PRODUCTION AND ANALYSIS OF CASTOR OIL FROM CASTOR BEAN SEEDS

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MINNA,
NIGER STATE

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A RESEARCH PROJECT
PRESENTED TO

THE CHEMICAL ENGINEERING DEPARTMENT
SCHOOL OF ENGINEERING AND ENGINEERING TECHNOLOGY
FEDERAL UNIVERSITY OF TECHNOLOGY
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A FINAL YEAR PROJECT WORK PRESENTED TO THE DEPARTMENT OF CHEMICAL ENGINEERING IN PARTIAL FULFILLMENT OF THE REQUIREMENT FOR AN AWARD FOR BACHELOR OF ENGINEERING (B.ENG) DEGREE IN CHEMICAL ENGINEERING OF FEDERAL UNIVERSITY OF TECHNOLOGY MINNA, NIGER STATE, NIGERIA

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DECLARATION

I Sani Suleiman Manu of the Department of Chemical Engineering, School of Engineering and Engineering Technology, Federal University of Technology Minna hereby declare that this work was carried out strictly by me under the close supervision of my supervisor Eng Isa Abubakar. All information that has been retrieved from published and unpublished works have been duly acknowledged.

Signature..... Date.....8/12/2005

CERTIFICATION

This is to certify that this project work was supervised and approved by the following persons on behalf of Chemical Engineering Department, Federal University of Technology Minna, Niger State.

Signature

Project Supervisor Date 5/12/05

Eng. Abubarkar Isah

Signature

Head of Department Date.....

Dr. F.A. Aberagba

Signature

External Examiner Date.....

DEDICATION

This project is dedicated to my Mum, Hajiya Hadeezah Sani.

ACKNOWLDGEMENT

All praise is to ALLAH, the LORD of the universe for His care, guidance and protection.

I wish to express my profound gratitude to my able supervisor Engr. Abubarkar sah for his advice and relentless effort in making this course a success. I will like to express my gratitude to the entire staff of Chemical Engineering Department.

will like to use this opportunity to thank my mother, Hajiya Hadeezah Sani, my father Alhaji Sani Isyaku and my Brother Alhaji Idris Sani for their moral and financial support hrough out my academic career. Also my special thanks go to General Ibrahim Badamasi Babangida, Prince Shedrack Akolokwo, and the Staff of Environmental Protection and Control Department of NDDC.

My acknowledgement will not be complete if I forget to express my gratitude to ny friends, specifically D12, and the remaining members of my family.

ABSTRACT

This project was carried out to produce and analyse castor oil from castor bean seed using Soxhlet apparatus. The extracion was done using different time and weight of the castor bean seed. Both physical and chemical properties of the sample were evaluated and when compared to ASTM specification it was confirmed that the property of the oil was within the specification. A 2^2 full factorial design was carried out in this work to investigate the variation of the oil yield with the time of extraction and the weight of the castor seed. The equation obtained from the factorial design was simulated and very small error between the experimental and simulated yield of the oil extracted from the castor seed was discovered.

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CHAPTER ONE

1.0 INTRODUCTION

1.1 GENERAL

The castor bean, Ricinus communis, is a native of tropical Africa cultivated in several varieties for the oil found in its leaves and for its bold foliage (Moshkin, 1986). The stalked leaves consist of usually eight radiating, pointed leaflets with slightly serrated edges and prominent central veins. Many varieties are green, but some are reddish brown. The flowers are green and inconspicuous, but pink or red in the pigmented varieties. Many stamens are near the base and branching pistils are near the top of the flower. The soft-spined fruits containing attractively mottled seeds are distinctive features of the plant. To many people the castor plan is just an overgrown, undesirable weed, and yet it produces one of nature's finest natural oils (Simpson and Ogorzaly, 1986).

The oil extracted from the castor bean already has a growing international market, assured by more than 700 uses, ranging from medicines and cosmetics to replacing petroleum in plastics and lubricants (Nawar, 1996). Castor beans are processed to extract castor oil which is used for medicinal purpose. Ricin (a toxic substance present in the plant) does not partition into the oil because it is water soluble; therefore, castor oil does not contain Ricin, provided that no cross contamination occurred during its production (Fellows, 1996).

Preliminary studies on castor oil packs done at the George Washington School of medicine indicate that they improve immune system functioning (Edgar, 1971). Castor oil is also used in the manufacture of fiber optics, bulletproof glass and bone prostheses. It is indispensable for preventing fuels and lubricants utilized in aircraft and space rockets from freezing at extremely low temperatures. Castor oil is the best substance for producing biodiesel because it is the only one that is soluble in alcohol, and does not require heat and the consequent energy requirement of other vegetable oils in transforming them into fuel (Mario, 2001).

Recent studies and genetic improvement have increased the oil content of the castor bean from 24 to 48 perce[illegible] (Mario, 2001).

Production of oil is carried by the process known as "extraction". Extraction is the term used for an operation in which a constituent of a liquid is transferred to another liquid (solvent) The term solid-liquid extraction is restricted to those situation in which a solid phase is present and includes those operation frequently referred to as leaching, lixiviation and washing. It always involves two steps; The first is the contact of the solvent with the solid to be treated so as to transfer the soluble constituent (solute) to the solvent and the second is the separation or washing of the solution form the residual solid. These two steps may be conducted in separate equipment or in the same piece of equipment (Brown, 1956).

A chemical and physical property of the castor oil is used to both identify and quantitatively characterize these oils. Iodine value is one of the important differentiating properties, and this is easily modeled from infrared measurement (Cook and Billingham, 1999) Saponification value, free fatty acids, trans-unsaturation and peroxide value that are measurable from IR (infrared) spectra are important parameters for the food industry. Internal Reflection is an excellent method, because it provides as ATR spectrum with ideal intensities for both qualitative and quantitative measurement (voor et al, 1999).

1.2 AIM AND OBJECTIVE

The aim of this project is to extract the oil from castor bean seed and to determine its chemical and physical properties. This aim will be achieved with the aid of the following objectives:

i. Extraction of the oil from the castor seed using the soxhlet apparatus with the aid of the solvent.

ii. Characterization and Refining of the castor oil.

iii. Evaluation of the castor oil produced using 2^k factorial experimental design.

1.3 SCOPE OF WORK

This work is limited to extracting oil from castor bean seed using soxhlet apparatus by varying the mass of the castor bean seed and the quantity of the solvent for different time of extraction to investigation the effect of the variations.

In addition, the castor oil to be produced will be analyzed and, thereafter, evaluated using 2^k factorial experiment design.

1.4 JUSTIFICATION

Obviously, castor oil and its derivatives has become an important commodity and is again a topic of interest to the chemical industry. For instance, the uses of castor oil have changed over the years. Sixty years ago, castor oil was used for medicinal purposes and as a general industrial lubricant. Soon after, chemical engineers were able to produce derivative of the oil that were of even more benefit to man

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 HISTORY AND SCIENTIFIC CLASSIFICATION OF CASTOR BEAN

The Castor bean (Ricinus Communis) is not a true bean, but e

Euphorbiacae or spurge family. It is the source of castor oil, which uses, and ricin, a poison. The name Ricinus is a Latin word for tick because it has markings and a bump at the end, which resembles cer, and is the specific epithet for Mediterranean ship tick (Ixodes ricinus). Apparently Linnaeas though the seed looked like ticks, particularly large tick engorged with blood (Weiss, 1971). "Communis" means common in Latin and castor were already commonly naturalist Corolus Linnaeas (Karivon linne) was given scientific first and last name of plants and animals over 200 years age.

It is interesting to trace the origin of the name " castor" Castor is a generic name of North American beaver (Castor Canadensis), one of the brightest double star in the constellation gemini in Greek and Roman Legend, Castor was one of the twin sons of Jupiter and Leda. According to E.A Weiss written in castor seame and Luminous star, or offspring of Greek and Roman Gods. Castor was apparently coined by English traders who confused with the oil by another shrub, vitex agnus – castus which the Spanish and Portuguese in Jamaica called "agno-castor". Although it is commonly know as castor bean plant. There are many others example of "bean" that are technically beans such as Mexican jumping "bean" and coffee bean.

Another castor plant is probably indigenous to Easter Africa and has become naturalized in tropical and warm temperate region throughout the world and is becoming increasingly abundant weed in the South – Western United States. Castor establishes itself easily as a "native" plant common along stream banks, near railroad, river beds, bottom land, and just about any hot areas where the soil is well drained with sufficient nutrients and moisture to sustain the vigorous growth. Although the beans are extremely poisonous, they are the source of numerous economically important products and are one of earliest commercial products. Castor bean has been found in ancient Egyptian tombs dating back to 400 BC and the oil was sued thousands years ago in wick lamps for lighting. To many

people, the castor bean plan is just an overgrowing undesirable weed, and yet it produces one of nature's finest natural oil (Vignolo and Naughton, 1991).

Castor plant growing along the edge of San Elijo Lagoon in Coastal San Diego Country, California. The castor plant in robust – annual that may grow 6-15 feet (2-5 meters) in seasons with full sunlight, head and adequate moisture, in areas with mild frost- free winter. At present more castor plant is grown in South American than any where else, Brazil alone producing 300,000 – 400,000 tones annually. Other castor producing countries include central Asia, Tanzania, Southern Kazakhstan, Indian, Thailand and Egypt. (Frank, 1974).

