A PROJECT REPORT

ON

APPLICATION OF 2 X 2 FULL FACTORIAL DESIGN TO EXTRACTION OF OIL FROM SOYBEAN.

BY

OLADIIPO ABIODUN KOYUMU
(93/4119)

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DEDICATION

This Project work is dedicated to the following, who influence my life and believe in me most:

My beloved Mother (**MRS. SAMURAT A. OLADIIPO QUDUS**) for her love.

My late father **(ALHAJI O. A. OLADIIPO QUDUS)** for sowing the fruit

And

My uncle **(ALHAJI DIMEJI OYELADUN**) for his kindness.

ACKNOWLEDGEMENT

DECLARATION

I **OLADIIPO ABIODUN KOYUMU** declare that this Project Presented for

the Award of Bachelor of Engineering in Chemical Engineering has not been

presented either wholly or partially for any degree elsewhere.

_____   _____

Student   **Date**

CERTIFICATION

This is to certify that this Project **"APPLICATION OF 2 X 2 FULL FACTORIAL DESIGN TO EXTRACTION OF OIL FROM SOYBEAN"** is the original work of **OLADIIPO A. K**. carried out wholly by him under supervision and submitted to the Department of Chemical Engineering, School of Engineering and Engineering Technology, Federal University of Technology, Minna.

_____   _____

Supervisor   **Date**
Mr. Duncan Aloko

_____   _____

Head of Department   **Date**
Dr. J. O. Odigure

_____   _____

External Examiner   **Date.**

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ABSTRACT

The soybean oil was leached using 250ml of hexane at temperature of 65℃ and at constant mass of 5g. The colour of oil obtained varied from golden yellow to deep yellow. As expected the percentage of oil obtained is inversely proportional to the particle size of the sample. For instance in 3 hours time of extraction; 24 16 per cent of oil was extracted when sample size was 0-250μm as opposed to 21.38 per cent of oil when sample size was 710-350μm. Higher percentage of oil was observed when leaching time increased to 6 hours at same: sample mass, temperature and particle size for 3 hours the following percentage were obtained: 25.72 per cent of oil and 24.18 per cent of oil respectively. It was safety concluded that the increase in percentage of oil extracted was not directly proportional to increase in leaching time.

CHAPTER ONE

1.0   INTRODUCTION

Since ancient times humans have known how to remove Oils and fats from their natural sources and make them fits for their own uses.   Animals fats were first consumed as food, but it was not long before they were burned for light and heat. (4).   Obtaining Oils from vegetable sources is of ancient origin, for the natives in the tropical regions of the globe have long been removing these Oils from nuts after drying them in the sun.

The demand for oils and fats is met largely from plant sources with animal fats and marine oils contributing less than 2.5 per cent of total production (6).   At present about 11.7×10^6 of edible vegetable oils, and about $4\ 2 \times 10^6$ t of edible animal fats and oil are produced in the United State each year (4).

The crop, which contributed most significantly to oils and fat market, is the soybean.   Although it has only about 18 percent oil content.   Nevertheless it contributes over 8 millions metric tons of Oil out of 11.7 millions metric tons of oil from other vegetable source annually in United State.   Further details about fat and oil Soybean oil, their classification, composition; uses etc. is presented under literature review. (Chapter two).

1.1   EXTRACTION

Extraction is another key word very much relevant to this work.   It's a method of separating the constituents of a mixture utilizing preferential solubility of one or more components in a second phase commonly the added second phase is a liquid, while the mixture to be separated may be either liquid or solid.

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1.1.1.　　　Liquid/Liquid Extraction.

Liquid/liquid extraction separates the components of homogenous liquid mixture on the basis of differing solubility in another liquid phase. Because it depends on difference in chemical potential, liquid/liquid extraction is more sensitive to chemical type than to molecular size. This makes it complementary to distillation as a separation technique

1.1.2.　　　Liquid/Solid Extraction

Liquid/ Solid extraction may be considered as the dissolving one or more components in a solid matrix by simple solution, or by the formation of a soluble form of chemical reaction. The field can be sub-divided into the following categories. Washing extraction, diffusional extraction and leaching.

- In washing extraction the solid is crushed to break the cell walls, permitting the valuable soluble product to be washed from the matrix.

In diffusional extraction, the soluble product diffuses across the denatured cell walls (no crushing involved) as is washed out of the solid.

Leaching involves the contacting of a liquid and a solid and the imposing of a chemical reaction upon one or more substances in the solid metrix so as to render them soluble. The process may be used either for the production of a concentrated solution of a valuable solid material, or in order to remove an insoluble solid, such as pigment from the soluble material with which it is contaminated.

1.2　　FACTORS AFFECTING EXTRACTION

Extraction Process is influenced by the factors, which are responsible for limiting the extraction rate. The factors are as follows:

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Particle Size: - The leaching process will be favoured by increased surface per unit volume of the solid to be leached and by decreased radial distance that must be transversed within the solids, both of which are favoured by decreased particle size. Fine solids on the other hand cause slow percolation rate, difficult solids separation and possible poor quality of solid product. The basis of an optimum particle is established by these characteristics.

Solvent: - The liquid chosen should be a good selective solvent and its viscosity should be sufficiently low for it to circulate. Generally, a relative pure solvent will be used initially, but as the extraction proceeds the concentration to solute will increase and the rate of extraction will progressively decrease, because of concentration gradient will be reduced, and secondly because the solution will be generally become viscous.

Temperature:- In most cases, the solubility of the material, which is being extracted, will increase with temperature to give a higher rate of extraction. Furthermore, the diffusion coefficient will be expected to increase with rise in temperature and this will also improve the rate of extraction.

Agitation of fluid:- Agitation of the solvent is important because this increase the eddy diffusion and therefore the transfer of materials from the surface of the particles to the bulk of solution.

1.3 Oil Processing

Oil is obtained commercially from oil seeds (Soybean etc) by one of the three basic methods, which can be modified or combined to suit specific conditions. These are: batch hydraulic processing in which oil is expressed by the hydraulic pressure from a mass of oil bearing material, continuous mechanical processing in which the oil bearing material is squeezed through a

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tapering outlet, oil being expressed by the increasing pressure, and solvent extraction in which oil bearing material is taken into solution with a solvent, the solution is separated from insoluble residue and oil recovered from the solution.

Solvent extraction method is going to be used in this work. It will involve counter-current flow of solvent and oil-bearing materials in the extraction vessel. The yield, using this process, is usually higher than that of mechanical method and residue usually contain less than 2 per cent oil.

Solvent extraction plants are becoming more common as the Processing industry now aim to produce meals with minimum oil content at commercially acceptable production level without impairing oil or meal quantity.

For seeds or nuts containing higher oil than soybean it becomes customary to press the material in screw press to remove larger proportion of oil before extraction.

1.4.0.   Aims and Objective.

The aim of this project is to study means of improving solvent extraction method of extracting oil from Soybean by investigating the effect of particle size distribution and extraction time individually and collectively, at constant solvent volume, sample mass and temperature.

Also to determine optimum particle size and optimum time of extraction of oil from Soybean. And to develop mathematical model using 2 x 2 full factional design.

1.4.1.   Scope of the work.

The experiment was carried out on four different Particle sizes (0-250, 250-500-710, 710-850μm) and 3 and 6 hours were used as the extraction time.

Hexane is the only solvent used for the work.

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CHAPTER TWO

2.0. LITERATURE REVIEW

Botanical name	-	Glycine max (L) Merrill
Family name	-	Leguminosae
Synonyms	-	Glycine Soja siets, Soja max (L) Piper.
Common nates	-	Soya bean, Soybean (England); Soja (France); Nong Taw (China); Sojabohn (Germany).

2.1.0.   BOTANICAL DESCRIPTION

Soybean is the annual crop, which is dominant in both the protein and vegetable oil markets. A wide range of types are available with different growth habits and for different lengths of growing season and different latitudes and climates, but mostly restricted to a range from the warmer temperate zones to the cooler or more elevated tropics.

2.1.1.   PLANT

The soybean grown for seed production is a leguminous annual, normally bushy, erect and usually less than 75cm in height, much branched, well developed roots and producing members of small pods containing round usually yellow or black seeds.

2.2.2   ROOTS

The root system is extensive, with a taproot, which may exceed 1.5.m in length, giving rise to laterals branches usually in the 0-30cm horizon. The root has bacterial nodules which fix nitrogen from the soil atmosphere, due to activity

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of bacterial of the Rhizobium genus which lives in symbiosis within the root of legumes.

2.1.3 LEAVES

Soybean leaves are alternate variable in shape, hairy in some varieties, normally trifoliate. The pestioles are long and narrow, the leaflet is ovale or lanceolate. The most common colour is a dark green, but atimes appear tinted with strades of brown, red or blue when growing in the field. Maximum leaf area index values of 5-8 are usually attained at main flowering and reduce to 4-6 at physiological maturity.

2.2.4. FLOWERS

Flowers are born on short racemes originating in leaf axes, each inflorescence bearing up to twenty small, purple or white flowers, which are typically leguminous in shape. The ovary is hairy; stamens is free or joined at the base and stigma captivate. The flowering period can be up to 6 weeks, but it is usually between 3 and 4 weeks.

2.2.5. FRUITS

The fruit is normally a short, hairy pod which can vary from 2 to 10cm in length and 2-4cm in width according to the variety and is usually some shades or brown or black but can have a green, red or purple tint. Pod number per plant can vary from a few dozen to several hundred, although the number is basically a varietal characteristics, it is heavily dependent on climatic conditions during growth and flowering. The pods usually contain three, occasionally more seeds.

