

**DESIGN AND OPTIMIZATION OF PROCESS
PARAMETERS FOR PLANT BASED OIL
EXTRACTION BY DESIGN OF EXPERIMENTS (DOE)**

Submitted in partial fulfillment of the requirements for the award of

Master of Science

By:

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SATHYABAMA

**INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)**

Accredited with Grade "A" by NAAC

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PARAMETERS FOR PLANT BASED OIL EXTRACTION BY
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I wish success in all his future endeavors.



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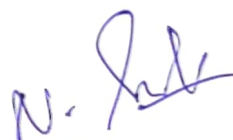
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I **SANTHOSH KUMAR N**, hereby declare that the Project Report entitled **"DESIGN AND OPTIMIZATION OF PROCESS PARAMETERS FOR PLANT BASED OIL EXTRACTION BY DESIGN OF EXPERIMENTS (DOE)"** done by us under the guidance of **Dr.K.CHENNAKESAVULU, M.Sc.,Ph.D.,** Associate Professor Department of Chemistry, Sathyabama Institute of Science and Technology is submitted in partial fulfilment of the requirements for the award of **Master of Science degree in Chemistry in May 2023**

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SANTHOSH KUMAR N

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ABSTRACT

Flotation is a process used for separation of minerals from the ore. This process depends on the collector reagent that binds the mineral to the air bubbles causing the separation of minerals from the gangue materials. The biodegradable collector in this process is the oil extracted from cotton seed. Cotton seed oil extraction is crucial process in the production of vegetable oils. This thesis focus on the design and optimization of process parameters for cotton seed oil extraction using the design of experiments (DOE) methodology. The objective is to develop an efficient and cost effective extraction process by identifying the key factors that influence oil yield and determining their optimal levels. The findings of this research provide valuable insights into the design and optimization of process parameters for cotton seed oil extraction. The optimized conditions identified through DOE methodologies can enhance the efficiency , productivity, and cost effectiveness of the extraction process. Moreover, the developed mathematical models serve as predictive tools for oil yield under different operating conditions, aiding in process control and optimization. The outcome of this study contribute to the advancement of cotton seed oil extraction techniques by utilizing DOE principles and statistical analysis. The optimized process conditions and predictive models obtained in this study can be used to guide process control and optimization in industrial applications of cotton seed oil extraction. The research contributes to the advancement of the cotton seed oil extraction process and demonstrates the potential of DOE for optimizing process parameters and improving process efficiency and sustainability.

KEY WORDS : Non coking coal, Cotton seed oil, Soxhlet extraction, Froth floatation, Design of experiment, Rotary evaporator.

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LIST OF ABBREVIATION

S.NO	ABBREVIATION	EXPANSION
1	FTIR	Fourier Transform Infrared Spectroscopy
2	GC-MS	Gas Chromatography Mass Spectrometry

CHAPTER 1

INTRODUCTION

1.1. SEED GERMINATION

Seed germination is the foundation of sexually reproducing plant proliferation, efficient crop production, and a successful crop improvement research programme. Cotton (*Gossypium* spp.), the focus of this review, is frequently sensitive to germination conditions.

1.2. COTTON

The toughness of the cotton seed coat, storage, extreme temperatures, and dormancy are all factors that can have an impact on cotton seed germination. These obstacles disproportionately affect research programmes studying exotic and wild cotton species. While seeds are an important form of plant dispersal and propagation in the environment, they had to develop mechanisms to ensure the survival of the next generation in adverse conditions. Dormancy is a major survival adaptation of seeds.

This phenomenon allows the seed to delay and coordinate germination in response to environmental conditions, and it is critical to a species' conservation and proliferation. In general, dormant seeds begin or resume germination when they detect environmental signals indicating suitable conditions for plant development, such as temperature fluctuations, water availability, and even smoke.