FIG. 2.1: Castor bean plant showing large, tropical, palmately- lobed leaf and cluster of spiny red fruits

2.1.1. CASTOR BEAN FLOWERS AND SEEDS

Flowers occur most of the year in dense terminal clusters (inflorescences), with female flowers just above the male flowers. This species is clearly monoecious, with separate male and female flowers on the same individual. There are no petals and each female flower consists of a little spring ovary (which develop in to the fruit or seed capsule) and a bright red structure with feathery branches (stigma lobes) that receives pollen from male flowers. Each male flower consists of a cluster of many stamens which literally smoke as they shed pollen in a gust of wind (Robinson, 1964)

FIG 2.2: Flower cluster (inflorescence) of castor bean (ricinus communis). The upper spiny ball (ovaries) with red, star shaped stigmas are the female flowers. The lower

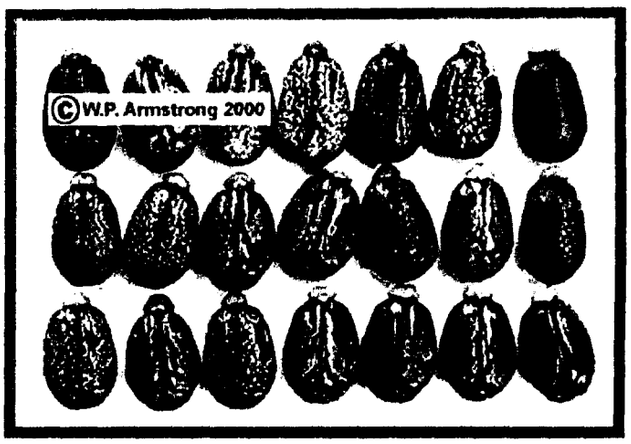
male buds open into whitish-yellow clusters of stamens the wind-pollinated flower have no petals.

The spiny seed pod or capsule is composed of three sections or carpels which split apart at maturity. Each section sections (carpel) contain a single seed, and as the carpel dries and splits open, the seed is often ejected with considerable force. Walking among large castor shrubs on a hot summer day can be quite an experience, with the sound of exploding carpels and seeds flying through the air and bouncing off road signs, side walks and yar head (Simpson and Ogorzaly, 1986).

FIG 2.3: Castor bean fruit (ricinus communis): the spiny, globose seed capsule (left) dries and splits into 3 sections called carpels (center). Each carpel (right) splits open and forcibly ejects a large seed. in the related mexican jumping bean (sebastiana pavoniana), a moth occupies each carpel and feeds on the seed tissue inside.

The spiny seeds of castor plants are a little larger than pinto beans and have very beautiful and intricate designs. At one end is a small, spongy structure called the caruncle, which aids in the absorption of water and the seeds are planted, like human faces, finger prints or the spots on a leopard, on two seeds have exactly the same pattern. They are unquestionably among the most deadly seeds on earth, and it is their irresistible appearance that makes them so dangerous (Windholz et al, 1983).

FIG. 2.4: The many "faces" of castor seeds. Like the faces and fingerprints of people, the beautiful designs on castor seeds exhibit infinite genetic variation. The small structure on the end of each seed is a caruncle. The seeds superficially resemble the bodies of ticks, particularly ticks engorged with blood.



2.1.2: TOXICITY OF CASTOR BEAN SEED AND TOXIC PRINCIPLE

Ricin is poisouons if inhaled, or ingested as a toxin by the inhibition of protein synthesis there is no known non- antidote, only symptomatic and supportive treatment is available. In small doses such as typical doses contain a measure of castor oil, ricin causes digestive cramps.

A principal toxic of castor seed is ricin, which is a Latin also termed a taxolblumin. Ricin may comprise up to 3% of seed weight, taxolblumin are very toxic plant derived compound that combine carbohydrate and protein moieties or components, Ricin is water- soluble and is not present in castor oil. Another phytotoxine in castor seed, riciminel is reportedly goitrogenic but the significance of this compound is not clearly established (Lewis, 1977).

2.2: CASTOR CAKE

Castor cake is a residue of castor seed after the oil have been extracted, the cake contains 8-10% oil. Castor cake are used as an ingredient in some animals feeds after the oil has been extracted or in an activated by heating for 20 minutes at 14^0C. Attempt to use castor cake in feed for livestock involve different method of inactivation ricin which maintaining nutritional value. The castor cake can be used as organic manure for agricultural crops, the castor meal contain about 5.6-6% Nitrogen, 2.5% phosphorus, 1-1.5% potassium on a dry weight basis. The overall protein content of castor cake is about 40% (Weiss, 1983).

2.3 CASTOR OIL

The uses of castor oil have changed over the years. Sixty years ago, castor oil was used for medicinal purposes and as a general industrial lubricant. Soon after, chemical engineers were able to produce derivatives of the oil that were of even more benefit to man. Sulphonated (sulfated) castor oil, or Turkey red oil, was the first synthetic detergent after ordinary soap, and other forms of the oil became important for the treatment of leather, industrial lubricants, and other industrial uses. Today, chemical engineers have come up with many uses of castor oil and its derivatives such as polyamide 11 (Nglon 11) engineering plastics, lubricating grease, coatings, inks, sealant, aircrafts lubricants, surfactants, emulsifiers, encapsulants, plastic films, plasticizer for coatings and components for shatter proof safety glass. Castor oil has even made its way into cosmetics and related products due to its none comedogenicity (does not exacerbate skin or contribute to acne). (Vignolo, 1991). Obviously, castor oil and its derivatives has become an important commodity and are again a topic of interest to the chemical industry. (Fellows, 1996)

2.3.1 EXTRACTION OF OIL

Analysis usually requires transfer to liquid or gas phase. Most methods are based on liquid solid-extraction. Classical method is soxhlet extraction.

Soxhlet apparatus provides multiple extractions in a simple, unattended process. Solvent is delivered by distillati . o sample in a filter chamber. The chamber fills to a

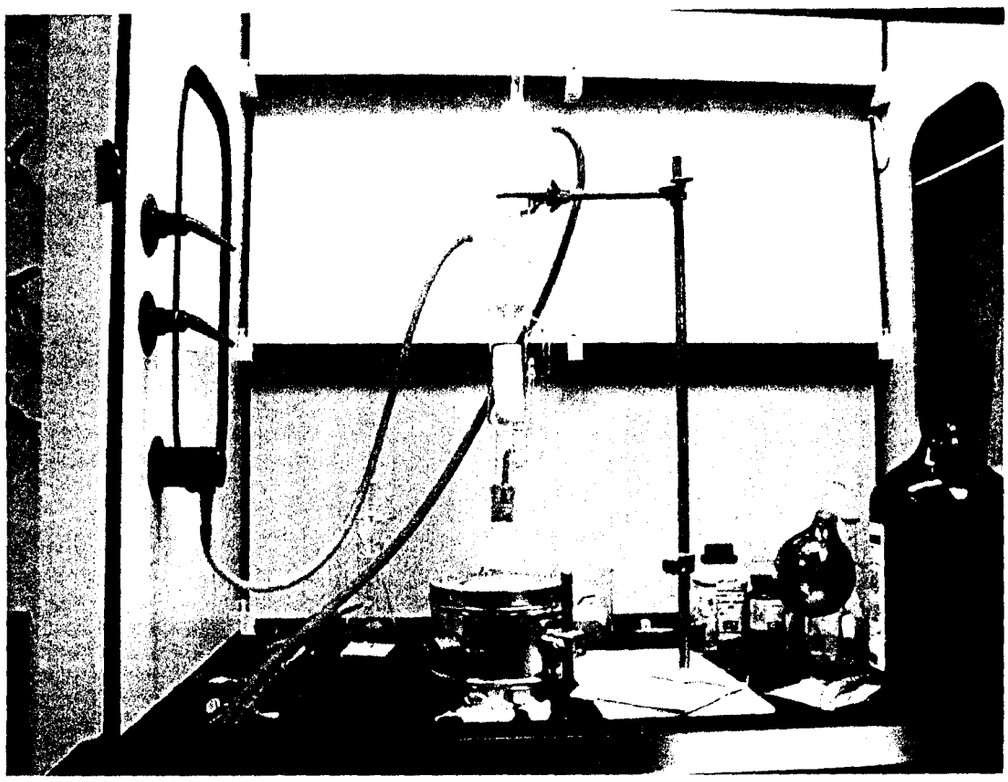
maximum level and then autodrains into the solvent reservoir at the bottom of the distillation apparatus. Extracted compound are gradually transferred to the solvent reservoir over the course of many fill / auto-drain cycles (Nawar, 1996).

2.3.1.1 SOXHLET APPARATUS

The procedures for the extraction of oil from oil seeds are as outlined below.

1. Solvent in the round bottomed flask is heated to boiling
2. The vapors rose through the outer chamber and into the condenser.
3. The vapors condenses into liquid and fall back into the bottom of the soxhlet chamber.
4. As the distilled solvent rises in the chamber it seeps through the permeable cellulose extraction thimble that holds the Masu leaves.
5. The solvent extracts the compounds of interest and leaves the solid mass behind. The extraction is usually indicated by observing that the solvent has a different colour than it has in its pure form in the flask.
6. As the solvent level rises, the solution is forced through the small inner tube, and the chamber is flushed due to a siphoning effect.
7. The flushed solvent returns to the flask taking the extracted compounds with it.
8. The solvent is redistilled from the solution in the flask and condenses in the chamber, repeating the extraction with fresh solvent. The process can be repeated as many times as possible.
9. Each time the process is repeated, the more concentrated the solution in the flask becomes because more is being extracted from the solid mass.
10. The Soxhlet extraction is usually completed when the solution in the soxhlet chamber is the same color as the pure solvent. This means that nothing more is being extracted from the leaves by the solvent.

FIG. 2.5: Soxhlet apparatus set up.



2.3.1.2. OIL EXPRESSION TECHNIQUE

Castor oil can be derived from the seed of Ricinus communis L. which grows in tropical or subtropical regions such as control Asia, Brazil, Tanzania and southern Kazakhstan to name a few. It occurs as perennial or annual plant and is considered a drought resistant crop in India (Moshkin, 1986). Unfortunately in 1972, economic pressures created circumstances, which led to the United State losing its domestic supply of castor oil, and the US became dependent on foreign countries for both the seed and the oil. As of 1991, any castor seed produced in US has to be shipped to Mexico for extraction of the oil from the seed (Browning, 1991), As a result, The US is many years behind in the expression technology. However, The extraction of the oil from this seed is done in a similar manner to most other oil seeds. The seeds are collected when ripe: as the capsules dry, they open and discharge the seeds cooked and dried prior to extraction. Cooking is done in order to coagulate protein, which is necessary to permit efficient extraction, and to free the oil for sufficient pressing. It is done at 80^0C, under airtight conditions. After cooking, the material is dried at 100^0C, to reach a moisture content of approximately 4% (Sleggs, 199[illegible]).