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2.1.6.   SEEDS

Seed is small, hard, round or ovoid in shape, usually between 5 and 10mm in diameter with a smooth, shiny taste and a small distinct hilum.   Seed colour can be yellow, green, red, brown, black, slightly method or occasionally bi-coloured according to variety.

Pale Yellow is the most commercially acceptable colour for soybeans intended for human consumption and oil production.   Mature beans contain a higher percentage of protein than any other pulse and most other food crop.

2.2.0.   CULTURAL REQUIRMENT AND GROWTH PERIOD

2.2.1.   Propagation and Planting.

Seed are sown 2-4 cm deep on the flat or ridges 45-60cm apart; seeds 8-12cm apart in the row, depending on the vigour of cultivar.

2.2.2.   Nutrient Requirement

Phosphorous and Potassium are generally applied before sowing; acid soils may require dressings of lime if calcium is deficient.

2.2.3.   Irrigation

Irrigation is not normally required since the seeds are usually sown during a wet period, the plant becoming well established before dry season begins.   It may be necessary if the soil water deficit increases to a point where growth is likely retarded.

2.2.4.   Harvesting

Pods may be harvested 80-120 day from sowing, depending on the characteristic of the cultivar and the degree of maturity required.

2.2.5. Yields

Very variable, from 800-1500kg/ha of dried seeds.

2.2.6. Seed Production

Soybeans are self fertile and are normally self pollinates. Crops pollination by insects occur to a limited extent.

2.3.0. ORIGIN DISTRIBUTION AND IMPORTANCE

2.3.1. Origin

Soybean is a cultigen, probably originating in North East China earlier than 1000BC; grown in Korea, Manchuria and Japan at an early date. Introduced into Europe in the late eighteen century and to United State in 1304 and Nigeria in 1904 and widely distributed throughout the tropics (5).

2.3.2. Areas of Cultivation

Soybeans is presently the world's most important grain legume in terms of total production and international trade. The United State is the world's largest exporter and together with China and Brazil account for over 90 per cent of the world's production of Soybean. (FAO 1986).

Geographical location of areas of cultivation of Soybeans are: Tropical Asia (China, India, Indonesia, Philippine); East and West African (Tanzania and Nigeria); the Caribbean; central and South America (Brazil): Japan; North and South Korea; the South and mid Western State of the United State.

2.3.2. Importance of Soybean

Soybeans are probably the most important legumes in the world. The leaves are used for hay, silage and fodder, and green manure. The plant fixes nitrogen. The unripe seeds are cooked and eaten as a vegetable. The dried

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seeds may be eaten sprouted, fermented, boiled whole or split or may be used to make Soya milk and Soya Cheese.

These beans are high in protein, containing all the essential amino acids, and rich in oil.

In essence edible use of soybean derivatives is numberless ranging from pure oil, blended oil products, fats, margarine, shortening processed foods, meat and milk substitutes and protein. Flowers either daffted or straight are a basic raw material for wide range of protein foods together with cake are a staple ingredient in livestock feed.

2.4.0. FATS AND OILS

2.4.1. Oils the term.

The term oils is used in a generic sense to describe all substances that are greasy or oily fluids at ordinary temperatures. The term also refer to individual fatty or fixed (i.e. non volatile) oils such as Soybean oils; to hydrocarbon or mineral oils and derivates such as petroleum, state oil etc.; to odoriferous and volatile essential oils and to synthetic material that processes the characteristics of oiliness or lubricity. The mineral or hydrocarbon and odoriferous oil do not come within the scope of this work.

2.4.2. Fats and Oils Composition and Structure

Oils and fats consist primarily of glyceride, which are ester formed by the reaction of three molecular fatty acids with one molecules of triglyceride and the three molecule of water. When the three fatty acids are the same, the product is simple glyceride, when there are at least two different acids the product is mixed triglycerdies. Simple glyceride rarely occur in nature.

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\begin{array}{lll}
\mathrm{CH_2 - OH} & \overset{\displaystyle O}{\overset{\|}{\mathrm{HO - C - R}}} & \overset{\displaystyle O}{\overset{\|}{\mathrm{CH_2 - O - C - R}}} \\[2mm]
\mid & & \mid \\[1mm]
\mathrm{CH_2 - OH} \quad + & \overset{\displaystyle O}{\overset{\|}{\mathrm{HO - C - R^{i}}}} \longrightarrow & \overset{\displaystyle O}{\overset{\|}{\mathrm{CH_2 - O - C - R^{i}}}} \\[2mm]
\mid & & \mid \\[1mm]
\mathrm{CH_2 - OH} & \overset{\displaystyle O}{\overset{\|}{\mathrm{HO - C - R^{ii}}}} & \overset{\displaystyle O}{\overset{\|}{\mathrm{CH_2 - O - C - R^{ii}}}} \\
\text{Glycerol} & \text{Three different} & \text{Triglyceride} \\
& \text{Acids} & (R_1 \, R^{1} \text{ and } R^{ii} \text{ represents} \\
& & \text{carbon chain of different} \\
& & \text{length})
\end{array}
$$
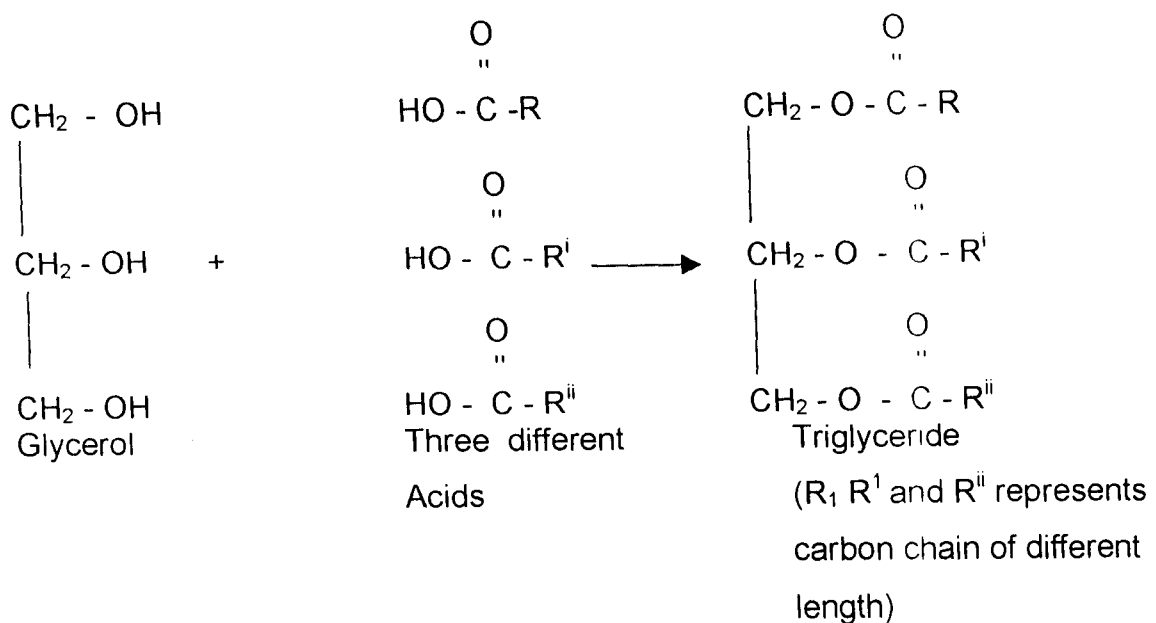
Since all triglycerides (both fat and oils) are tri esters of glycerol, the only difference among their molecules, most lie in the structure of their fatty acid chains.

The shorter the side chain (the lower is the molecular weight the lower the melting point) the more likely that the triglyceride is an oil, kind vice versa for fats. Also the more unsaturated the side chain, the lower the melting point and again, the more likely that the triglyceride is an oil.

2.4.3.   Sources Of Oils And Fats.

Oils and fats are substances of plant or animal origin. They are now volatile, insoluble in water, and oily or greasy to the touch. They constitute along with protein and carbohydrates; one of the main of foodstuff and they are widely distributed in nature.

Oils are usually liquid at ordinary temperatures such as $25^{0}c$ ($77^{0}f$), fat are solids at this temperature. There is no basis, however, for chemical distribution between the two. The difference is only physical one that can be removed by the physical changes. Therefore fats can be defined as plastic solid consisting of a mixture of crystalline particles and liquid oil.

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Animal oils and fats. Most important animal oil are derived from fish and other mane animal, while most animals fats are obtained from land animals. Example of animal oils include cod-liver oil and whale oil, example of animal fat include lard, bee tallow and butter fat.

Vegetable oils are derived from vegetables like soybean, castor seed, groundnut seed etc. while vegetable fats include cocoa butter, palm oil and coconut oil.

2.4.4. Classification of Fatty Oil.

Fatty oils may be drying, semi-drying or non drying. Drying Oils:- Drying oil dry completely and form a tough film when exposed to air. They do not dry by a process of evaporation like water, but as a result of a series of chemical reactions in which the oils combine with oxygen from the air.

Semi drying Oil:- When Semi - drying oil is exposed to atmosphere and become tacky, but do not dry completely. Examples are:- Soybean oil, Cotton seed oil etc.

Non - drying Oils:- Non drying oils, when exposed to atmosphere, do not dry at all but remain liquid and may eventually turn rancid. Example of non drying oil include: Peanut, Olive and eater oil.