If unfavourable conditions persist and seeds do not germinate for an extended period of time, ageing may have a negative impact on this process. Seed structure modifications, such as the hard seed trait in cotton, can also affect germination. Hard seeds, also known as physical dormancy, are water impermeable and prevent gas exchange and water uptake by the embryo. Cotton (*Gossypium* spp.) has been cultivated since prehistoric times in various parts of the world. Since then, it has been domesticated from a wild perennial plant to a herbaceous annual crop.

Cotton is grown as an annual crop frequently using growth regulators and termination methods such as chemical, temperature, or mechanical. The cotton plant's seed is another distinctive feature. Both maternal and filial tissues have economic importance because the embryo cells generate protein and oils while a portion of the seed coat epidermal cells develop into cellulose-rich fibres.

1.3. COTTON SEED ANATOMY AND PHYSIOLOGY

The embryo in a mature cotton seed contains the radicle, hypocotyl, a primordial epicotyl, and two cotyledons.

The energy for germination and the early stages of seedling development comes from the cotyledons. Under typical environmental circumstances, water enters the cotton seed through the chalazal cap and travels via capillary action to the seed's opposite, pointed side, or micro Pyle. The seed coat is softened by this diffuse water absorption, which also creates a moisture reserve for the embryo in case water is in short supply during germination. The seed swells as water is absorbed, rupturing the seed coat at the micropylar end.

The embryonic axis lengthens; the radicle emerges through the micropyle, develops downward, and eventually becomes the plant's taproot. As part of this procedure, oxygen is also taken up by the seed coat, starting respiration. If the circumstances are right, the seed will begin to undergo full metabolic activity, and a new cotton plant will grow [28].

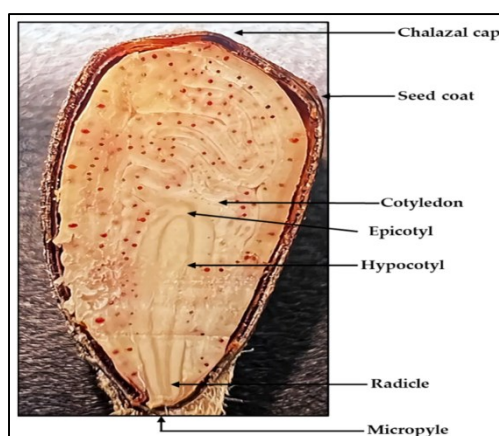


Fig 1.1.Cotton seed coat

Cotton (*Gossypium arboreum*) is a member of the Malvaceae, sometimes known as the mallow family. It has long been used to weave clothing in Africa after

being harvested from the wild, and it provided the foundation for various textile enterprises in northern Nigeria. The juice squeezed off of the leaf is used locally for the treatment of cobra snake split into the eyes, and the seed itself has a number of medical benefits. When a boll reaches maturity, which is when the seeds and fibres are encapsulated, it opens up to reveal the white fibres.

The seed includes an oil-bearing kernel enclosed in a tough, black outer hull that yields fibres and linters, as well as 20–25% high-quality protein, carbohydrates, lecithin, and sterols. Moreover, it is said to be extremely high in tocopherols, which keep it from going bad and lengthen the shelf life of anything made from it. Ertugrul and Filiz reported that the oil has good lubricating qualities and is a good lubrication oil addition. The refined oil can be consumed, and the byproduct makes nutrient-dense feed for animals. The safety of the seed has been questioned since it contains a toxin called gossypol, which is often fed to cattle unprocessed.

There aren't many studies on the transesterification of cotton seed oil to biodiesel, but those that have been done include those that found similar properties to diesel, performed performance evaluations and found increased Nox emissions, and examined the performance and emissions of cotton seed oil methyl ester in diesel engines for B5, B20, B40, B75, and B100 blends. This work details the characteristics of the cotton seed planted in northern Nigeria because the properties of vegetable oils vary greatly depending on where they were produced [1].