First stage of extraction is pre-pressing using a high pressure continuous screw press- expeller. The expeller usually consists of a barrel containing a stainless steel belical screw. The pitch of the screw flights gradually decreases towards the discharge end, to increase the pressure on the pulp as it carried through the barrel (Fellows 1996). Extracted oil is filtered, and collected in a settling tank. Material re moved from the oil, called foot, is fed back into the stream of fresh material. Material discharged from the press, called cake, contains 8% to 10% oil. It is crushed into coarse meal, and subjected to solvent extraction with hexane or heptanes. Continuous processing is used based on the principle of counter flow of solvent and oil-bearing material. The oil is removed effectively, as the material comes into contact with increasingly purer solvent. After extraction, solvent is removed by distillation and then resulting oil is processed in a similar manner as oil from the pressing step (Weiss, 1971).

FIG 2.6: The serew press diagram.

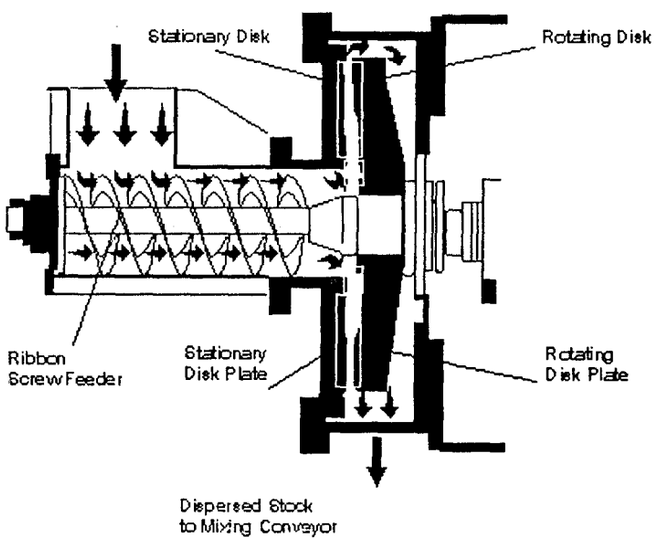
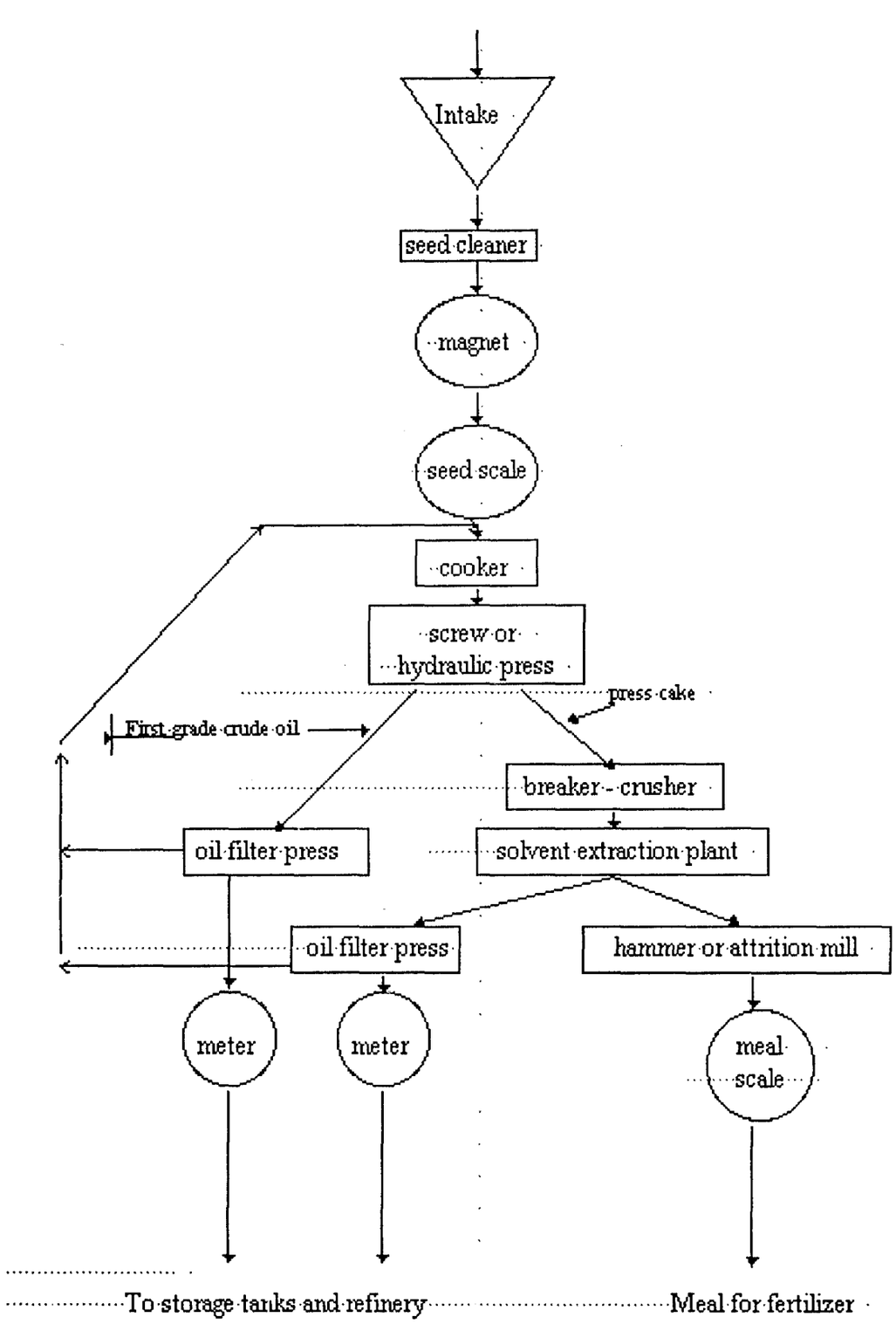


FIG 2.7: Flow sheet for castor seed oil extraction with pre-pressing and solvent extraction.



2.3.1.3. OIL PURIFICATION METHOD.

Once the oil has been expressed from the seed, it is necessary to remove any impurities from the oil that makes it such an important commodity. The oil is essentially a pure triglyceride, and contains 90% of glyceryl tricinoleate. It is the ricinoleic triglyceride that is needed in order to produce high quality castor oil that will be used for the chemical reactions. Characterizing properties of castor oil include a higher density, viscosity and reactivity than common triglycerides found in the other vegetable oil. These properties are exploited when refining the oil from the impurities. The steps to refining the crude oil

include settling and degumming of oil, bleaching, neutralization and deodorization of the oil (Nawar, 1996). The settling and degumming of the crude oil is done to remove the aqueous phase from the lipid and to remove phospholipids from the oil. Bleaching of the oil resects in the removal of colouring materials and the removal of phosphor lipids and oxidation product due to adsorption of the impurities to neutral clay. Care must be take because a highly acid activated clay can react with the oil and cause an undesirable dehydration reaction. Neutralization can be done in one or two ways; by alkali (chemical) or steam stripping (physical) means. The neutralization step is necessary to remove free fatly acids from the oil. Caustic soda (alkali) is mixed in proper amounts and the aqueous solution (called soap stock) is removed, leaving the neutral oil behind. Unfortunately, the use of alkali to neutralize the oil results in poor soap stock separation and high neutral oil losses. This is why steam stripping is preferable. Steam stripping is done under vacuum to remove moisture, free fatly, acids, odour bodies and other impurities from the oil. Because it is performed under vacuum conditions, the oil can be kept at a low temperature, preserving its chemical structure and not subjecting it to temperature in which undesirable dehydration reaction can occur. (Windholz et al, 1983)

2.4 USES OF CASTOR OIL

The chemical structure of castor oil is of great interest because of wide range of reactions it affords to the oleochemical industry and the unique chemicals that can be derived from it. These derivatives are on par with petrochemical products for use in several industrial applications. Infact they are considerably superior since they are form renewable sources, biodegradable and eco-friendly. Castor oil is regarded as one of the most valuable laxatives in medicine, castor oil forms a clean, and light- colored soap, which dries and hardens well and is free from smell. Externally, the oil has been recommended for various cutanous complaints. Castor oil is an excellent solvent of pure alkaloids and such solutions of Atropine, cocaine, and e.t.c, as are used in ophthalmic surgery. It is an essential component in some artificial rubbers, in various descriptions of celluloid, and in the making of certain water proof preparations, and one of the largest use is in the manufacture of transparent soaps. It also furnishes sebacic acid that is employed

in the manufacture of candles, and caprylic acid, which enters into the composition of varnishes. (Schery, 1972)

2.4.1 CASTOR OIL IN PAINTS

The castor plant has many uses, particularly the thick, yellowish or almost colorless oil obtained from the seeds. There are an astonishing number of industrial applications for castor oil and its derivatives, and new ones are continually being discovered. When dehydrated, castor oil is converted into quick-drying oil used extensively in paints and varnishes. In fact, one of the largest single markets of castor oil in the United States is the paint and varnish industry. Its water-resistant qualities make it ideal for coating fabrics and for protective coverings, insulation, food containers and guns (Browning, 1991).

2.4.2 CASTOR OIL IN NYLON

Castor oil is the primary raw material for the production of sebacic acid, which is the basic ingredient in the production of nylon and other synthetic resins and fibers. Approximately three tons of castor oil is necessary to produce one of nylon. Sebacic acid is a 10-carbon dicarboxylic acid with a carboxylic group (C – OOH) at each end of the molecule. It is reacted with 1,6-haxenediamine, a 6 –carbon molecule with an amino group ($C\text{-}NH_2$) at each end. The free carboxylic and amino ends of these molecules begin bonding together in a chain reaction called condensation polymerization, in which a water molecule is produced at each link. The resulting nylon polymer is called Nylon 6,10 to denote the 6–carbon diamine and 10 – carbon sebacic acid (Hill, 1952)

2.4.3 CASTOR BEAN MOTOR OIL

The superior "oiliness" of castor oil and its ability to "Cling" to very hot moving parts makes it outstanding racing oil for high performance engines. Infact it is the basic ingredient of castor–R racing motor oil for high-speed automobile and motorcycle engines. Castor oil is a popular fuel additive for two cycle engines and imparts a distinctive aroma to the exhaust of these engines.