2.4.5. Soybean Oil Composition.

A variety of products are made by processing soybeans in different ways. The most important process is oil extraction. Soya oil belongs to the group of oils most valuable for human nutrition, with the highest content of poly saturated fatty acids, linoleic acid, and linolenic acid. The high content of linoleic acid (essential fatty acid) makes soya oil superior in nutritive value to other vegetable oils, since this cannot be synthesized by the body and most be provided by the diet. The

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other advantage of linoleic acid is its ability to reduce blood cholesterol levels, thus decreasing the risk of developing heart and blood vessel diseases (3). On the other hand the presence of Linolenic acid disadvantages as it makes oil susceptible to oxidation.

Table: 2.4.5.:-

Fatty acid composition of Soya oils

Saturated	Composition %	Unsaturated	Composition %
Palmitic	12.0	Oleic acid	23.3
Stearic	4.0	Linoleic acid	52.3
Arachidic	0.5	Linolenic acid	7.9

2.4.6.   Soybean Oil Uses

Soybean oils has always had an essential role as food for human kinds however, our modern industrial world has found many important application for them. The oil is used in more than 50 different food products including margarine's, salad oils and cooking oils. In industry it is used in the making of paints, varnishes, insecticides, soaps, explosives, inks, lubricating oils, linoleum and many other products soybean oil is a major sources of high purify stearic acid used in food emulsifiers and floculation agents.

2.5.0.   Test and Analysis

The analysis and testing of oils and fats is needed for the assessment of quality and purity as well as for their identification. It is also the basis for commercial standards and trading rules.

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2.5.1.　　Iodine Value

The iodine value of a glyceride is related to its unsaturation: the higher the iodine value, the greater the unsaturation and the greater the liquidity. It is expressed in grams of iodine absorbed by 100g of oil.

2.5.2.　Saponification value

In indirect procedure, the ester is saponified and then back titrated to determine the mean molecular weight of the glyceride molecule. The lower the molecular weight, the greater the Saponification value.

2.5.3.　　Acid Value

The acid value of an oil or fact indicates the amount of non esterified fatty acid present. The determination is made by alkaline titration. It indicates correct amount of alkali to be added if the fat is to be subjected to alkali refining.

2.5.4.　　Colour.

Colour is almost invariable related to the quality: the darker the colour, the poorer the quality.

2.5.5.　　Peroxide value

Peroxide value is a measure of the extent of oxidative determination of the oil. It is determined by iodine titration in which peroxide equivalent is found in terms of elemental iodine liberated from potassium Iodide.

2.5.6.　　Titer.

Titer is the temperature at which the first permanent cloud appears when a molten sample is cooled in a specifies manner. It is an important test for detecting small amounts of contaminants.

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2.5.7. Refractive Index.

This is a physical attribute of oils measured by the angle through which a beam of light is bent when passing through a thin film of melted fat. The index of each fat falls within a narrow range and can be used as a characteristic of the fact in checking purity or specific gravity.

2.6.0 Solvent extraction

This is a process for separating one component of a substances by use of a solvent in which component is soluble. The process is widely used in separating oil from oil bearing material. Principally and most practically when the oil content is low as in soybeans.

Solvent extraction processes can be divided into two broad categories according to the origins of the differential solubility. On the one hand, it arises from purely physical differences between the two solutes, such as the polarity, while in the other case it can be traced to definite chemical interaction between solute and solvent.

2.6.1 Method of operation

Solvent extraction system are distinguished by operating cycle (batch, continuous or multi batch); by direction of streams (co current countercurrent or multi batch); by staging (single stage, multistage or differential stages); and by method of contacting (sprayed percolation, immersed percolation or solid dispersion).

The most important part of method of operation is the direction of streams; which is categorized into three types. Hybrid flow (single stage) contacting, the two phases (feed and solvent) are mixed into equilibrium and allow to settle, after which the extract phase (solvent plus dissolved solute) and raffinate are

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separately withdrawn. The separation attainable by this method is limited by the equilibrium prevailing under extraction conditions.

In cross current extraction, fresh solvent is added at each stage; enhanced separation is obtained; but solvent requirement is high.

Counter current extraction is the most efficient and is the choice for commercial operation whenever possible. In this approach, raffinate and extract phase flows counter currently and emerged at opposite ends of the contractor.

2.6.2 Solvent selection

Properties of a solvent such as it's boiling point, density and viscosity affect its suitability and selectivity to effect more complete and economical extraction of the desired solute. Other factors that have to be considered are toxicity, recoverability, availability, price, chemical reactivity, solvent power, inflammability etc.

CHAPTER THREE

3.0.0   METHODOLOTGY

3.1.0   Equipment and Apparatus

A.   **Electric Balance:-** Balance was used to weigh samples of seed powder for experiment, and was also used to weigh dried sample from oven.

B.   **Sieve:-** It was used to classify samples into different particle sizes.

C.   **Electric oven:-** It was used to remove moisture from seeds in preliminary preparation and it was also used to dry samples after the extraction.

D.   **Blender:-** It was used for size reduction of seeds.

E.   **Water Bath:-**Traces of solvent and moisture were removed from the extracted oil by using this equipment.

F.   **Soxhlet extractor:-**This is an equipment in which the extraction of oil and distillation of solvent is done. It serves as reactor and distillation column in this work.

G.   **Heating mantle:-** This equipment supplied necessary heat used during the experiment.

3.1.1 Apparatus

A.   Beaker

B.   Filer paper

C.   Sample bottle

D.   Desicator

E.   Spatular

F.   Tong

G.   Sample bottle

H. Crucibles

3.2.0 REAGENTS

A. Hexane

B. Distilled water

C. Sodium thiosulphate

D. Starch solution

E. Hubl reagent

F. Isopropyl Alcohol / Toluene mixture

G. Phenolphthalein indicator

H. Alcoholic potassium hydrochloride solution

3.2.1 REAGENTS PREPERATION

A. Hubl reagent:- 5g iodine was dissolved in 100ml ethanol in a brown glass bottle. 2g mercuric chloride was then weighed into another bottle and dissolved in 50ml ethanol. Equal volumes of these two solution were mixed before it was used.

B. **Isopropyl Alcohol / Toluene mixture:-** Equal parts of isopropyl and toluene were mixed in a 500ml conical flask. The mixture was then neutralized with 0.1 mol/dm^3 Sodium hydroxide solution. The end point was known when the pink colour persisted for about one minute. This mixture is called neutral solvent.

C. **Alcoholic potassium chloride solution:-** This solution was prepared by dissolving 5.6g of sodium hydroxide in one liters of methyl alcohol. The solution was standardized using standard solution of hydrochloric acid.

3.2.2. Material

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3.3.0   Methodology

They are different means and method of oil extraction from oil seeds both in laboratory and industry. To remove or extract oil from oil seeds with a relatively low oil content such as soybeans, solvent extraction is the efficiency and low operating cost of solvent extraction method, led to the reason of employing solvent extraction method in this work.

Preparation:- The process of operation consist of preparation, extraction and meal finishing phases.

3.3.1   Preparation.

A.   Preparation Stage:- This is the first stage of extraction. and it involves the following stages

i.   **Pretreatment:-**   The 'processing chain' beings with the cleaning of the beans, which passed through a screening operation (manual operation). With this means metal pieces were removed, larger and smaller sized seeds were separated from the seeds. Light foreign matter and dust were blown out.

ii.   **Drying:-**   Soybeans were dried in an electric oven for about fifteen hours at 70°C. Drying process was stopped, when there was no more significant charge in the previous and new weight of the sample seed. (The weight of sample approximately remain constant). The aim of drying was to reduce moisture from the seeds.

Oven type:-   Hot box oven, size 2. (made in Britain)

iii.   **Size reduction:-**   dried seeds were reduced to powdery form with the aid of the blending machine. This is done because, finely powdered sample are homogenous and can be sampled with greater precision and

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accuracy. They also present a large surface area: volume ratio to any potential solvent or reagent used in dissolution.

iv.   **Size classification:-** Electric vibrational sieve was used to classified powder soybeans into different particle sizes This classification allow production of closely graded materials. For this work eight different aperture sizes were used, they are 0-170μm. 170-250μm, 250-335μm, 335-774μm, 0-250μM, 250-500μm, 500-710μm and 710-350μm.

v.   Weighing;- An electric weighing balance, was used to weigh constant 5.000g classified sample of soybean power; which was th sample weight used for the experiment.

Type: Mettler PE 160, (made in Britain).

3.3.2   Extraction

Extraction was carried out in the laboratory with the aid of soxhlet extractor, soxhlet apparatus is a three in one equipment made of reflux condenser, extractor and round bottom flask. The equipment clamped on regulated heating mantle, and water pipe was connected to the condenser inlet, and made to come out through the outlet in the same condenser.

The sample was placed in soxhlet thimble, a disposable porous container made of stiffened filter cloth. It was assumed that solvent and extracted solute molecules, but not solid sample particles can flow freely through the thimble. Reflux organic solvent flows through the thimble and leaches out sample soluble component. The solvent with extracted components flows through the thimble and inner tube gradually fills up with the solution. When the level reaches the side arm however, all the solvent plus extracted components siphon off into the

flask and thimble starts to fill with solvent again, until the fixed time elapsed, and the equipment is put off.

The advantage of this equipment is that, it can be used before distilling solvent away from the extract oil. The third accept of processing; meal finishing is out of this work scope.