1.4. FATTY ACID PROFILE

The oil is 73.63% unsaturated, making it a preferred oil for consumers who are concerned about their health, and it mostly comprises the saturated acids 22.42% palmitic acid and 2.40% stearic acid, as well as unsaturated acids 25.6% oleic acid and 40.37% linoleic acid. The profile for biodiesel followed the same pattern, but the weight of the double-bonded acids decreased while the weight of monounsaturated acids grew, increasing the total unsaturation from 73.63% for the oil to 79.60% for the biodiesel. Particularly noteworthy is the oil's 17.31% oleic acid content, which is below the quoted average of 45% for vegetable oils. Oleic acid value is significant when taking into account both the future potential for soap

generation during trans-esterification as well as cold flow properties and oxidation stability.

In contrast to other common vegetable oils, cottonseed oil has a comparatively high concentration of palmitic acid (usually 23%), oleic acid (17%), and linoleic acid (56%). A particularly valuable subset of vegetable oils, cottonseed oil's fatty acids are primarily C-16 and C-18 fatty acids with no more than two double bonds. Given that oleic and linoleic fatty acids account for over 75% of the total, its fatty acid profile is characteristic of the oleic/linoleic group of vegetable oils. Cotton seed oil's fatty acid profile

Cottonseed oil is thought to have a non-random distribution of fatty acids, with saturated fatty acids predominately located in the sn-1 or sn-3 locations and unsaturated fatty acids in the sn-2 position. Most triglycerides comprise some mix of linoleic, oleic, and palmitic fatty acids because they make up over 90% of cottonseed oil's fatty acid composition.

One of the first oils to be derived from a seed was cottonseed oil. Cottonseed oil has primarily been utilised for food uses, but it has also been used in the production of soap, lubricants, sulfonated oil, medicines, protective coastings, rubber, and, to a lesser extent, leather, textiles, printing ink, polishes, synthetic polymers, and resins. The goal of this research project was to extract oil from cotton seeds using a Soxhlet apparatus and n-hexane solvent in order to use the seed oil for making soap [35].

Gas chromatography was used to determine the FA profile of CSO. Initially, the produced CSO underwent selective derivatization using the Miranda et al. (2013) methodology, which included combining 200 L of CSO with 1 mL of 0.2N HCl-CH₃OH and heating the mixture at 60 °C for 4 hours. Next, 0.2 mL of distilled water and 2 mL of hexane were then added. A HP Model 6890 gas chromatograph outfitted with a flame ionisation detector and an HP-INNOWAX polar capillary column (30 m X 0.32 mm X 0.25 m) was then filled with two L of the extract. According to López-García et al., the temperature programme. By contrasting FA's retention times with those seen in a Supelco 37 FAME Mix standard, FA were found.

Table 1.1. Fatty acid profile of cotton seed oil

Fatty Acid	%Weight
C8:0	0.74
C10:0	2.43
C14:0	0.88
C16:0	20.91
C18:0	2.59
C18:1	16.54
C18:2	55.91
Saturated fatty acids	24.96
Unsaturated fatty acids	72.45
SFA/UFA ratio*	0.4

1.5. COTTON SEED OIL – INDUSTRIAL PURPOSE

Cottonseed oil (CSO) is a byproduct of the cotton industry that is produced from delinted and decorticated cottonseed for use in cooking oil and other industrial purposes.

In many nations, cotton is grown as an annual crop for economic purposes. Several commercially advantageous *Gossypium hirsutum*, *Gossypium arboreum*, *Gossypium herbaceum*, *Gossypium barbadense*, and other cotton varieties were produced traditionally to provide improved agronomic types of cotton for use in textile, fibre, oil, and animal feed. The production of cottonseed was 43 million metric tonnes in the world in 2019–2020.