2.4.4 FRUIT FLAVOURS FROM CASTOR OIL

Although castor oil is rather malodorous and distasteful, it is source of several synthetic flower scents and fruit flavours (esters), such as jasmine, apricot, peach, plum,

rose, banana, and lemon. The chemicals (esters) responsible for these flavors and aromas are obtained from ricinoleic acid, one of the important ingredients of natural castor oil. Castor oil is also used in making soaps, inks, and plastics, for preserving leather; as an illuminant; in turkey red oil for dyeing and finishing textiles, and in brake fluids and certain insecticidal oils. Even after the oil has been removed, the poisonous crushed seeds or oil cake (pomace) makes an excellent fertilizer.

(Vignolo and Naughton, 1991).

2.4.5. OIL PACKAGING AND SHELF LIFE

The castor oil contains double bonds in its lipid structure, it is prone to an undesirable reaction called lipid oxidation. Lipid oxidation occurs when the double bonds in the fatty acid react with oxygen to form peroxides and change the chemical nature of the oil. There are many factors which influence the rate of oxidation in foods: fatty acid composition, free falty acids versos the corresponding acylglyeerols, oxygen concentration, temperature, pro-oxidants, radiant energy (Visible and Ultraviolet light), and the presence of antioxidants. In generally the oil is stored in a controlled environment. That includes removing oxygen, storing the oil in a cool place, placing the oil in an opaque contain, removal of pro-oxidants (e.g. cobalt, copper, iron, manganese and nickel) and possibly adding antioxidants (Nawar, 1996). Castor oil is not as prone to oxidation as much as other oils unless exposed to high temperatures (Weiss, 1971).

2.5 CHEMISTRY OF PLANT OILS

Plant oils are typically composed of triglyceride molecules (technically esters) composed of a 3-carbon alcohol (glycerol) plus three 18-carbon (or 16-carbon) fatty acids, unlike the saturated fatty acids of animal fats which are solid at room temperature,plant fatty acids are typically unsaturated and liquid at room temperature, with one or more double bonds between the carbon atoms (monounsaturated and polyunsaturated). (Note: the palm fatty acid palmitin is saturated and contains 16 rather than 18 carbon atoms). Below is the structure of a typical plant fat molecule (triglyceride) composed of glycerol plus 3 fatty acids. Since it contains unsaturated fatty acid it is liquid at room temperature and is often referred to as oil. The fatty acids may be saturated (with

all single bonds), mono saturated (with one double bond) or poly unsaturated (with 2 or more double bonds) (Lewis and Elvin – Lewis, 1977).

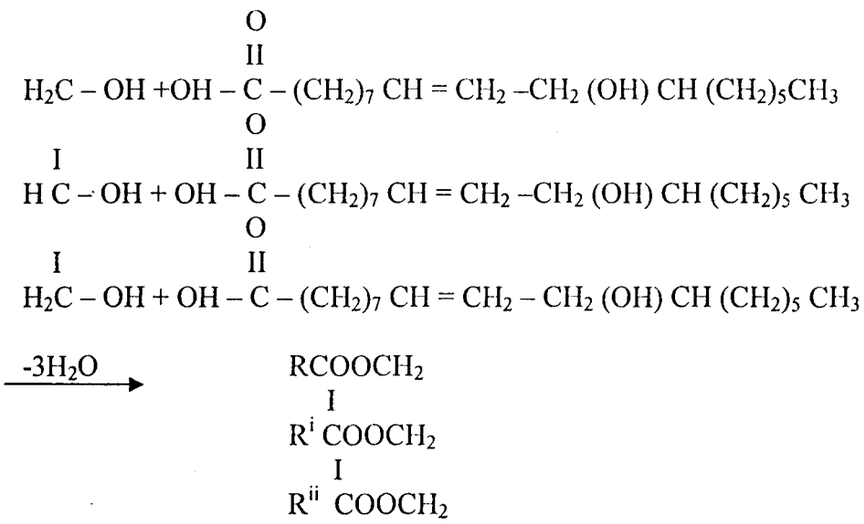


TABLE 2.1: **CONSTITUENTS OF CASTOR SEEDS**

MATERIAL	PERCENTANCE %
FATTY	42 – 55
PROTEINS	20 – 25
LECTINS	0.1 – 0.7
RICINS	3 – 4

TABLES 2.2: FREE FATTY ACID COMPOSITION OF CASTOR OIL

RICINOLEIC	89. 5%
LINOLEIC	4.2%
OLEIC	3.0%
STEARIC	1.0%
DIHYDROSTEARIC	0.7%
LINOLENIC	0.3%
PALMETIC	1.0%
ELCOSANONIC	0.3%

TABLE 2.3: INTENATIONAL CASTOR OIL SPECIFICATION (ASTM) STANDARD.

CONSTANTS	RANGES	SELECTED.
SAPONICATION VALUE	176 – 187	181
HYDROXLY VALUE	161 – 169	MINIMUM 160
FREE FATTY ACID VALUE	0.4 – 4.0	MAXIMUM 3
COLOUR (GADNER)	NOT DARKER THAN 2-3	MAXIMUM 3
SPECIFIC GRAVITY	0.958-0.968	0.96
REFRACTIVE INDEX 20/25 ^{0}C	1.473 -1.477	-
VISCOSITY AT 25 ^{0}C	6.3 – 8.8 ST	-
BOINLING POINT	3.3^{0}C	-
SOLUBILITY IN WATER	VERY POOR	-
I0DINE VALUE	81 – 91	85

(Weiss 1983).

2.6 ANALYSIS OF OIL VIA CHEMICAL AND PHYSICAL PROPERTIES

Chemical and physical properties have been in the analysis of castor oil. These include the iodine value, acid value, saponification value, pH value, refractive index, viscosity, specific gravity, boiling point and melting point.

2.6.1 CHEMICAL PROPERTIES

2.6.1.1 SAPONIFICATION VALUE

This is the number of milligrams of potassium hydroxide required to neutralize the falty acid resulting complete hydrology of 1g of substance.

2.6.1.2 IODINE VALUE

Iodine value (IV) is a measure of the total number of double bonds present in fats and oils. It is generally expressed in terms of "number of grams of iodine that will react with the double bonds in 100 grams of fats or oils". A high IV oil contains a greater number of

double bonds than a low IV oil. Edible oil with high iodine value are usually less stable and more susceptible to oxidation (Bruker 2005).

In other words, this is the measure of unsaturation of fatty acids of their esters. It is the number of grams of iodine absorbed by 100g of the sample and expressed the weight of the iodine. The value measured determines the average degree of unsaturation of the oil or fat. The higher the value, the higher the degree of unsaturation of the oil (Ishaq, 2004)

2.6.1.3 ACID VALUE

This is the number of milligrams of potassium hydroxide required to neutralize the free fatty acid present in the oil or fat. Castor oil contains enzymes which cause glycerides to hydrolyse and decompose into free fatty acid glycerin.

2.6.1.4 pH VALUE

This is the degree of acidity and alkalinity of the oil at room temperature using pH electrode to determine the degree of acidity castor oil (Ishaq.2004)

2.6.2 PHYSICAL PROPERTIES

2.6.2.1 VISCOSITY

This measures the relative case, which the oil close conducts or allows particles to move through it. It is measured using bubble tubes, falling sphere and efflux and torsion viscometer (Ishaq.2004).

2.6.2.2 REFRACTIVE INDEX (N)

This is the ration of the velocity of light in vacuum to its velocity in the substance; it varies with the wavelength of the light used in the measurement. It is also temperature dependant and is usually measured at 40^0C, the temperature at which most oil and fat are liquid (Ishaq, 2004).

2.6.2.3 SPECIFIC GRAVITY

Specific gravity or relative density is defined as a density of substance relative to that of water and this measures the comparativeness of the sample relative to that water (Ishaq, 2004).

2.7 REFINING

Once the oil has been extracted from the seed, it is necessary to remove any impurities from the oil that makes it such an important commodity. Characterization properties of the castor oil include a high density, viscosity, boiling point and reactivity than common triglycerides found in other vegetable oil. The steps to refine the castor oil include settling and de – gumming of the oil, bleaching, neutralization, deodorization of the oil (Nawar, 1996).

2.7.1 DE GUMMING

Castor oil contains a quality of natural gum and phosphotides. These gum is a good emulsifying agent, and if left in the oil would give rise to a serious loss of oil at the next stage of the process. It is usually treated with water to recover commercial amount, which is widely used as an emulsifier. Boiled water or steam is sprayed on the oil and it is agitated and allowed to come to rest, the gum with water will settle to the buttom of the vessel and de-gumming process is a partial refining since free-acid is not reduced and even the gum is not completely removed. A more de-gumming may be obtained by adding a little concentrated phosphoric acid to the oil before mixing. These removes most of the so-called "non hydratable phosphotides."

2.7.2 BLEACHING

Bleaching of the oil results in removal of colouring materials and the removal of phospholipids and oxidization products due to adsorption of impurities to neutral clay, it is sometimes called decolorization. Care must be taken because highly acid activated clay can react with the oil and cause an undesirable dehydration.

2.7.3 NEUTRALIZATION

Neutralization can be done in two ways: By alkali (chemical) or steam striking (physical) means. The neutralization step is necessary to remove free fatty acids from the oil. Caustic soda (alkali) is mixed in proper amount and aqueous solution (called soap stock) is removed leaving the neutral oil behind.

2.8 EXPERIMENTAL DESIGN

2.8.1. BASIC PRICIPLES OF EXPERIMENTAL DESIGN

If an experiment is to be performed most efficiently, then a scientific approach to planning the experiment must be considered. By statistical design of experiments, we referred to the process of planning the experiment so that appropriate data will be collected which may be analyzed by statistical method resulting in valid objective conclusions. The statistical approach to experimental design is necessary if we wish to draw meaningful conclusions from the data. When the problem involves data that are subjected to experimental errors, Statistical methology is the only objective approach to analysis. Thus, there are two aspects to any experimental problem, the design of the experiment and the statistical analysis of the data. These two subjects are closely related, since the method of analysis depends directly on the design employed (Isah, 2004).