3.4.0 DETERMINATION OF MOISTURE CONTENT OF THE SEEDS

A metallic dish was dried in an oven at $80^{o}C$ for 20 minutes, and cooled in desicator and weighed as (W_1). Few gramms of the sample was taken into the dish and weighed (W_2). The dish with the sample was then dried at $80^{o}C$ for 5 hours (until constant weight was reached), and it was quickly transferred to a desicator to cool. It was then quickly weighed as (W_3) with minimum exposure to the atmosphere. The loss in weight of the sample during is the moisture content.

$$\text{Moisture (\%)} = \frac{W_3 - W_1}{W_2 - W_1} \times 100$$

3.4.1 DETERMINATION OF OIL CONTENT OF THE SEED

A simple apparatus of soxhlet for extraction of oil soybean as shown in the figure setup .

5g of the sample powder taken into a thimble of known weight (W_1). They together weighed (W_2). The thimble with sample were placed inside a soxhlet extractor. 250ml of hexane was poured into a round bottom groved joint flask. The soxhlet extractor with the thimble plus sample was filled into the flask, which was situated in electrically connected heating mantle. The heating mantle is switched on and its temperature regulator is put at boiling point temperature of the solvent. The heat increased carefully and slowly until the solvent boils, at this point timer is switched on. (when solvent started boiling).

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Condensed solvent vapor collects in the thimble and dissolved the lipid in the sample. The solvent with dissolved lipid will continue to run back into the flask. The heating and so extraction process is continued for specified time (i.e 3hours, fours, 6 hours). The thimble with contents is removed, dried in oven at $50^{o}C$ for some hours, cooled in a desicator and weighed (W_3)

$$\text{Lipid (\%)} = \frac{(W_2 - w_3)}{(W_2 - W_1)} \times 100$$

W_1 = weight of thimble

W_2 = sample + thimble weight

W_3 = weight of dried sample

The solvent is distilled off to about 20ml, the oil in solvent solution is quantitatively transferred on to an evaporating dish, cooled, dried in a desicator.

3.4.2 Advantages of soxholet apparatus

A. Extraction of the sample always occur with pure solvent (because it distils into the sample)

B. Extraction may continued unattended, until complete

C. Extraction occurs at an elevated temperature (essentially that of the boiling point of solvent.)

3.4.3 Disadvantages

The extracted solute are being continually heated in the solution containing vessel. This might cause thermal degradation, if solute are thermally liable.

3.5.0. Factorial Design.

It has been established that for multivariable experiments such as the extraction of oil from oil seeds in which purposefully changes are made to the

regressor variables so as to identify reasons for changes in the output response, the statistical methods are most appropriate (7).

Factorial design are widely used in research work especially in experiments involving several factors where it is necessary to study the joint effects of the levels of factors are considered in the factorial design.

3.5.1 2^2 Factorial design

A 2^2 Factorial design provides the time frame and order of experimental runs for designing the extraction of oil from soybeans.

From results obtained from the experiments, a first order multiple linear relationship provides an acceptable approximation to the true functional relationship between the regressor variable X_i and the independent variable (y). The generalized regression equation of the model will be:

$$Y = b_0 X_0 + b_i X_i + b_2 X_2 + b_{12} X_{12}$$

Where b_0, b_i, b_{ij} are regression coefficients. Sign improve X_0, X_l, X_{ii} are coded signs.

3.5.2. THE EXPERIMENTAL DESIGN METHOD

The run-by-run experimental design of a two variable two level factorial experimental is shown in table 3.5.2. The runs were replicated two time (r=2) giving a total number of samples as 4 x 2 = 8 (N x r) samples.

In 2^2 factorial design the low and high levels of the factors are coded as "- " and "+" respectively.

Table 3.5.2. Design matrix (Table) for 2^2 full factorial experiment

Run N_0	X_0 b_0	X_1 b_1	X_2 b_2	X_{12} b_{12}
1	+	-	-	+
2	+	+	-	-
3	+	-	+	-
4	+	+	+	+

The level of the two factors are listed in standard order in the column X_1, X_2 in the table and their interaction in column X_{12}. Matrix is obtained by starting column X_1 with one minus and signs are alternated down the column, till row (run) 2^k is reached, start column X_2 with two minus signs and signs are alternated in block of two until row 2^k is reached. While X_{12} is the combination of both signs (X_1 & X_2). The sequence of "+" and "-" signs in the column tell us how to combine the observations to get the main effect and the interaction.

3.5.3. Regression Analysis

Regression is concerned with replacing data by a single line, that also represents a mathematical equation. The purpose of finding this line generally is to predicts. The advantages of this analysis is that, these is no necessity for a course and effect relation to exist in order for one to want to predict one valve from the other.

The general model for two variables X_1 and X_2 dependent and independent variable respectively.

$$X_{1r} = a_{1.2} + b_{12} X_2.$$

For multi variables, (one dependent X_1 and two independent X_2, X_3) the generalised model is:

$$X_{1r} = a_{1.23} + b_{12.3} X_2 + b_{13.2} X_3$$

$a_{1.2}$, b_{12}, $a_{1.23}$, $b_{12.3}$ and $b_{13.2}$ are regression equation constant.

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3.5.5. Correlation Analysis

Correlation analysis is a means of showing the degree to which variables are linearly related. Pearson product moment correlation is the method used for this work.

Pearson Product Correlation Value (r) ranges from -1 to +1 and values in between the range. Positive value of r indicates a line group up to the right (as X increases so does Y). While negative valves of r indicate a line going down to the right (as one values increases or decreases). And when r=0, it implies no linear relation.

3.5.6. Coefficient of Determination

Coefficient of determination provides a measure of how much of the observed variation can be essentially explained or accounted for through the correction coefficient of the regression line. The value is r^2.

3.6.0 Mass transfer in leaching Operation

From the concept of thin film as providing resistance of transfer, the equation of mass transfer is give as:

$$\frac{dm}{dt} = \frac{K^1 A (C_s - C)}{b}$$

A is the area of the solid -liquid inter face

B is the effective thickness of liquid

C is the concentration of the solute in the bulk of solution at time t

C_s is the concentration of saturated solution in contact with the particles

M is the mass of solute transferred in time t and

K^1 is the diffusion coefficient.

For a batch process, in which V, the total volume of solution is assumed to remain constant then:

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$$dm = vdc$$

and $$\frac{dc}{dt} = \frac{K^1 A(C_s - C)}{bV}$$

The time t taken for the concentration of the solution to rise from its initial value C_0 to a value C is found by integration, on the assumption that both b and A remain constant.

Rearranging:

$$\frac{ds}{C_s - C} = \frac{K^1 A}{vb} dt$$

$$in \quad \frac{C_s - C_0}{C_s - C} = \frac{K1A}{Vb} t$$

If pure solvent is used $C_0 = 0$ and

$$1 - C = e^{-(K1A/bv)t}$$

$$C = C_s (1 - e^{-(K1A/bv) t})$$

Which shows that solution approaches a saturated condition exponentially.

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CHAPTER FOUR

4.0.0 RESULTS OF EXPERIMENTAL AND DISCUSSION

4.1.0. Yield (%) $\quad=\quad \dfrac{\text{Extract}}{\text{Mass of sample}} \times 100\%$

Average extract $\quad= \dfrac{1^{st}\text{ extract} + 2^{nd}\text{ extract}}{2}$

4.1.2.   STAGE 1:   3 HOURS EXTRACTION

Table 4.1.1 Experimental data table

Sample Size μm	Ave Size μm	Mass of Sample g	1st run g	2nd run g	Ave extract g	Yield %
0-250	250.000	5.000	1.220	1.216	1.216	24.160
250-500	375.000	5.000	1.170	1.174	1.173	23.460
500-710	605.000	5.000	1.144	1.136	1.140	22.800
710-850	780.000	5.000	1.068	1.070	1.069	21.380

Table 4.1.2
STAGE 2: 6 HOURS EXTRACTION

Sample Size μm	Ave Size μm	Mass of Sample g	1st run g	2nd run g	Ave extract g	Yield %
0-250	250.000	5.000	1.285	1.287	1.286	25.720
250-500	375.000	5.000	1.229	1.235	1.232	24.640
500-710	605.000	5.000	1.220	1.220	1.220	24.400
710-850	780.000	5.000	1.212	1.206	1.209	24.180

Table 4. 1.3. Combination of both stages, the yield, % yield at different time.

Table 4:13 Comparison table

		3 hours		6 hours		Percentage Increment in extraction between 3 & 6 hours Yield
Sample size μm	Mass of Sample g	Yield g	Yield %	Yield g	Yield %	%
0-250	5.000	1.218	24.160	1.286	25.720	5.600
250-500	5.000	1.173	23.460	1.232	24.640	5.020
500-710	5.000	1.140	22.800	1.220	24.400	7.010
710-850	5.000	1.069	21.380	1.209	24.180	13.096

Table 4.1.4:- Experimental data table

STAGE 3 : 6 HOURS EXTRACTION

Sample Size μm	Ave Size μm	Mass of Sample g	1st run g	2nd run g	Ave extract g	Yield %
0-170	170.000	5.000	1.382	1.372	1.377	27.320
170-250	210.000	5.000	1.299	1.305	1.302	25.040
250-335	292.500	5.000	1.258	1.244	1.251	25.020
335 - 774	554.500	5.000	1.216	1.216	1.216	24.320

Stage 4: 4 hours Extraction

Table 4:1.5: Experimental data table.

Sample Size μm	Ave Size μm	Mass of Sample g	1st run g	2nd run g	Ave extract g	Yield %
0-170	170.000	5.000	1.254	1.264	1.264	25.280
170-250	210.000	5.000	1.201	1.221	1.211	24.220
250-335	292.500	5.000	1.186	1.184	1.184	23.220
335 - 774	554.500	5.000	1.097	1.087	1.092	21.840

Table 4.1.6: Table of Yield for both 4 hours and 6 hours at the different particle size.