Oil is extracted from clean, dry cottonseeds and contains between 15% and 20% CSO, depending on the quality and variety. The amount of oil is also influenced by the weather, the growth, and maturity of cottonseed, and the oil production varies from cottonseed variety to cottonseed variety and from place to place. The oil content of 20 wild *Gossypium* types ranges from 11.22 to 24.82%. (Sharif et al. 2019). Several traditional and unconventional approaches are used to extract CSO.

Other biochemical and mechanical techniques are included in the non-traditional methods to extract oil from oilseeds to a significant extent, in addition to conventional methods such as organic solvent extraction.

Following extraction, CSO is analysed for its fatty acid composition and physiochemical characteristics, such as the amount of free fatty acids, acidity, iodine value, saponification value, peroxide value, specific gravity, refractive index, colour, quality, and stability of the oil. For usage in human consumption and industrial purposes, CSO is additionally tested for bioactive components such as total antioxidants, flavonoids, sterols, tocopherols, and gossypol.

Due to its higher level of unsaturated fatty acids (USFA) like oleic (17%) and linolenic (56%) with 23% of saturated fatty acids (SFA), CSO is regarded as a nutritionally balanced oil (palmitic acid).

1.5.1 EXTRACTION OF COTTON SEED OIL

Oil seed extraction is the technique of separating triglycerides from different seeds. To increase oil yield, quality, and physiochemical qualities, extraction techniques have progressed from traditional to non-conventional approaches. Biochemical methods include enzyme-assisted extraction (EAE), whereas mechanical methods include solvent extraction, supercritical fluid extraction (SFE), high pressure extraction (HPE), microwave-assisted extraction (MAE), ultrasound-assisted extraction (UAE), and mechanical extraction.

To get uniformly sized cottonseed meal or cake, clean, dry cottonseeds must be ground and sieved prior to extraction. The downstream process of oil extraction is favourably impacted by the same soybean particle size, and grinding is a crucial step in the oil extraction process to control the flow of extraction and to produce a larger oil output.

Clean and dry cottonseeds are used directly for extraction by screw or hydraulic pressing in the ME extraction process. In contrast to other methods that required toxic chemicals and produced oil that was colourless, odourless, and low in nutrients after numerous extraction and purification steps for crude oil, screw and hydraulic pressing techniques are physical methods of extracting oil that

produce high-quality oil with natural colour, aroma, taste, and other natural ingredients.

Traditionally, CSO is extracted at a laboratory scale using the Soxhlet technique and several solvents. SE is based on a solvent's capacity to break down and extract oil from oilseeds. The SE method uses a variety of solvents, including chloroform, methanol, acetone, ethanol, water, n-hexane, petroleum ether, etc.

To study the kinetics and thermodynamics of CSO extraction using hexane and ethanol as a solvent, *Gossypium hirsutum* seeds were ground, vacuum dried, and separated to optimise various parameters: 0.6-, 1.2-, and 1.6-mm particle size, 5:1 to 15:1 solvent to solid ratio, 288 to 318 °C for 1- to 180-min time interval. The ability of the solvent to solubilize a significant amount of oil in the solvent during the process determined the solvent to solid ratio, and CSO is slightly more soluble in n-hexane than ethanol. Small particle size of seeds was easily dissolved in the solvent than large particles, which is highly resistant to the oil seed extraction.

Surface oil dissolves readily in the solvent, and as the extraction time interval rises, so does the solvent's ability to dissolve residual CSO. Both the hexane and the ethanol extraction processes yield higher oil yields at 0.6 mm particle size and 318°C temperature after 3 hours of extraction; this study came to the conclusion that ethanol was a good substitute for hexane [24].

1.6. COLLECTOR REAGENT

These are the substances that are added to the froth flotation process to make minerals hydrophobic and separate them from gangue materials. The majority of minerals are not hydrophobic by nature. To render them hydrophobic, a collector reagent is therefore employed.

Minerals become hydrophobic as a result of the collecting agents adhering to their surface, which also makes them easier to connect to air bubbles and enable them to float atop the froth layer. The froth layer can readily be used to gather the minerals [2].