The two basic principles of experimental design are replication and randomization. By replication, we mean a repetition of the basic experiment. If a treatment is alloted to 'r' experimental units in an experiment, it is said to be replicated r times. If in a design each of the treatments is replicated r times, the design is said to have r replication. Replication is necessary to increase the accuracy of estimates of the treatment effects. It also provides an estimate of error variance which is a function of the difference among observations from experimental units under identical treatment. Although the more the number of replications, the better it is, so far as precision of estimates is concerned, it can not be increased indefinitely as it increases cost of experimentation (Isah,2004).

Randomization is the cornerstone underlying the use of statistical methods in experimental design. By randomization, we mean both the allocation of the experimental material and the order in which the individual runs or trials of the experiment are to be performed are randomly determined. Statistical methods require that the observations (or errors) are independently distributed random variables. Randomization usually made this assumption valid. By properly randomization of the experiment, we will also assist in 'averaging out' the effect of extraneous factors that may be present (Isah, 2001).

In order to use the statiscal approach to designing and analyzing an experiment, it is necessary that everyone involved in the experiment has a clear idea in advance of exactly what is to be studied, how the data is collected and at least a qualitative understanding of how this data is to be analyzed. The recommended procedures are outlined below:

1. Recognition and statement of the problem: This may seem to be a rather obvious point but in practice it is often not simple to realize that a problem requiring experimentation exists, and to develop a clear and generally accepted statement of this problem. It is necessary to develop all ideas about the objective of the experiment, a clear statement of the problem often contributes substantially to a better understanding of the phenomena and the final solution of the problem.
2. Choice of factors and levels: The experimenter must select the independent variables or factors to be investigated in the experiment. The factors in an experiment may either be quantitative or qualitative. If they are quantitative, thought should be given as to how these factors are to be controlled at the desired values and measured. We must also select the values or levels of the factors to be used in the experiment. These levels may be chosen specifically or selected at random from the set of all possible factors levels (Isah 2001).
3. Selection of response variable: In choosing response or independent variable, the experiment must be certain that the response to be measured really provides information about the problem under study. Thought must be given to how the response will be measured, and the probable accuracy of the measurements.
4. Choice of experiment design: This step is of primary importance in the experiment process. The experimenter must determine the difference in true response he wishes to detect and the magnitude of the risk he is willing to tolerate so that an appropriate sample size (number of replications) may be chosen. He must also determine the order in which the data will be collected and the method of randomization to be employed. It is also necessary to maintain a balance between statistical accuracy and cost. Most recommended expericient designs are both statically efficient and economical, so that the experiment effort to obtain statiscal accuracy usually result in economics efficiency.

A mathematical model for the experiment must also be proposed, so that statistical analysis of the data may be performed.

5. Performing the experiment: This is the actual collection process. The experimenter should carefully monitor the process of the experiment to ensure that it is proceeding according to the plan. Particular attention should be paid to randomization, measurement accuracy, and maintaining a uniform experiment environment as possible.
6. Data Analysis: Statistical method should be employed in analyzing the data from the experiment. Numerical accuracy is an important factor here, although present day computers have largely relieved the experiment from this problem, and simultaneously reduced the computational burden. Graphical methods are also frequently useful in the analysis process.
7. Conclusion and recommendation: Once the data have been analyzed, the experimenter may draw conclusions or inferences about his results. The statiscal inferences must be physically interpreted, and the practical significance of those findings evaluated. Then recommendations concerning these findings must be made. These may include a further round of experiments, as experimentation is usually an iterative process, with one experiment answering some questions and simultaneously posing others (Montgomery, 1976).

2.8.2 FACTORIAL EXPERIMENT

Factorial experiment involves simultaneously more than one factor each at two or more levels. If the number of levels of each factor in an experiment is the same, the experiment is called symmetrical factorial; otherwise it is called asymmetrical factorial or sometimes called mixed factorial. These experiments provide an opportunity to study not only the individual effects of each factor but also their interactions. When the experiments are conducted factor by factor, changing the levels of one factor at a time and keeping the other factor constant, the effect of interaction can not be investigational. In many biological and clinical trials, factors are likely to have interactions. Therefore factorial types of experiment are more informative in such investigations. They have the further

advantage of economizing experimental resources. When experiments are conducted factor by factor, much more resources are required for some precisions than when they are tried in factorial design (Das and Giri, 1979).

2.8.3 FULL FACTORIAL EXPERIMENT.

There are several special cases of general factorial designs that are important because they are widely used in research work and also they form the basis of other designs of considerably practical values. The fist of these special cases is that of two factors, each at only two levels. These levels may be quantitative or qualitative, such as two values of temperature, weight, pressure or time, or they may be qualitative such as two machines, two operators, the "high" and "low" levels of factors or perhaps the presence and absence of a factor. Such design requires a 2x 2x2.....$^{x}2 = 2^{k}$ observations and is called a 2k factorial design. The special second case is that of k factors, each at three levels, which is called 3k factorial design (Montgomery,1976).

A 2k factorial design requires us to choose just two levels of each factor and then calls for simulation runs at each of 2k possible combination of factor levels. Usually we use a minus sign with one level of a factor and a plus sign with the other level. Which sign is associated with which levels is quite arbitrary, although the quantitative factors are less confusing if we associate the minus sign with the lower numerical value. No general prescription can be given for how one should specify the levels (Averill and kelton, 1996).

2.8.4 A GENERAL 2K DESIGN

Before any 2k factorial could be analyzed, G-test is used to check if the out put factors have the maximum accuracy of the replication. It ascertains the possibility of carrying out the regression analysis. The condition of homogeneity is;

$$G[\alpha,(r-1), N] > G \text{ cal}$$

$$\text{Here G-cal} = \frac{Su^2 \max}{\Sigma^{2k} Su^2} \qquad \text{2.1}$$

The value of Su^2 Max. and Σ^{2k} Su^2 are gotten from the table of response and their replicate.

The method of analysis that we have we have presented so far may be generalized to the case of a 2^k factorial design. If the coded factors are X_1, X_2, X_{12}, e.t.c. The regression coefficient for response yi, may be calculated using the general formular:

$$bo = 1/2^k \sum_{i=1}^{2k} yi \quad \text{_____ 2.2}$$

$$bj = 1/2^k \sum_{i=1}^{2k} (Siyi) \quad \text{_____ 2.3}$$

The significance of coefficient of the ression model could be tested using the individual F-test. We use F-test by rejecting the null hypothesis,

Ho: bj = O _____ 2. 4

When F cal > F [α, dfR, N(r -1)] _____ 2. 5

a coefficient is significant

$$F\ cal = MS_R = \frac{SS_R}{df_R} \Big/ \frac{SS_E}{N(r-1)} \quad \text{_____ 2. 6}$$

The sum of squares for any contrast can be computed from equation 2.6, thus

$$SS_R = SS_{bj} = \frac{(r.\ contrast)^2}{r.\ N} \quad \text{_____ 2.7}$$

The total sum of squares is found in the usual way by,

$$SS_T = \sum_{i=1}^{r} (yr\ i)^2 - \frac{\left[\sum_{i=1}^{r} yr\right]^2}{r.\ N} \quad \text{_____ 2.8}$$

and

$$SS_E = SS_T - \sum SS_R \quad \text{_____ 2.9}$$

2.8.5 A 2^2 DESIGN

The first design in the 2^k series is one with only two factors say A and B, each run at two levels. This design is the simplest case of 2^k series and is called a 2^2 factorial design. The levels of the factor may be arbitrarily called "Low" and "High". The treatment combination and response of this design is displayed below:

TABLE 2. 4

Treatment combination and response for 2^2 designs

Treatment combination	Response, R
A Low, B Low	R_1
A high, B Low	R_2
A Low, B high	R_3
A high, B high	R_4

By convention we denote the effect of a factor by a capital latin letter. Thus "A' refers to the effect of factor A. "B" refers to the effect of factor B and "AB" refers to the AB interaction. In the 2^2 design, the low and high levels of A and B are denoted by O and 1 respectively on the A and B axis

(Isah, 2001).

The coordinates of the vertices of the square also represent the four treatment combinations as follows: OO represent both factors at low level, 10 represents A at the high level and B at the low level, 01 represents A at low level and B at the high level, 11 represent both factors at the high level. These treatment combinations are usually represented by lower case letters, as shown in table 2.4, we can see from the figure that the corresponding lower case letter denotes the high level of any factor in the treatment combination, and the low level of a factor in the treatment is denoted by the absence of the corresponding letter. Thus "a" represent the treatment combination of A at the high level and B at the low level, "b" represents A at low level, and B at high level, and "ab" represents both factors at high level. By convention, "1" is used to denote both factors at the low level

(Isah, 2001).

It is often convenient to write down the treatment combinations in order "1", "a", "b", "ab". This is referred to as standard order.

Table 2.5 THE CONTRAST COEFFICIENT FOR ESTIMATING EFFECT

A	(1)	a	b	Ab
A:	- 1	+1	-1	+1
B:	-1	-1	+1	+1
AB:	+1	-1	-1	+1

Note that the contrast coefficients for estimating the interaction effects are just the product of the corresponding coefficients for the two main effects. The contrast coefficient is always +1 and –1, and a table of plus and minus signs such as in Table 2.5 can be used to determine the proper treatment combination. The column headings in Table 2.5 are the main effects (A and B), the AB interaction and I, which represents the total or average of the entire experiment. Notice that the column corresponding to the I has only plus signs. The row designators are the treatment combinations . To find the contrast for estimating any effect, simply multiply the signs in the appropriate columns of the table by the corresponding combination and add (Montgomery, 1976).

TABLE 2.6: ALGEBRAIC SIGNS FOR CALCULATING EFFECTS IN THE 2^2 DESIGN.

Treatment	Factorial Effect			
Combination	I	A	B	AB
(1)	+	-	-	+
A	+	+	-	-
B	+	-	+	-
AB	+	+	+	+

CHAPTER THREE

3.0 EXPERIMENTATION.

List of Equipment used for the project.

	Equipment	Manufacturer
i.	Soxhlet apparatus	Pyrex England
ii.	Reflux Condenser.	Corning Ltd. Lab. Div.Stone staff Eng.
iii.	Heating mantle.	Corning Ltd. Lab. Div
iv.	Measuring cylinder	Pyrex England.
v.	Beaker and Conical flask	Pyrex England.
vi.	Burette and pipette.	Pyrex England
vii.	Thimble and filter paper.	Labsman India
viii.	Electronic Weighing balance	Ohaus U.K
ix.	Seperating funnel and funnel.	Corning Ltd. Dab. Div.
x.	Oven	Gallen Kamp, England.