Sample size μm	Mass of Sample g	4 hours Yield g	4 hours Yield %	6 hours Yield g	6 hours Yield %	Percentage Increment in extraction between 4 & 6 hours Yield %
0-170	5.000	1.264	25.230	1.377	27.540	8.939
170-250	5.000	1.211	24.220	1.302	25.040	6.989
250-335	5.000	1.184	23.680	1.251	25.020	6.700
335-774	5.000	1.092	21.840	1.216	24.320	11.355

Table 4.1.7:- Table of experiment result in increase order of particle size with their corresponding yield and yield percentage at different time duration.

Sample size μm	Average sizes g	3 hours Yield g	3 hours Yield %	4 hours Yield g	4 hours Yield %	6 hours Yield g	6 hours Yield %
0-170	170.000	-	-	1.264	25.280	1.377	27.540
170-250	210.000	-	-	1.211	24.220	1.302	25.040
0-250	250.000	1.218	24.160	-	-	1.286	25.720
250 -335	292.500	-	-	1.184	23.680	1.251	25.020
250-500	375.500	1.173	23.460	-	-	1.232	24.640
335-774	554.500	-	-	1.092	21.840	1.215	24.320
500-710	605.000	1.140	22.800	-	-	1.220	24.400
710-850	780.000	1.069	21.380	-	-	1.209	21.380

Table 4:1.8: The experimental result of both Hexane and Benezene at the same particle sizes for 3 hours extraction.

3HOURS EXTRACTION

SOLVENT	HEXANE		BENZENE	
Sample Size μm	Yield %	Yield %	Yield %	Yield %
0-250	1.218	24.160	0.960	38.400
250-500	1.173	23.460	0.890	35.600
500-710	1.140	22.800	0.790	31.600
710-850	1.069	21.380	0.700	28.000

** for Hexane, sample mass was 5g and for Benzene sample mass was 2.5g

Table 4:1.9: The experiment result of both hexane and benzene at the same particle sizes for 6 hours extraction.

6 HOURS EXTRACTION

SOLVENT	HEXANE		BENZENE	
Sample Size μm	Yield %	Yield %	Yield %	Yield %
0-250	1.286	25.720	1.000	40.000
250-500	1.232	24.640	0.920	35.800
500-710	1.220	24.400	0.890	35.600
710-850	1.209	24.180	0.800	32.000

** for Hexane, sample mass was 5g and for Benzene sample mass was 2.5g

*** Benzene date source is reference No 6.

4.2.0.   Discussion of Results

The extraction of oil from soybean was done in four stages, with the stage 1 and stage 2 serves as one sub-group, while stage 3 and 4 serves as another sub-group. Determinant factor in each group were time of extraction and particle size. While temperature of extraction, sample mass and solvent (hexane remain constant throughout the during of experiment .

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Table 4.11 and 4.12 shows the experimental result of stage 1 and stage 2, which corresponds to the 3 hours and 6 hours of extraction respectively with the same particle sixes used for both time of extraction. The results show that oil extracted is inversely proportional to the particle size.

The pattern could be attributed to the fact that there is greater interfacial area between the solid and solvent as the particle size decreases, it implies that the smaller the particle size, the shorter the distances, the solvent has to travel within the solid, thereby increasing the rate of reaction based on extraction principles.

Table 4:13   Shows the Yield and percentage yield for both 3hours and 6hours of extraction. The percentage difference or increment in yield between 3 hours and 6 hours of extraction rages between 5 and 13 per cent. The extraction does not increase linearly with increment in time. It's also observed from the table 4.13 that yield at 0-250μm for 3hours is better than that of 710-850μm size at 6 hours of extraction.

Table 4.14 and 4.15 :-   shows experimental result of stage 3 and stage 4, which corresponds to 6 hours and 4 hours of extraction respectively. Both tables also observed inverse relationship between the particle size and time of extraction. The tables show candidly that extraction with proportionate yield is achievable at particle size of 170μm.

Table 4.16:- Shows the yield and percentage yield over a different particle size at both 4 hours and 6 hours time of extraction. The percentage increment between the extraction of 4 hours and 6 hours at different particle sizes ranges between 6.7 – 11.35 per cent. While table 4.17 is summary of the table 4.11 to

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table 4.1.6. Despite combination of two experimental sub-groups together, the inverse relationship was still established.

Table 4.1.8 and 4.1.9:   Shows the comparison of yield of hexane with that of benzene at 3 hours and 6 hours of extraction respectively. Benzene also observed inverse relationship.

Figure 4.1. and 4.2. is a graph of oil extract against particle size, it gave a scattered point which can be approximated to a straight line with negative gradient. The figure also simultaneously contain predicted valves, by using regress equation, which gave better straight line with negative gradient. Figure 4.3 is a plot of yield against particle sizes an time simultaneously. the point of intersection of both lines gave optimum time, (4.7hours) & particle size (470μm), which correspond to 1.195g yield of oil.

The regression analysis of table 4.1.1 presented in table 6.1 gave a equation of X_{1r} = 1.2808 – 2.6029 X_2. The negative gradient is 2.6029 with the intercept 1.2808. the coefficient of correlation is found o be –0.98, which implies strong but negative relationship between yield and particle size. Sample coefficient of determination "r^2" is found to be 0.96, which is a measure of how much observed variation could be essentially explained for through the correlation coefficient of the regression line. While standard error is 1.5 per cents.

For six hours time of extraction, the regression equation was gotten to be X_{1r} = 1.3003 – 1.2655 X_2. With the correlation coefficient and coefficient determination "r^2" to be –0.878 and 0.77 respectively with the standard error of 2 per cent.

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By using multiple linear regression for particle size, time and yield together, the regression equation gotten is,

$X_{1r} = 1.10714 - 0.1634 X_2 + 0.000008032 X_3$. This gives better estimate than normal regression -0.1634 is a negative gradient, while 0.00000832 indicates that the time of extraction has little effect on the extraction in compared with yield and particle size. The standard error was found to be 6.80 per cent.

The regression of yield on sample particle was found to be

$X_{1r} = 1.289 - 1.921X_2$ which implies negative gradient 1.921 with intercept 1.289 and standard error of 6.7 per cent. The error is less than that of multiple regression because of negative effect of time on the extraction

Also the regression of yield on time was found to be

$X_{1r} = 1.1.803 + 0.000008034 X_2$, this implies 1.1.803 is the intercept, but with positive 0.000008032 intercept, which means, yield and time are directly proportional to each other but insignificant. The standard error was found to be 7.9 per cent, which is better than bivariate regression in estimate and also effect of time on yield is very low in compared with particle size.

By factorial design the fitted model is

$Y_u = 1.1196 - 0.057 X_1 + 0.052 X_2 + 0.018 X_{12}$.

This model is valid for the range of valve for which experiment is performed.