The collector reagents used can be of two types,

1. Conventional collector reagents
2. Non-conventional collector reagents

1.6.1 Conventional collector reagents

These chemicals are produced using non-renewable materials. Kerosene and diesel oil are the two common reagents most frequently employed in flotation processes. The price of these agents is rising every day because they are not renewable. In India, imports satisfy 70% of the country's oil needs. Since this source is getting harder to find, another reagent must be synthesised to take its position as the standard collector. These chemicals have a negative influence on the environment, are dangerous, and affect the flotation products chemically [25].

1.6.2 Non-conventional collector reagents

These reagents are made from renewable resources and biodegradable materials. For traditional collectors, they serve as a substitute source. because they are safe for the environment, inexpensive, and readily available. The heteropolar, polar and non-polar groups are present in the biodegradable oils. For the flotation of non-coking coal in this study, spent tea powder oil is used as an unconventional collector reagent. Due to the hydrophilic functional groups on its surfaces and the high oxygen concentration, this form of coal is difficult to float. For the flotation of non-coking coal, the use of unconventional collector reagents is shown to be extremely effective [3].

1.7.COAL

The term "col," which means "glowing ember," is where the word coal first appeared. It is a sedimentary rock that is often black or brown and contains carbon, hydrogen, oxygen, sulphur, and other elements [7].

1.7.1 Formation of coal

The formation of coal takes millions of years. A lengthy process known as carbonisation creates coal from the ancient plant and animal remnants. Under

intense pressure and heat, dead plant is transformed into carbon-rich coal in this process [8].

The world was covered with vast seas and lush forests during prehistoric periods. The woods and animals became trapped beneath the swamp as a result of natural disasters such as floods, earthquakes, volcanoes, and others. As additional soil was added, they sank farther, and because of the intense pressure, high temperature, and lack of air inside the ground, dead plants and animals are carbonised into coal [9].

1.7.2 Coal in India

In India, coal is important for the development of the metallurgical and energy industries. In terms of the top producing nations of coal worldwide, India comes in third. China and the United States are in the top two spots. India's coal reserves total 301 billion tonnes, of which 260 billion tonnes are non-coking coal and the remaining 100 billion tonnes are coking coal.

The drift origin causes the low quality coal reserves, which are exploited through mechanised bulk mining. When compared to coal from other nations, Indian coal has a high ash content and a low heat value in nature, making it the country's main energy source. Opencast mines in India produce almost 90% of the country's coal [18].

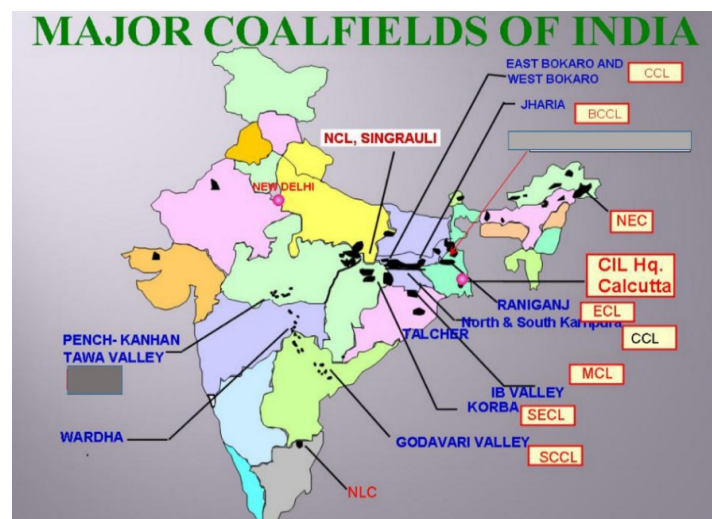


Fig. 1.2: Coal fields in India

1.7.3 Categories of coal

There are two main categories of coal: Coking coal & non-coking coal

1.7.3.1 Coking coal

It is also known as metallurgical coal, and it is mainly used to produce coke which is a major raw material in steel making [11]. It has high carbon content, less ash percentage, less sulphur content, and less moisture [12]. It is further divided as:

- Hard coking coal
- Medium coking coal
- Semi-coking coal
- Pulverized coal [1].