List of Materials used

i.	Castor seed	Kabba, Kogi.
ii.	Solvent (Petroleum spirit)	May and Baker Ltd.
iii.	Clay sample	Bosso.
iv.	Filter paper	Labsman India

Reagent used.

i. phenolphthalein indicator.

ii. Hydrochloric acid (HCl), sodium hydroxide (NaOH)

iii. Potassium Iodide (KI), potassium hydroxide (KOH)

iv. Sodium thiosulphate ($Na_2 S_2 O_2. 5 H_2O$).

v. Dam's reagent.

3.1 EXPERIMENTAL PROCEDURE

3.1.1 Castor Bean Processing

The processing of castor bean under goes various stages of operation, the unit operations are as follows,

3.1.2 Cleaning

The castor bean seed contains some foreign materials and dirt, which were separated by hand picking.

3.1.3. Drying

The castor bean was dried under the sun, until the capsule splits and sheds the seed. The seeds were decoated manually to remove the coat.

3.1.4 Winnowing

The shells were separated from the nibs using tray to blow away the shells in order to obtain a clean seed.

3.1.5 Cooking

The cleaned seeds were cooked under air light condition at a temperature of 80-90^{o}C. This was done to coagulate the protein, which was necessary to permit efficient extraction. The seed was then dried is the oven at 100^{o}C for 7 hours prior to extraction.

3.1.6 Grinding

Mortar and pestle were used to crush the seed into paste; this was done to weaken the cell walls in order for it to release castor fat for extraction.

3.2 Refining of Extracted Oil

3.2.1 Preparation of Clay

The clay was grounded and sieved using sieve machine of mesh 250-micrometer size, the clay was then mixed with water to remove dirt and stone particles. The clay was activated; 2M of hydrochloric were added to clay slurry in around bottom flask, the mixture was boiled for 1 hour 30 minutes at a temperature of about 120^{o}C. After which the mixture was washed with distilled water to remove the acid, the recovered clay was dried in the oven for 2 hours at a temperature of 100^{o}C and later grounded.

3.2.2 De Gumming

The extracted oil was de gummed by addition of boiled water. The mixture was allowed to stand in a separating funnel for 5 minutes with agitation and allowed to cool to rest, the gum and water removed. The procedure was repeated to remove the gum completely from the oil.

3.2.3 Neutralization

About 50g of the degummed oil was poured into a beaker and heated at 90°C, 35ml of 1M NaOH was added and stirred. This was transferred into a separating funnel and allowed to stand for 2 hours, the soap formed was separated from the oil. Hot water was added again and again to oil solution until the soap in the solution was removed leaving the neutral oil and later poured into a beaker.

3.2.4 Bleaching

The neutralized oil was poured into a beaker and heated to about 80°C. 7g of the activated clay were poured into a beaker; the mixture was stirred for 30 minutes. The temperature was allowed to rise to 100°C. The content was then filtered hot.

3.3 CHARACTERIZATION OF EXTRACTED CASTOR OIL

3.3.1 Determination of Iodine Value.

Procedure:

The method specified by international standard Organization (IS0) 3961 (1989) was used. 0.5g of sample was weighed into a conical flask. 20ml of carbon tetrachloride and 25ml of DAM's reagent was added to the flask. Stopper was then inserted and the content of the flask was vigorously swirled. The flask was then placed in the dark for 2 hours. At the end of the time, 20ml of potassium iodide solution and 150ml of water were added. The contents of the flask were titrated with 0.1M sodium thiosulphate solution until the yellow colour due to iodine has almost disappeared. Few drops of starch were then added and titration continued until the blue colour disappeared after vigorous shaking.

The iodine value is given by the expression:

$$I.V = \frac{12.69C\,(V. - V_2)}{M}$$

Where: C = Concentration of sodium thiosulphtate.

V_1 = Volume of sodium thiosulphate solution

V_2 = Volume of sodium thiosulphate solution used for determination.

M = Mass of the test sample.

3.3.2 Determination of saponification Value.

of the sample weighed into conical flask, 25ml of ethanolic potassium hydroxide was then added by the aid of pipette. A reflux condenser was placed on the electric heater and the content was allowed to boil gently for 65 minutes with shaking from time to time. 1Ml of phenolphthalein indicator was then added to the flask and was titrated with 0.5M hydrochloric acid until the pink colour of the indicator just disappeared.

The saponification value is given by:

$$S.V = \frac{(V_o - V_1) \times C \times 56.1}{M}$$

Where. V_o = Volume of hydrochloric acid solution used for blank test.

V_1 = Volume of hydrochloric acid solution used for determination

C = The exact concentration of hydrochloric acid.

M = Mass of the samples

3.3.3 DETERMINATION OF UNSAPONIFIABLE MATTER

This method uses diethyl ether. 2g of sample was weighed into a conical flask; 5oml of 1M solution of ethanolic potassium hydroxide was added with a few anti- bumping granules. The flask was attached to reflux condenser and the content was allowed to boil for the 1 hour. 100ml of water was added through the top of the condenser with continuous swirling of the flask. The flask was allowed to cool and the contents now transferred into 500ml separating funnel. The conical flask was rinsed by using diethyl ether. The content was allowed to stand until there was complete separation of the two phases. The lower layer was then run off completely as possible into a second separating funnel. The ethanolic soap solution was extracted twice more each time in the same way with diethyl ether and the contents of the extract was collected in a seperation funnel containing 40ml of distilled water. The layers were allowed to separate completely and the lower aqueous layer was drawn off. The ethercal solution was

washed successively with potassium hydroxide solution and water until no longer pink colour of the solution on addition of a few drop of phenolphthalein solution.

The ethercal solution was transferred through the top of separating funnel into conical flask previously dried and weighed. The solvent was evaporated by placing the flask on a boiling water bath. The residue was dried in an oven and allowed to cool in a desiccator.

The unsaponifiable matter content expressed as a percentage by mass of the sample is:

$$= \frac{100\ (M_1 - M_2 - M_3)}{M_o}$$

Where M_o = Mass of sample.

M_1 = Mass of residue.

M_2 = Mass of residue obtained with the blank.

M_3 = Mass of free fatty acid and equal to 0.28VC

And V= volume of 1 molar ethanol potassium hydroxide solution used for Titration

C= concentration in molar of ethanolic potassium hydroxide solution

3.3.4. DETERMINATION OF ACID VALUE

The mixture of ethanol and toluene was first neutralized by using ethanolic potassium hydroxide solution in the presence of indicator. 5g of sample was weighed into a 250ml conical flask, 50ml of previously neutralized mixture of toluene and ethanol was added to the flask. A few drops of phenolphthalein indicator was added and the content was titrated against o.1 ml/ lit solution of ethanolic potassium hydroxide solution until the indicator changes pink colour.

The acid value analysis is expressed as

$$= \frac{V \times C \times 56.1}{M}$$

Where V = Volume of ethanolic potassium hydroxide solution

C = exact concentration of ethanolic potassium hydroxide solution Used.

M = Mass of samples

56.1 = molar mass expressed in grams per-role of potassium hydroxide

3.3.5 Determination of pH Value.

3Oml of the sample was poured into a cleaned dried beaker and 13ml of hot distilled water was added to the sample and stirred to 5°C slowly. The pH probe was standardized with buffer solution and the probe was inserted into the oil, the pH Value was read and noted.

3.3.6 Determination of Specific Gravity.

Density bottle was used to determine the density of the oil. A cleaned, dried bottle of 25ml capacity was weighed (M_o) and filled with the oil, then the bottle and its content was re-weighed to give (M_1). The oil was then substituted with water of the same volume and re-weighted to give (M_2). The expression for specific gravity is given below:

$$\text{Sp. Gr} = \frac{M_1 - M_0}{M_2 - M_1}$$

$$\text{Specific Gravity} = \frac{\text{Mass of the substance}}{\text{Mass of equal volume of water}}$$

3.3.7 Determination of Refractive Index

Refractometer was used in determining the refractive index of the oil, few drops of the sample was placed until glass slide of refractometer. Water at 300°C was circulated around the glass slide to keep its temperature uniform. The refractormeter was viewed through the eyepiece, the dark portion viewed was adjusted to be in intersection of the cross. At no parallax error, the pointer on the scale pointed on the refractive index, this procedure was repeated and the mean value was taken.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION OF RESULTS

4.1 RESULTS

The tables below show the result of this project.

Table 4.1: Volume of oil extracted

I- HOUR EXTRACTION					
Sample	**Wt of empty thimble, w_1 (g)**	**Wt of thimble + sample before extraction w_2 (g)**	**Wt of thimble + sample after extraction w_3 (g)**	**% yield**	**% Average yield**
5g					
A 1	0.9	5.93	5.5	8.548	
2	0.96	5.96	5.52	8.8	
3	1	6	5.57	8.6	8.67
10g					
B					
1	4.24	14.24	12.59	16.5	
2	4.01	14.01	12.36	16.5	
3	4	14	12.36	16.4	16.47
2- HOUR EXTRACTOIN					
5g					
C 1	0.92	5.92	5.05	17.4	
2	0.98	5.98	5.11	17.4	
3	0.94	5.94	5.08	17.2	17.33
10g					
D 1	4.02	14.02	11.1	29.2	
2	4.02	14.02	11.11	29.1	
3	4.06	14.06	11.13	29.3	29.20

Table 4.2: Free fatty acid value (FFA)

S/No	Mass of oil (g)	Initial vol.(cm^3)	Final vol. (cm^3)	Vol. Required (cm^3)
1	5	0	7.5	7.5
2	5	7.5	16.1	8.6
3	5	16.1	24.5	8.4

Table 4.3: Iodine value

S/No	Mass of oil (g)	Initial vol.(cm^3)	Final vol. (cm^3)	Vol. Required (cm^3)
1	0.5	0	7.9	7.9
2	0.5	7.9	15.1	7.2
3	0.5	15.1	22.5	7.4

Table 4.4: Saponification value (SV)