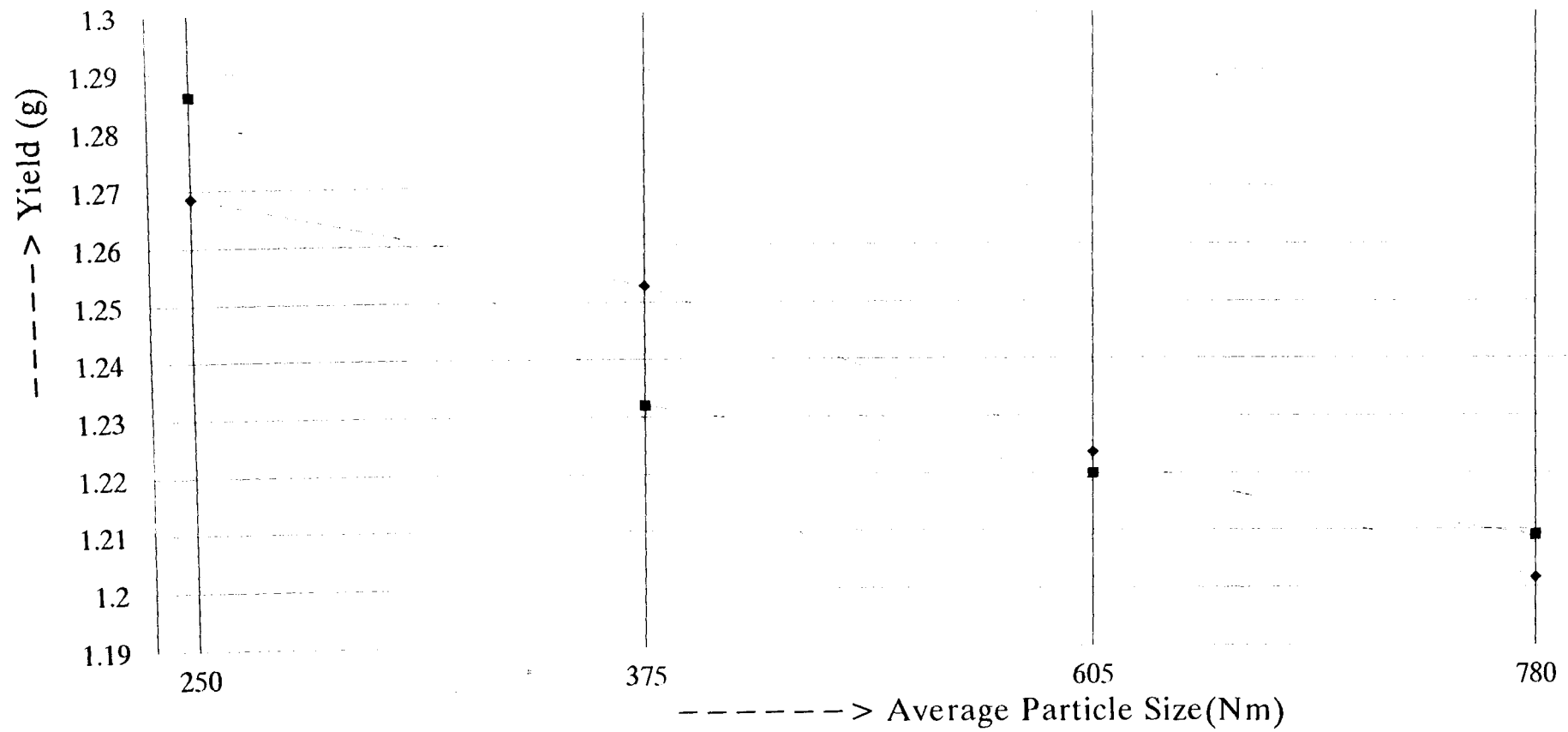Fig 1

Yield Vs Average Particle Size | 3 Hours Extraction|

key
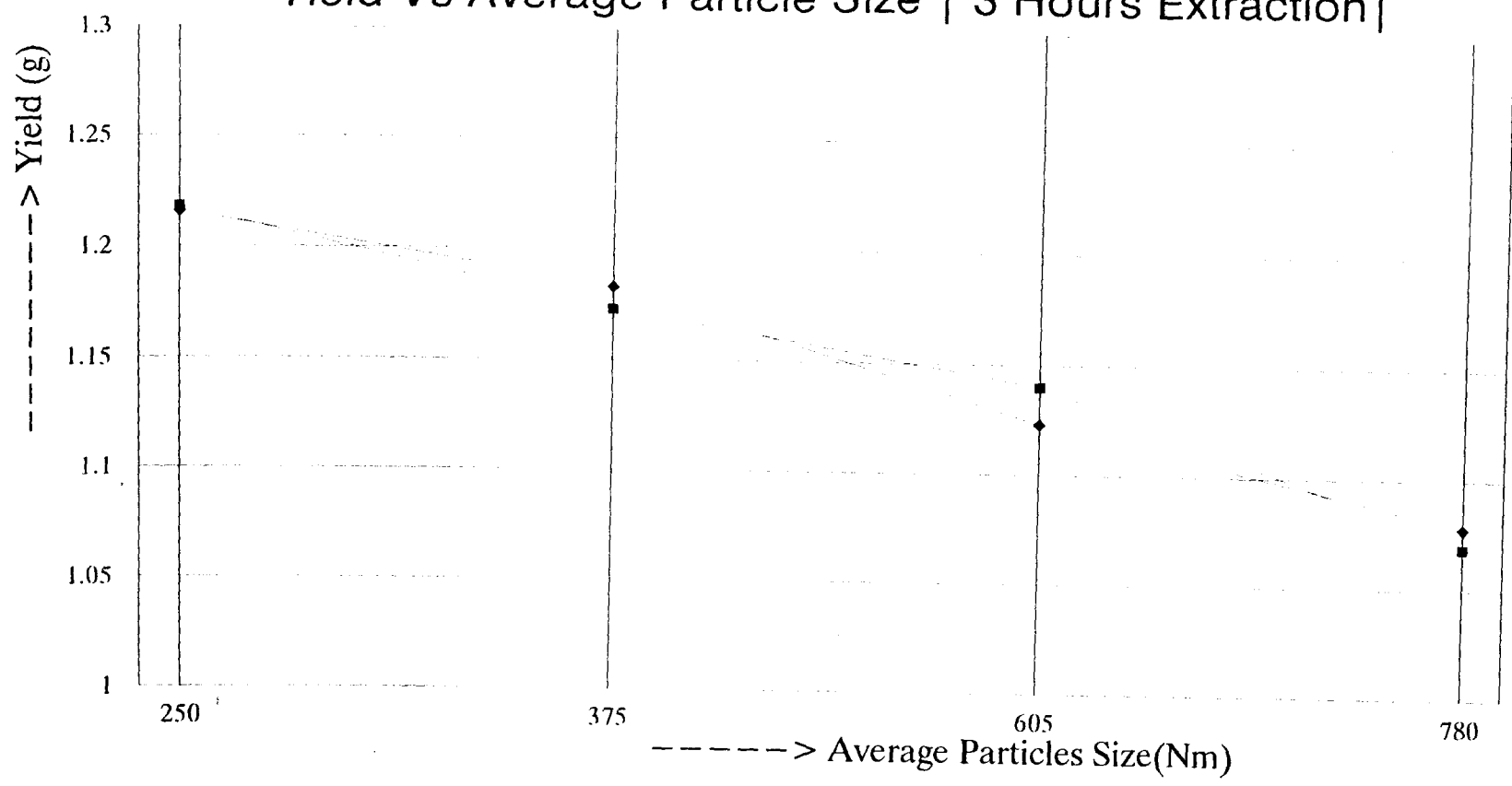
♦   Predicted values

■   Experimental values

fig 2

YIELD VS AVERAGE PARTICLE SIZES
6 HOURS EXTRACTION

key

♦   Predicted Values

■   Experimental Values.

CHAPTER FIVE

5.0.0   CONCLUSION AND RECOMMENDATION

5.1.0   CONCLUSION

Hexane was used for extraction of oil from Soybean. for three hours of extraction, the amount of oil extracted were 1.218g, 1.1739. 1.140g and 1.069g for the average particle sizes 250μm, 375μm and 780μm respectively. For the same average particle size, extract for 6 hours were 1.286g. 1.232g, 1.220 and 1.209g.

Oil varying from golden yellow to deep yellow colour was obtained, by leaching Soybean oil with hexane. The percentage oil obtained varied inversely with particle size of the sample and increased with leaching time. Also it was obtained that below 250μm average particle size i.e (210μm and 170μm) the percentage of oil extracted increased as the particle seize continue to decreases and as time also increases.

The optimum time of 4.7 hours and average particle size of 470μm were obtained for this experiment, with factorial design fitted model of:

$Y_u = 1.196 - 0.057X_1 + 0.052 X_2 + 0.018X_{12}$.

The developed model is valid only for valves of X_i that fall within the intervals of values used in predicting it. The value outside the interval is an extrapolation, which is suspectable.

5.2.0   RECOMMENDATIONS

1.   There are mixture of solvents that are being tested for the better affinity for extraction than that of hexane, such solvents can be used to increase the yield of oil.

2. The experiment should be carried out to determine the optimum volume of solvent required for each particular weight of sample, and time of extraction simultaneously. Because it's noticed that the same volume of solvent for different extraction affect the higher time of extraction negatively. The extraction is normally incomplete at the particular instance, they become supersaturated before extraction time elasped.

3. Design of plot plant will also be recommended.

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APPENDIX 1

STATISTICAL ANALYSIS

a. Regression Analysis

b. Correlation Coefficient

c. Standard Error

d. Coefficient of determination.

a. Regression Analysis

Least square method for estimating linear regression of a homoscedestic distribution is:

For Bivariate, X_1 dependent variable and X_2 independent variable

$$X_{1r} = a_{1.2} + b_{12} X_2 \qquad\qquad - \quad 1$$

$$b_{12} = \sum x_1 x_2 \Big/ \sum x_2{}^2 \qquad\qquad - \quad 2$$

$$a_{1.2} = \bar{x}_1 - b_{12} \bar{x}_2 \qquad\qquad - \quad 3$$

$$\sum x_1 x_2 = \sum x_1 x_2 - n\, \bar{x}_1 \bar{x}_2 \qquad\qquad - \quad 4$$

$$\sum x_2{}^2 - \sum x_2{}^2 - n\, \bar{x}_2{}^2 \qquad\qquad - \quad 5$$

$$\sum x_1{}^2 = \sum x_1{}^2 - a_{1.2} \sum x_1$$

c. Standard error for bivariate

$$S^2{}_{1.2} = \frac{\sum x_1{}^2 - a_{1.2} \sum x_1 x_2}{n-2}$$

for multiple regression; estimate of plane of regression of X on X_2 and X_3 is given as

$$X_{1r} = a_{1.23} + b_{12.3} X_2 + b_{13.2} X_3 \qquad\qquad - \quad 7$$

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Gives:

$$b_{12.3} \sum x_2^2 + b_{13.2} \sum x_2 x_3 = \sum x_1 x_2$$

    -   8

$$b_{12.3} \sum x_2 x_3 + b_{13.2} \sum x_3^2 = \sum x_1 x_3$$

$$a_{1.23} = \bar{x}_1 - b_{12.3} \bar{x}_1 - b_{13.2} \bar{x}_3$$ - 9

$$\sum x_1^2 = \sum X_1^2 - n \bar{x}_1^2$$ - 10

$$\sum x_2^2 = \sum X_2^2 - n \bar{x}_2^2$$ - 11

$$\sum x_3^2 = \sum X_3^2 - n \bar{x}_3^2$$ - 12

$$\sum x_1 x_2 = \sum X_1 X_2 - n \bar{x}_1 \bar{x}_2$$ - 13

$$\sum x_1 x_3 = \sum X_1 X_3 - n \bar{x}_1 \bar{x}_3$$ - 14

$$\sum x_2 x_3 = \sum X_2 X_3 - n \bar{x}_2 \bar{x}_3$$ - 15

$$\bar{x}_1 = \frac{\sum X_1}{n}$$ - 16

$$\bar{x}_2 = \frac{\sum X_2}{n}$$ - 17

$$\bar{x}_3 = \frac{\sum X_3}{n}$$ - 18

c.   Standard error of estimate ($S_{1.23}$) is given by

$$S^2_{1.23} = \frac{\sum x_1^2 - b_{12.3} \sum x_1 x_2 - b_{13.2} \sum x_1 x_3}{n-3}$$ - 19

b.   Correlation coefficient:

$$r = \frac{n \sum X_1 X_2 - \sum X_2 \sum X_1}{\sqrt{[n \sum X_2^2 - (\sum X_2)^2]} . [\sum X_1^2 - (\sum X_1^2)]}$$ - 20

d.   Coefficient of determination = r^2 - 21

Table 6.1: Statistical data analysis for the sample sizes (g) and yield (g)