1.7.3.2 Non-coking coal

They have high ash content and high sulphur content. Hence, they cannot be used in steel and iron industries. It cannot be used in the manufacture of coke, and thermal power generation. It also has less percentage of carbon.

1.7.4. Beneficiation of non coking coal

After going through pre-treatment procedures such, new techniques have been implemented for using non-coking coal in the steel and iron industries, power plants for the generation of energy. Liquid fluidized heavy media bath, column flotation technology, chemical and biological leaching, and an advanced dewatering method. These procedures are employed to remove the high levels of ash and sulphur from Indian coal. In this study, tea waste is employed as a collector reagent in a column flotation process for the beneficiation of non-coking coal [14].

1.7.5. Proximate analysis of non coking coal

The most popular and straightforward chemical analysis, proximate analysis, can be used to evaluate the coal. The moisture, volatile, ash, and fixed carbon contents of a coal are identified using this study.

(a) Moisture content

Determination of moisture is carried out by placing a 1 gm sample of powdered non- coking coal material in a tared porcelain dish and covers it with a watch-glass. Place the dish in an airoven maintained at temperature $150 \pm 1^\circ\text{C}$ and remove the watch-glass from it. Heat the sample for two hours in Hot air oven and cover the dish with the watch-glass before taken it out of the oven. Cool the dish in a desiccator and weigh. The loss in weight represents moisture.

$$\text{Moisture}\% = \frac{\text{Weight of moisture}}{\text{Weight of sample}} \times 100$$

(b) Volatile Matter (VM)

Introduce in a weighted silica crucible with tightly fitted lid about one gram of the moisture free sample. Heat the crucible in a muffle furnace maintained at a temperature of $925 \pm 25^\circ\text{C}$ for 7 min. The bottom of the crucible shall not rest on the muffle floor. Remove the crucible from the muffle after 7 min; Allow it to cool in a desiccator and weigh. The loss of weight is taken as weight of volatile matter.

$$\text{Volatile matter}\% = \frac{\text{Weight of volatile matter}}{\text{Weight of original sample}} \times 100$$

(c) Ash content

Weigh accurately about 1 to 2 g of the moisture free material in a silica crucible, keep in a muffle furnace and heat to $500 \pm 10^\circ\text{C}$ within one hour and $775 \pm 10^\circ\text{C}$ in two hours. A slow stream of air is maintained through the muffle furnace. When the carbon is completely removed as indicated by the absence of black particle upon stirring with a platinum wire, the temperature is further increased to $950 \pm 25^\circ\text{C}$ and-kept for one hour. Cool in a desiccator and weigh. The loss of weight is taken as weight of Ash content.

$$\text{Ash \%} = \frac{\text{Weight of ash}}{\text{Weight of sample}} \times 100$$

(d) Fixed carbon(FC)

Fixed Carbon or FC is derived by subtracting from 100 the value of moisture, volatile matter and ash.

$$\text{Fixed Carbon \%} = 100 - (\text{Moisture \%} + \text{Volatile Matter \%} + \text{Ash \%})$$

1.8. MINERAL PROCESSING

Processing of minerals Mineral dressing is a type of metallurgy that is used to remove minerals from ores. It serves as an extraction method.

Metals: iron, copper, lead, magnetite, zinc, bauxite, aluminium, etc. rocks, including sand, stone, clay, and marble. Apatite, quartz, barite, and other types of mineral ore.

The required crude material is extracted through four phases of processing.

1.8.1 Grinding and crushing

It is the method of dividing up a sample of huge rocks for other procedures. It also goes by the name "comminution."

