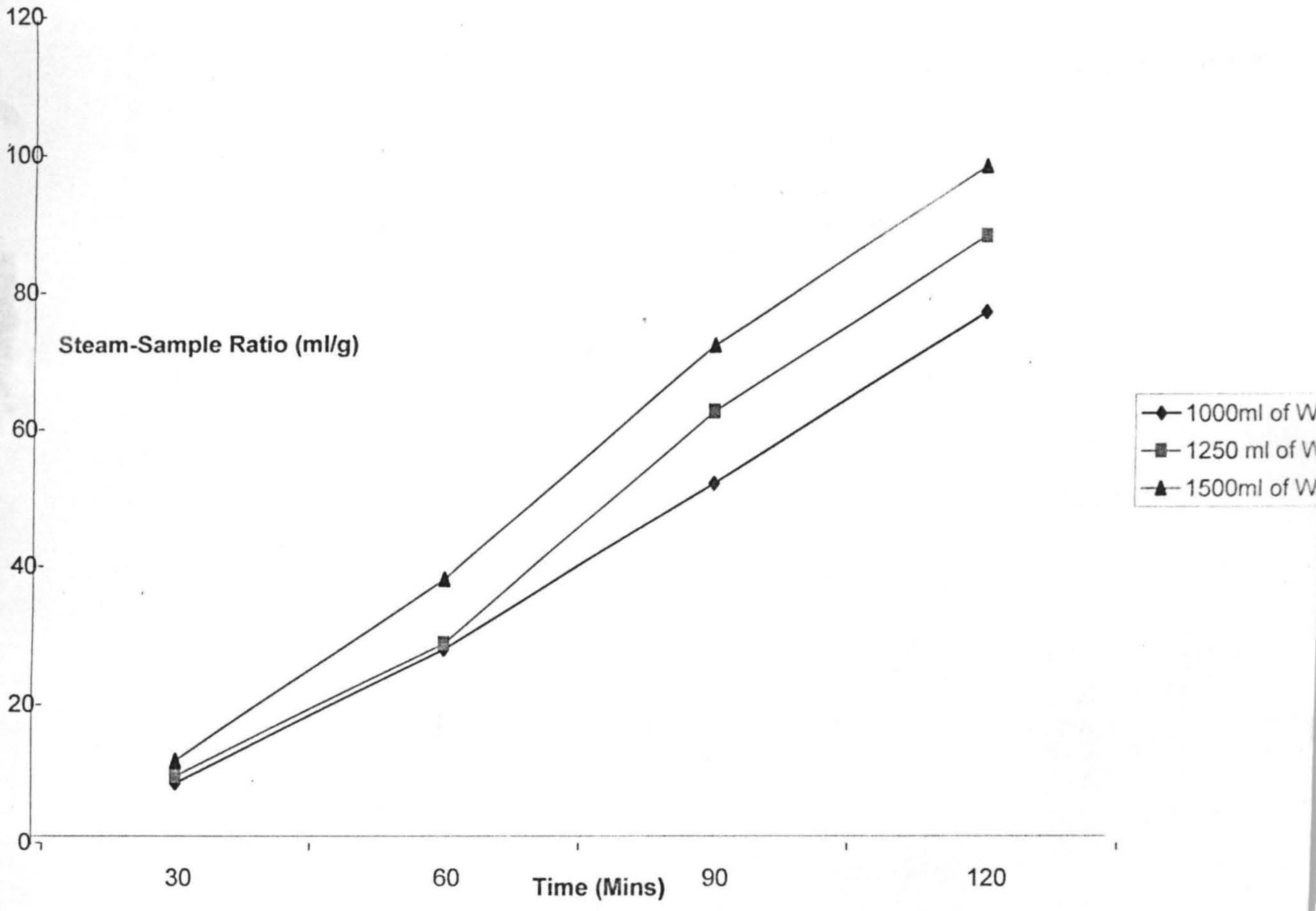

Figure 4.3: Steam-Sample Ratio per Time with Steam Generated from Various Volumes of Water at Varying Steam Heating Rates