X_1(g)	X_2 (cm)	X_1X_2	X_1^2	X_2^2
1.2180	0.0250	0.03056	1.4835	0.0006
1.1730	0.0375	0.0439	1.3759	0.0014
1.1400	0.0605	0.0690	1.2996	0.0037
1.0690	0.0780	0.0834	1.1428	0.0061
4.6000	**0.201**	**0.2268**	**5.3018**	**0.0118**

By 16: $\bar{x}_1$ = $\dfrac{4.600}{4}$ = 1.15

By 17: $\bar{x}_2$ = $\dfrac{0.201}{4}$ = 0.05025

By 4: $\sum x_1 x_2$ = 0.2268 – (4) (1.15) (0.05025) = -0.0043605

By 5: $\sum x_2^2$ = 0.0118 - 4(0.05025)^2 = 0.00167525

By 2: b_{12} = $\dfrac{-0.0043605}{0.00167525}$ = -2.6029

By 3: $a_{1.2}$ = 1.15 - (-2.6029) (0.5025) = 1.2808

By 1: X_{1r} = 1.2808 - 2.6029 X_2 ⟶ A

Standard error

$S^2_{1.2}$ =

By 6: $\dfrac{5.3018 - 1.2808 (4.6000) – (-2.6029) (0.2268)}{2}$

$S^2_{1.2}$ = 0.000221638

$S^2_{1.2}$ = 0.015 ≅ 1.5% ⟶ B

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Correlation coefficient

By: 20　　r　=　$\dfrac{4 \times 0.2268 - (0.2010)(4.6000)}{\sqrt{(4 \times 0.0118 - (0.2010)^2 \cdot 4 \times 5.3018 - (4.6000)^2)}}$

　　　　r　=　-0.98　　　　　　　　　——————➤ C

Coefficient of determination

By 21: $(-0.98)^2$　　= 0.96　　　　　——————➤ D

Predicted valves:- Using the Model

X_1 By A:　　$X_{1r} = A$

X_2	0.0250	0.0375	0.0605	0.0780
X_1	1.2180	1.1730	1.1400	1.0690
X_{1r}	1.2160	1.183	1.123	1.0778

Table 6.2: Statistical data analysis for the sample sizes (g) and yield (g)
6 HOURS OF EXTRACTION

X_1(g)	X_2 (cm)	X_1X_2	X_1^2	X^2_2
1.2860	0.0250	0.0322	1.6538	0.0006
1.2320	0.0375	0.0462	1.5178	0.0140
1.2200	0.0605	0.0738	1.4884	0.0037
1.2090	0.0780	0.0943	1.4617	0.0061
4.9470	0.2010	0.2465	6.1217	0.0118

By 16:　　$\bar{x}_1$　　= $\dfrac{4.9470}{4}$　　= 1.23675

By 17:　　$\bar{x}_1$　　= $\dfrac{0.201}{4}$　　= 0.05025

By 4:　　$\Sigma\, x_1 x_2$　　= 0.2465 – 4 (1.23675) (0.05025) =　　-0.00212

By 5:　　$\Sigma\, x_2^2$ = 0.0118 - 4(0.05025)^2　　=　　0.00167525

By 2:　　b_{12}　=　$\dfrac{-0.00212}{0.00167525}$　　=　　-1.2655

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By 3: $a_{1.2}$ = $1.23675 - (-1.2655)(0.05025)$ = 1.3003

By 1: X_{1r} = $1.3003 - 1.2655\ X_2$

Standard Error

By 6: $S^2_{1.2}$ = $\dfrac{6.1217 - (1.3003)(4.9470) - (-1.2655)(0.2465)}{2}$

$S^2_{1.2}$ = 0.000404

$S^2_{1.2}$ = 0.020 $\cong$ 2.0% $\longrightarrow$ B

Correlation Coefficient

By 20:

$$r = \frac{4 \times 0.2465 - (0.201)(4.9470)}{\sqrt{4 \times 0.0118 - (0.2010)^2}\ .\ 4 \times 6.1217 - (4.9470)^2}$$

= -0.878 $\longrightarrow$ C

Coefficient of determination

By 21 = $(-0.878)^2$ =   0.77 $\longrightarrow$ D

X_2	0.0250	0.0375	0.0605	0.0780
X_1	1.2860	1.2320	1.2200	1.2090
X_{1r}	1.2686	1.2528	1.2237	1.2016

Linear regression for X_1 (yield) and X_2 (sample sizes)

By 16: $\bar{x}_1$ = $\dfrac{9.547}{8}$ =   1.193375

By 17: $\bar{x}_2$ = $\dfrac{0.402}{8}$ =   0.05025

By 4: $\Sigma\ x_1 x_2$ = $0.4733 - 8(1.193375)(0.05025)$ = -0.00643675.

By 5: $\Sigma\ x_2^2$ = $0.023551 - 8(0.05025)^2$ = 0.0033505.

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By 2: b_{12} = $\dfrac{-0.00643675}{0.0033505}$ =   -1.921131174

By 3: a_{12} = 1.193375 – (-1921131174) (0,05025) = 1.289911842

X_{1r} = 1.289 – 1.921 X_2 ⟶ A

Standard Error

By 6:

$$S^2_{1.2} = \dfrac{11.4325 \ - (1.28911842)\,(9.547) \ - (-1.921131174)\,(0.4733)}{6}$$

$S_{1.2}$ = 0.06705

Linear regression of yield (X_1) on time (X_3)

By 16: x_1 = 1.193375

By 17: $\bar{x}_2$ = 16200

By 5: $\sum x_2^2$ = 23328×10^5 - 8(16200)^2 = 2332800000

By 4: $\sum x_1 X_2$ = 156535.2 - 8(1.193375) (16200) = 1873.8

By 2: b_{12} = $\dfrac{1873.8}{2332800000}$ = 0.00000803

By 3: $a_{1.2}$ = 1.193375 – (0.00000803) (16200)   = 1.180362

By 1: X_{1r} = 1.1803 + 0.00000803 X_2 ⟶ A

Standard Error

By 6: $S^2_{1.2}$ = $\dfrac{11.4325 - (1.180361777)\,(9.5470) - 0.00000803\,(156535.2)}{}$

$$S_{1.2} = 0.079465242 = 7.9\% \longrightarrow B$$

Table 6.3: Statistical data analysis for multiple regression with respect to yield (X_1), sample sizes (X_2) and time (X_3)

*Table 6.3: is formed from table 4.13, by rearranging yield in descending order for both 3 hours and 6 hours.

X_1 (g)	X_2 cm	X_3 $(5) \times 10^5$	$X_1 X_2$	$X_1 X_3$ $\times 10^5$	$X_2 X_3$ 10^2	X_1^2	X_2^2	X_3^2 10^{10}
1.2860	0.0250	0.2160	0.0322	0.2778	5.4000	1.6538	0.0006	0.0467
1.2320	0.0375	0.2160	0.0462	0.2661	8.1000	1.5178	0.0014	0.0467
1.2200	0.0605	0.2160	0.0738	0.2635	13.0680	1.4884	0.0037	0.0467
1.2180	0.0250	0.1080	0.0305	0.1315	2.7000	1.4835	0.0006	0.0117
1.2090	0.0780	0.2160	0.0943	0.2611	16.8480	1.4617	0.0061	0.0467
1.1730	0.0375	0.1080	0.0439	0.1267	4.0500	1.3759	0.0014	0.0117
1.1400	0.0605	0.1080	0.0689	0.1231	6.5340	1.2996	0.0037	0.0117
1.0690	0.0780	0.1080	0.0834	0.1155	8.4240	1.1428	0.0061	0.0117
9.5470	**0.4020**	**1.2960**	**0.4733**	**1.5654**	**65.1240**	**11.4325**	**0.0236**	**0.2333**

By 16: $\bar{x}_1 = \dfrac{9.5470}{8} = 1.193375$

By 17: $\bar{x}_2 = \dfrac{0.402}{8} = 0.05025$

By 18: $\bar{x}_3 = \dfrac{12960}{8} = 16200$

By 10: $\sum x_1^2 = 11.4325 - 8(1.193375)^2 = 0.03935$

By 11: $\sum x_2^2 = 0.023551 - 8(0.05025)^2 = 0.0033505$

By 12: $\sum x_3^2 = 23328 \times 10^5 - 8(16200)^2 = 233280000$

By 13: $\sum x_1 X_2 = 0.4733 - 8(1.193375)(0.05025) = -0.00643675$

By 14: $\sum x_1 X_3 = 156535.2 - 8(1.193375)(16200) = 1873.8$

By 15: $\Sigma x_2 X_3 = 6512.4 - 8(0.5025)(16200) = 0$

By 8: $b_{12.3}(0.03935) + b_{13.2}(0) = -0.00643675$

$b_{12.3}(0) + b_{13.2}(23328000) = 1873.0$

$b_{12.3} = -0163576874$

$b_{13.2} = 0.000008032$

By 9: $a_{1.23} = 1.193375 - (-0.163576874)(0.05025) - (0.000008032)(16200)$

$= 1.071476338$

$X_{1r} = 1.0714 - 0.1634 X_2 + 0.000008032 X_3 \longrightarrow A$

Standard Error

By 19:

$$S^2_{1.23} = \frac{0.03935 - (-0.163576874)(-0.00643675) - (0.000008032)(1873.8)}{5}$$