S/No	Mass of oil (g)	Initial vol.(cm^3)	Final vol. (cm^3)	Vol. Required (cm^3)
1	2	0	14.9	14.9
2	2	14.9	17.7	2.8
Blank		0	22	22

Table 4.5: Physical properties of refined castor oil

Property	Refined Castor Oil	Standard Value
Viscosity at 25^0C	6.41	6.3 - 8.8 St
Refractive index at 27^0C	1.46	1.473 - 1.477
Specific gravity	0.96	0.958 - 0.968

Table 4.6: Chemical properties of refined castor oil

Property	Refined Castor Oil	Standard Value
Iodine value	87.56	81 - 91
Acid value (mg NaOH/g of oil)	1.46	0.4 - 4
Saponification value (mg NaOH/g of oil)	184.43	176-187
pH value	6.12	

4.2. DISCUSSION OF RESULTS.

Going by the results, it can be observed that extraction was carried out at different masses of the seed and at different time of extraction. Specification, the extraction of 5g and 10g of seeds was carried out at the time of 1 and 2 hours. The result of percentage oil yield extracted (that is, the oil content of the seed) as shown in table 4.1, for 1-hour extraction, the percentage oil yield on 5kg and 10kg of castor seed were 8.67% and 16.47% respectively. Also, on 2 hours extraction, the percentage oil yield on 5kg and 10 kg of castor seed were 17.3% and 29.2%. Actually, this result deviated from the percentage oil content of castor seed found in the literature (which is, 50-55%). This deviation may be attributed to the variety of castor seed used (because different varieties of castor seed contain different percentage of oil). Besides, the deviation of the results can also be due to the method of extraction employed in this work because it was reported that the most efficient method of extraction was screw press expeller or combination screw press expeller (Fellows 1996). Where as the one used in this work was sox hlet extraction method.

Table 4.5 shows; the physical properties of refined oil extracted in the course of this work as well ass the standard value of each parameter. From the results, the viscosity, refractive index and specific gravity of the refined oil were 6.41 St, 1.46mg NaOH/g of oil and 0.96 respectively. As shown in table 4.5, it was observed that the values of the three parameters were within the range obtained.

Also, the chemical properties of the refined extraction castor oil are shown in table 4.6. From the results, it was shown that all the chemical properties of the refined oil tested which were iodine value, acid value, saponification value, and pH were with the theoretical limits For instance the acid value was calculate to be 1.46mg NaOH/g of oil and this is with the theoretical value of 0.4-4mg NaOH/g of oil. Of importance is the saponification value of the oil 184.43mg NaOH/g of oil when the value found in the literature was between 176 to 187mg NaOH/g of oil.

In order to investigate the variation of the yield with the time of extraction and mass of seed used, a 2x2 full factorial design was carried out in this work. The equation obtained from the factorial design was stimulated and it was discovered that there were only very small errors between the experimental and stimulated yields of the oil extracted from the castor seed.

For instance, the square of the errors for the run 1,2,3 and 4 were calculated using spreadsheet to be 0.0100,0.0064, 0.0025 and 0.0025 respectively.

Based on the results of the analysis of the oil extracted, it can be concluded that the oil can be extracted from castor seed and the oil the properties of the oil extracted in this work has all of its physical land chemical propertied tested in this work within the theoretical range.

CHAPTER FIVE

5.0. CONCLUSION AND RECOMMEDATIONS

5.1. CONCLUSIONS

The results of this work shows that the values for the physical properties of the oil such as the viscosity, refractive index and specific gravity were calculated to be 6.41St, 1.46 and 0.96 respectively while the chemical properties of the oil such as the acid value, iodine value and saponification value were calculated to be 1.46, 87.56 and 184.43mg NaoH/g of oil respectively values fall within the range of the standard specification values of ASTM specification.

In an attempt to predict the variation of oil yield with the time of extraction and mass of seed, a 2x2 full factorial design was carried out and the equation obtained is given as

$Y = 17.745 + 4.77x_1 + 5.455.x_2 + 1.080x_{12}$

where Y is the oil yield, x_1 is the time and x_2 is the mass of castor seed used. Since the simulation of the model equation shows that there is a good agreement between the experimental and simulated results, the model equation can be used to predict the variation of oil yield with the time and mass of seed.

5.2. RECOMMENDATIONS

It is recommended in this project that:

1. Since the oil extracted still contains some solvent, it should not be used for cosmetics
2. The refined oil can be used for industrial purpose to produce other materials, like the lubricating grease, printing ink and hydraulic fluid that do not have direct contact with the skin.
3. Adequate requirement extraction equipment should be provided to make the research work easy.
4. Since the choice of level does not affect the model, it is recommended that one can decide to choose the level anyhow, but it is advisable to choose high numerical value as high level and low numerical value as low level to avoid confusion.

REFERENCES

- Browing, J. (1991). Outlook for U.S castor production is positive. Inform Vol.2 Number 8, pp. 700-01
- Carr, R.A (1976) Refining and de gumming system for edible fats and oil Jaobs, SS; 766-700
- Fellows, O.J (1996). Food Processing Technology, Wood head publishing limited. Cambridge England. Pp146.
- Isah, A.G (2001); Prediction models for the percentage extract of Oxalic acid from the bark of Eucalyptus Camaldulnesis, Federal university of technology Minna, Niger state, Nigeria. pp11-35.
- Moshkin, V.A (1996), Castle. Amerind publishing co. New Dechi. Pp207-209.
- Nawar, W (1996). Lipids, Ch5 in food chemistry. O.Fennema (Ed) pp300-301.
- Robertus, J.D (1991) Structure and action of Ricin, a Cytotosic N-glycosides. Seminar in soil biology. Pp23-30.
- Robinson, T (1964). The organic constituents of higher plants; Their chemistry and inter relationship. Burgess publishing Co. Minneapolis, Minn. pp201-203.
- Schery. R.W.C (1972); Plants for man, prentice ahll, Inc. Engle wood cliff, New Jersey.
- Simpson, B.B and M.C Orgozaly (1986); Economic Botany: Plants in our World. Mc-Graw Hill, New York.
- Sleggs P.W (1990); Vaccum steam refining of castor oil, Inform. Vol.2 number 8, August. Pp702-03.
- Vignolo, R and Naughton, F. (1991) castor: A new sense of direction. Inform. Vol.2 Number 8,pp 692-99

APPENDIX 1

(i) Percentage of castor oil Extracted.

Sample $A_1 = \frac{W_2 - W_3}{W_2 - W_1} \times 100$

$= \frac{5.93 - 5.50}{5.93 - 0.93} \times 100$

$= 8.60\%$

$A_2 = \frac{5.96 - 5.52}{5.96 - 0.96} \times 100$

$= 8.8\ \%$

$A_3 = \frac{6.00 - 5.57}{6.00 - 100} \times 100$

$= 8.6\%$

$\%$ Average yield $= \frac{8.6 + 8.8 + 8.6}{3} = 8.67\%$

Sample $B_1 = \frac{W_2 - W_3}{W_2 - W_1} \times 100$

$= \frac{14.24 - 12.50}{14.24 - 4.24} \times 100$

$= 16.5\%$

$B_2 = \frac{14.01 - 12.36}{14.01 - 4.01} \times 100$

$= 16.5\%$

Average yield $= \frac{16.5 + 16.5 + 16.4}{3} = 16.47\%$

Sample $C_1 = \frac{W_2 - W_3}{W_2 - W_1} \times 100$

$= \frac{5.92 - 5.05}{5.92 - 0.92} \times 100$

$= 17.40\%$

$C_2 = \frac{5.98 - 5.11}{5.98 - 0.98} \times 100$

$= 17.40\%$

$C_3 = \frac{5.94 - 5.08}{5.94 - 0.94} \times 100$

$= 17.20$

% average yield = $\frac{17.40 + 17.40 + 17.20}{3} = 17.33\%$

Sample $D_1 = \frac{W_2 - W_3}{W_2 - W_1} \times 100$

$= \frac{14.02 - 11.10}{14.02 - 4.02} \times 100 = 29.2\%$

$D_2 = \frac{14.02 - 11.11}{14.02 - 4.02} \times 100 = 29.1\%$

$D_3 = \frac{14.06 - 11.13}{14.06 - 4.06} \times 100 = 29.3\%$

% Average yield = $\frac{29.2 + 29.1 + 29.3}{3} = 29.3\%$

APPENDIX 2

Determination of Chemical Properties.

i. Acid value:

Average volume of NaOH required
$= \frac{7.5 + 8.6 + 8.4}{3} = \frac{24.5}{3} = 8.167 cm^3$

$FFA = \frac{Vo \times 2.82}{Wo} \times 100$

$Vo = 8.167 cm^3$

$Wo = 5g$

$: FFA = \frac{8.167 \times 2.82}{5 \times 1000} \times 100$

$= \frac{23030.094}{5000} = 0.46\%$

A.V = FFA x 2 = 0.46 x 2 = 0.92mg NaOH /g of oil

ii. Iodine value (IV)

$V_1 = 7.9 + 7.2 + 7.4 = -7.5 cm^3$

Volume of blank solution = $42.00 cm^3$

$I.V = \frac{12.69 \times C (V_1 - V_2)}{M}$

$I.V = \frac{12.69 \times 0.1 (41.0 - 7.5)}{0.5}$

I.V = 87.56

iii Saponification valuc.

Average volume of HCl required

$\frac{14.9+2.8}{2}$=8.85

$$S.V=\frac{56.1xC\ (Vo-V)}{M}$$

$$=\frac{56.1x0.5(22-8.85)}{2}$$

$$=\frac{368.86}{2}=184.43$$

APPENDIX 3

Determination of physical of castor oil

i. Viscosity.

Time taken = 4 minute, 15 seconds

T = (4 x 60) + 15 = 255 seconds

Viscosity constant 8ΠIt

$$\text{Viscosity} = 8 \times \frac{22}{7} \times 255 = 6411.43\text{cl}$$

Viscosity = 6.4114St.

ii. Specific gravity (Sp.gr)

Weight of sample = 9.588g

Weight of Equal volume of water = 10.001g

$$\text{Sp.gr} = \frac{\text{Weight of sample}}{\text{Weight of Equal Vol. Of water}}$$

$$= \frac{9.588}{10.001} = 0.9587$$

APPENDIX 4

Factorial Design of Extraction of castor oil from castor Bean seed.