$S_{1.23} = 0.0682 = 6.8\% \longrightarrow B$

APPENDIX 2

Table 7.1:   Experimental data

Y_{u1} (g)	Y_{u2} (g)	Y_u	$Y_{u1}-Y_u$	$Y_{u2}-Y_u$	$(Y_{u1}-Y_u)^2$	$(Y_{u2}-Y_u)^2$	S^2_u
.220000	1.216000	1.218000	0.002000	-0.002000	0.000004	0.000004	0.000008
.068000	1.07000	1.069000	-0.001000	0.001000	0.000001	0.000001	0.000002
.235000	1.287000	1.286000	-0.001000	0.00100	0.000001	0.000001	0.000002
.212000	1.206000	1.209000	0.003000	0.003000	0.000009	0.000009	0.000018
							0.00003

Equations

The mean: $\overline{Y}_u = 1/r \sum_{u=1}^{r} Y_u$ -   1

The dispersion: $S^2_u = 1/r\text{-}1 \sum_{r} (Y_{uv}-Y_u)^2$ -   2

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The sum of dispersion: $\sum_{u=1} S^2_u$ — 3

Maximum dispersion: $S^2_u \, max$ — 4

From table 7.1

$S_u^2 \, max \quad = \quad 0.000018$

By 3: $\sum S_u^2 = \quad 0.00003$

Test

Using G-criteria to ascertain, the possibility of carrying out regression analysis.

$$G\text{-calculated} = \frac{S^2_u max}{\sum S^2_u} = \frac{0.000018}{0.00003} = 0.600$$

$G - tabulated \, (\alpha, N, r,) \cong G(0.05, 4, 2) = 0.768$

$N \quad = \quad$ number of experimental runs

$r \quad = \quad$ number of replicates

$\alpha \quad = \quad$ level of significance.

Since G cal < G tab, at 0.05 level of significance it implies that, regression analysis can be done.

DISPERSION, EXPERIMENTAL ERROR, GENERALISED MODEL, ANALYSIS OF VARIANCE AND ADEQUACY DETERMINATION.

$$S^2_{(u)} = \frac{1}{N} \sum (S^2_u) \qquad - \quad 5$$

$$S_u = \sqrt{S^2_u} \qquad - \quad 6$$

$$b_o = \frac{1}{N} \sum X_o Y_u \qquad - \quad 7$$

$$b_i = \frac{1}{N} \sum (X_i Y_u) \, ; \, I = 1,2 \qquad - \quad 8$$

$$b_{ij} = \frac{1}{N} \sum (X_i, i. Y_u) \, i \neq I \qquad - \quad 9$$

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$$SS_R = \frac{r}{N} (contrast)^2 \qquad - \quad 10$$

$$Contrast = \Sigma (X_0 \bar{Y}_u)^2 \qquad - \quad 11$$

$$SSb_i = \frac{r}{N} \Sigma (X_i Y_u)2 \qquad - \quad 12$$

$$SSb_{ij} = \frac{r}{N} \Sigma (X_{i,j} \bar{Y}_u)^2 \qquad i \neq j \qquad - \quad 13$$

$$SSb_{ij} = \Sigma Y^2_{uv} - \frac{\Sigma (Y_{uv})^2}{N.r} \qquad - \quad 14$$

$$SS_E = SS_T - SS_R \qquad - \quad 15$$

$$Fcal = \frac{Treatment\ mean\ square}{Error\ mean\ square} \qquad - \quad 16$$

$$S^2ad = \frac{r}{N} \Sigma (\bar{Y}_u - Y_u)^2 \qquad - \quad 17$$

$$F\ cal\ (ad) = \frac{S^2ad}{S^2(y)} \qquad - \quad 18$$

$$F\ table\ (anova) = \frac{SS_T}{SS_E} \qquad - \quad 19$$

$$F\ table\ (adequacy) = F(0.05, 4,4) \qquad - \quad 20$$

By 5: Dispersion taken as mean square error $(S^2_{(u)} = \frac{1}{4} \times 0.00003$

$$= 0.0000075$$

By 6: Experimental error; $S_u = \sqrt{0.0000075} = 0.00274$

Experimental design is orthogonal, the regression coefficient are calculated as follows.

The mean effect is estimated by 7.

By 7: $b_0 = \frac{1}{4}[(+1 \times 1.218) + (+1 \times 1.069) + (+1 \times 1.286) + (+1 \times 1.209)]$

$$= 1.1955.$$

The two main effects are estimated by 8

By 8: $b_1 = \frac{1}{4}[(-1.218) + (-1.069) + (-1.286) + (1.209)]$

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$$\frac{}{4}$$

$$= \quad -0.0565$$

By 8: $b_2 = \dfrac{1[(-1.218) + (-1.069) + (+1.286) + (+1.209)]}{4}$

$$= \quad -0.052$$

The one-two factor interaction is estimated by 9

By 9: $b_{12} = \dfrac{1[(+1.218) + (-1.069) + (-1.286) + (+1.209)]}{4}$

$$= \quad 0.018$$

The fitted or Predicted model:

$Y_u = 1.196 - 0.057 X_1 + 0.052X_2 + 0.018 X_{1.2}$ ——————→ A

By using A (fitted model), the predicted values of Y at the four points in the design are generated as follows:

$Y_1 = (1.196 - (0.057)(-1) + 0.052(-1) + 0.018(+1)$ = 1.2185

$Y_2 = (1.196(+1) - 0.0565(+1) + 0.052(-1) + 0.018(+1)$ = 1.0695

$Y_3 = (1.196(+1) - (0.0565)(-1) + 0.052(+1) + 0.018(-1)$ = 1.2865

$Y_4 = (1.196(+1) - (0.0565)(+1) + 0.052(+1) + 0.018(+1)$ = 1.2095

The summary of the mean experimental observations Y_{u1} the estimated fitted values Y_{u1} for the four points, the residual and square of residual are presented in table 7.2.

Table 7.2: The mean experimental observations, the fitted valves, the residuals and the square of residuals.

RUN NO	$\overline{Y}_u$	Y_u (Predicted)	$e_i = (\overline{Y}_u - Y_u)$	$e_i^2 = (\overline{Y}_u - Y_u)^2 \times 10^{-4}$
1	1.2180	1.2185	-0.0005	0.0025
2.	1.0690	1.0695	-0.0005	0.0025
3.	1.286	1.2865	0.0005	0.0025
4.	1.209	1.2095	0.0005	0.0025

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ANOVA TEST FOR THE MODEL

The adequacy of the fitted model is being tasted by this test.

The regression sum of squares for any effect is estimated by

By 12: SSb_1 = $\dfrac{2[(+1.213) + (-1.069) + (1.236) + (1.209)]^2}{4}$ = 0.025538

By 12: SSb_2 = $\dfrac{2[(-1.218) + (-1.069) + (+1.286) + (+1.209)]^2}{4}$ = 0.021632

By 12: SSb_{12} = $\dfrac{2[(+1.218) + (-1.069) + (-1.286) + (1.209)^2]^2}{4}$ = 0.021632

$$SS_R = \underline{\underline{0.049762}}$$

By 14: SST = $(1.220)^2 + (1.068)^2 + \text{-------} + (1.206)^2 - \dfrac{(1.220 + 1.068 + \text{---} + 1.206)^2}{4 \times 2}$

$$J = 0.049792$$

By 15: $0.049792 - 0.049762$ = 0.00003

Table 7.3: ANOVA TABLE

Analysis of variance for replicated 2^2 factorial for Soybean extraction.

Source of variation	Effects	Sum of square	Degree of freedom	Mean of square	Complete d F	Tabulated F 5%	1%
b_1	-0.0565	0.0255	1	0.0255	1656.67	6.16	15.21
b_2	0.052	0.0216	1	0.0216			
b_{12}	0.018	0.0026	1	0.0026			
Error	-	0.0000.3	N(r-1) = 4	0.00000075			
Total		0.0497	r(N-1) = 6				

By 19: F calculated = $\dfrac{0.0497}{0.00003}$ = 1656.67

The adequacy of the overall regression model can be further checked by calculating the dispersion of adequacy for the replicate experiment and compare the magnitude with the variance estimate given by mean square error.

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$$\frac{2}{4}\ (0.000001) \qquad = 0.0000005$$

...sher's test

$$F\ cal(ad) \quad = \quad \frac{0.0000005}{0.0000075} \quad = \quad 0.067$$

...9:   F (table) $(\alpha,\ N,\ N(r-1))\ \longrightarrow\ F\ (.05,\ 4,4,)\qquad = 6.39$

...cision: Fcal $<$ Ftable, it implies the fitted regression model can be regarded ...adequate.

APPENDIX 3

Characteristic of Crude Soybean Oil

1. Refractive index   1.46

2. Specific gravity   0.9

3. Density   -   0.897

4. Iodine value   -   130

5. Saponification   -   192

6. Titer   -   20-32

7. Unsaponification v`alue   -   0.5 – 1.5