Table 1: Factors and their coded levels.

Level of factors	**Code**	**Regressor variable**	
Base Level	0	x_1 (Time)	x_2 (weight)
Interval of variable	Δx_1		
High Level	+1	2	10
Low Level	-1	1	5

Table 2: Experimental Results.

	x_1	X_2	y_1	y_2
1	1	5	8.67	8.53
2	2	10	16.47	15.49
3	1	5	17.33	17.37
4	2	10	29.2	28.9

Table 3: Design matrix (table) for a 2^2 full factorial

Design (FFE)

No of Rub	**Xo**	**X_1**	**X_2**	**X_{12}**
1	+	-	-	+
2	+	-	-	-
3	+	-	+	-
4	+	+	+	+

Table 4: Results and their replication

Replicate Yu_1	**Yu_2**	**Yuv**	**Yu-Yuv**	**Yu_2-Yuv**	**$(Yu_1-Yuv)^2$**	**$(Yu_2-Yuv)^2$**	**Su^2**
8.67	8.53	8.6	0.07	-0.07	0.0049	0.0049	0.0098
16.47	15.49	15.98	0.49	-0.49	0.2401	0.2401	0.4802
17.33	17.37	17.35	-0.02	0.02	0.0004	0.0004	0.0008
29.2	28.9	29.05	0.15	-0.15	0.0225	0.0225	0.045

(i). Mean Yuv = 1/r $\sum$Yuv where r is the number of replicates.

Yuv = replicate observation, here r = 2

(ii) The dispersion of the replicated observation is given by:

$Su^2 = 1/r-1 \sum (Yuv - Yu)^2$

(iii) The sum of the dispersion $\sum Su^2 = 0.5358$

(iv) The number of experiments is = 4

(v) Su^2, maximum = 0.4802

(vi) The homogeneity of the dispersion was determined using cochron criterion

Calculated G – Value

$$G\ cal = \frac{Su^2, max}{\sum Su^2} = \frac{0.4802}{0.5358} = 0.89623$$

(Vii) The G-test was used to check if the output factors of the replication have maximum accuracy of the replication. It ascertains the possibility of carrying out regression analysis. The conclusion of homogeneity is:

$G[\alpha, (r-1), N] > Gcal$

Where N = Number of experimental runs = 4

r = Number of replicates

α = Level of significance

G (0.05, 1, 4) = 0.907 (from Statistical Table)

Since Gtab > Gcal, the regression analysis can now be carried out.

(1). The Mean Square Error was determined by

$S^2 (y) = 1/N \sum S^2 (y)$

is the average sample variance estimate

N =4, the number of experiments.

$S^2 (y) = 1/4 (0.0098 + 0.4802 + 0.0008 + 0.045) = 0.13395$

(2) The experimental error was given by:

$S (y) = \sqrt{S^2 (y)} = \sqrt{0.13395} = 0.365992$

(3) The mean effect is given as

$$bo = 1/N \sum (X_o, Y_o) = 1/4 (8.6 + 15.98 + 17.35 + 29.05)$$

$$= 17.75$$

Where Xo are coded signs in the X_o column of the design matrix

$$b_1 = 1/N \sum (X_1, Y_o) = 1/4 (-8.6 + 15.98 - 17.35 + 29.05)$$

$$= 4.77$$

$$b_2 = 1/N \sum (X_2, Y_o) = 1/4 (-8.6 - 15.98 + 17.35 + 29.05)$$

$$= 5.46$$

$$b_{12} = 1/N \sum (X_{12}, Y_o) = 1/4 (8.6 - 15.98 - 17.35 + 29.05)$$

$$= 1.08$$

5. The construction of confidence interval and testing of hypothesis about individual regression coefficients are frequently used in assessing their statistical significance. Confidence intervals for the regression coefficients with confidence coefficient, α, are of the general term

$$b \pm t [\alpha, N (r-1)] Sb.$$

Where Sb is the estimated standard errors in regression coefficient b and $t [\alpha, N(r-1)]$ is an appropriate standard t – value with N(r-1) degree of freedom. For full factorial experiments, error in each regression coefficient is the same and is determine by:

Testing statistical significant of the regression coefficient

$t [\alpha, N(r-1)] = t (0.05, 4) = 2.776$ (statistical table)

i.e. t (tab) = 2.776

5. $Sb = \frac{S(y)}{\sqrt{N.r}} = \frac{0.365992}{\sqrt{4 \times 2}} = 0.129398$

6. Confidence internal Δbi = t table x Sb

$$= 0.359208$$

7. Statistical significant (t cal)

$$To = \frac{bo}{Sb} = \frac{17.75}{0.129398} = 137.1354$$

$T_1 = \frac{b_1}{Sb} = \frac{4.77}{0.129398} = 36.86311$

$T_2 = \frac{b2}{Sb} = \frac{5.46}{0.129398} = 42.15687$

$T_{12} = \frac{b_{12}}{Sb} = \frac{1.08}{0.129398} = 8.346365$

Hence the fitted model in equation is:

$Y = b_o + b_1x_1 + b_2x_2 + b_{12}x_{12}$

$Y = 17.75 + 4.770x_1 + 5.455x_2 + 1.08x_{12}$

Since all t cal >t tab, then all the coefficients of regression are significant.

Table 5: The estimated effects, confidence intervals and calculated t- values.

ression coefficients	**Estimated Effects**	**Confidence Internal**	**t-Values**
b_0	17.75	±0.359208	137.1354
b_1	4.770	±0.359208	36.86311
b_2	5,455	±0.359208	42.15687
b_{12}	1.080	± 0.359208	8.346365

8. The practical value (using table 2).

$Y_1 = 17.745 (+1) + 4.770 (-1) + 5.455 (-1) + 1.080 (+1) = 8.5$

$Y_2 = 17.745 (+1) + 4.770 (+1) + 5.455(-1) + 1.080 (-1) = 15.9$

$Y_3 = 17.745 (+1) + 4.770 (-1) + 5.455(+1) + 1.080 (-1) = 17.3$

$Y_4 = 17.745 (+1) + 4.770 (+) + 5.455 (+1) + 1.080 (+1) = 29.1$

Table 6: Experimental land simulated Result

Run No.	Y_u	Y_{cal}	$e_i=Y_u-Y_{cal}$	e^2
1	8.6	8.5	0.1	0.01
2	15.98	15.9	0.08	0.0064
3	17.35	17.3	0.05	0.0025
4	29.05	29.1	-0.05	0.0025

The adequacy of the model will be evaluated using null hypothesis [Ho:bj =o] on the individual regression analysis coefficient. The analysis of variance is very useful in confirming the significance of the coefficients. In the 2^k factional design with replicates, the regression sum for any effect is:

(a) $SS_R = r/N (\text{constant})^2$

and has a single degree of freedom.

The total sum of squares was calculated by

(b) $$SS_T = \sum Yuv^2 - \frac{\sum (Yuv)^2}{N.r}$$

The error sum of square was given by

$SS_E = SS_T - \sum SS_R$

Testing the significance of individual coefficient was carried out by fisher's test (CF – Test):

$$Fcal = \frac{MS_R}{MSt} = \frac{SS_R/ dF_R}{SSt/ N (r-1)}$$

Where dF_R = degree of freedom regression = 1

The calculated F-value.are compared with the appropriate critical table value. The null hypothesis was rejected using:

$Fcal > F [\alpha, dF_R, N (r-1)]$

(C) Constant = $\sum (x_1 Y_o)^2$

$SSb_1 = 2/4\ (-8.6 + 15.98 - 17.35 + 29.05)^2 = 182.0232$

$SSb_2 = 2/4\ (-8.6 - 15.98 + 17.35 + 29.05)^2 = 238.0562$

$SSb_{12} = 2/4\ (8.6 - 15.98 - 17.35 + 29.05)^2 = 9.3312$

$SS_R = 182.0232 + 238.0562 + 9.3312 = 429.4106$

(10) Sum of square error: $SS_E = SS_T - SS_R$

$$SS_T = \sum Yuv^2 - \frac{\sum (yuv)^2}{N.r}$$

$\sum Yuv^2 = 8.67^2 + 16.47^2 + 17.33^2 + 29.2^2 + 8.53^2 + 15.98^2 + 17.37^2 + 28.9^2 = 2949.07$

$\sum (yuv)^2 = (8.67 + 16.47 + 17.33 + 29.2 + 8.53 + 15.98 + 17.37 + 28.9)^2 = 20.152.64$

Hence $SS_T = 2949.07 - \frac{20152.64}{4x2} = 429.9464$

Therefore $SS_E = 429.9464 - 429.4106 = 0.5358$

(11) The F-ratio was calculated using

(12) $$Fcal = \frac{SS_R/dFg}{SS_E/N(r-1)}$$

For $b_1 = \frac{182.0232/1}{0.5358/4} = 1358.889$

$b_2 = \frac{238.0562/1}{0.5358/4} = 1777.202$

$b_{12} = \frac{9.3312/1}{0.5358/4} = 69.66181$

Table 7: Complete analysis of variance

Source of variation	**Effect**	**Sum of square (SS)**	**Degree of Freedom (df)**	**Mega square (ms)**	**F-ratio(fcal)**
b_1	4.77	182.0232	1	182.0232	1358.889
b_2	5.46	238.0562	1	238.0562	1777.202
b_{12}	1.08	9.3312	1	9.3312	69.66181
Error		0.5358	N(r-1)=4		

Total		429.9464			

(12) The dispersion of adequacy for the replicate experiment is:

$S^2ad = \sum eu^2 = \sum (yu-yv)^2$

$=0.01+0.0064+0.0025+0.0025 =0.0214$

Number of inadequacy coefficient, $\lambda = 0$

$dfad = 4-0 = 4$

$S^2ad = \frac{2}{4} \times 0.0214 = 0.0107$

(13) Applying Fisher`s criterion

$Fcal = \frac{S^2ad}{S^2(y)} = \frac{0.0707}{0.13395} = 0.079881$

$F[\alpha, N-r, N(r-1)]$

$=F(0.05, 2, 4) =19.25$ (statistical table)

Somce Ftab > Fcal. The fitted model is adequate.