1.8.2 Sizing and classification

It is the process of using sieves to separate minerals into different sizes. In comparison to coarse materials, finer samples go through different steps of processing.

1.8.3 Concentration

In order to attain the necessary concentration, the sample must be broken down. There are various methods for concentrating,

a) Sorting

The particles can be automatically sorted using optical sensors depending on a variety of criteria.

b) Electrostatic Separation

The high tension roller is made up of electrodynamic separators and electrostatic sensors. As this separation involves electric current, the sample must

be dry. Mineral sand separation is mostly accomplished by running electricity through the material to separate the gangue elements.

c) Gravity Separation

It involves separating one or more materials based on how they react to the combined effects of gravity and magnetic or centrifugal force.

d) Magnetic Separation

In this procedure, electromagnets are used to separate the desired mineral from the ore [4].

e) Froth Flotation

It is a flexible method of processing a mineral, and flotation technology has made it possible to mine low-grade ore. It is a technique for physically separating materials that relies on variations in how well air bubbles stick to mineral surfaces. After the bubbles are removed from the surface, they are attached with priceless minerals.

The gangue materials are left behind as the mineral is transported to the froth. Only minerals that are hydrophobic can adhere to the air bubble. Since most minerals are not hydrophobic by nature, flotation reagents are used to convert them. In order to make the minerals hydrophobic and create bubbles in the slurry, it uses a frother and a collector.

1.8.4. Principle of froth floatation

It is a physiochemical separation method that takes advantage of the differences in the surface characteristics of minerals and gangue materials. Three phases—solid, froth, and water—along with a number of auxiliary processes and interactions are involved. Three processes are used to extract minerals:

- (1) Selective attachment to air bubbles
- (2) Entrainment in water passing through froth
- (3) Aggregation amongst particles in the froth attached with the air bubbles

The primary factor in recovering minerals for concentration is the attachment of minerals to bubbles. By using reagents, it is possible to change the variations in the surface characteristics of the minerals.

1.8.4.1. Types of flotation

There are two types of flotation in which the minerals can be processed.

- Direct flotation is a technique in which the mineral is transported to the froth during flotation concentration, leaving the gangue elements in the tailings.
- Reverse flotation - This process's opposite, in which the minerals are left behind and the gangue elements are moved to the foam, is known as reverse flotation.

1.8.4.2. Effect of hydrophobicity and hydrophilicity

Fundamentally, hydrophobicity is what causes floating. In the flotation cell, a mixture of wettable, hydrophilic, and non-wettable, hydrophobic minerals is used. Through the impeller, air is agitated, causing bubbles and particles to collide, which allows minerals to cling to the air bubble. The hydrophilic minerals will remain in the water while the hydrophobic minerals adhere to the bubble and ascend to the froth layer.

1.8.4.3. Flotation reagents

Chemical compounds can be used to make the mineral hydrophobic. By coating the mineral's surface with non-polar oil, the chemical treatments render it hydrophobic. The reagents utilised in flotation methods include:

- Collectors - By adhering to the surface of minerals, they become hydrophobic and make it easier for the mineral to cling to the air bubble.
- Frothers - They are used to maintain a stable froth layer that can be collected before an air bubble breaks by stabilising air bubbles.

- Regulators: These devices activate or inhibit the attachment of minerals to air bubbles, respectively, to regulate the flotation process. It also regulates the pH of the system

1.8.4.4. Flotation equipment

The laboratory flotation experiments are completed in batches. The flotation cell can process samples weighing 500 g to 2 kg. Mechanical factors such as air flow and the impeller's rotational speed are used to mechanically stir the cell. By means of an impeller, which splits the stream of air into tiny bubbles, air is forced down the standpipe. After interacting with mineral particles, these bubbles ascend to the froth layer and are collected as mineralized froth.



Fig.1.3. Flotation cell

1.8.5 Dewatering

In order to remove the gangue materials and obtain a final concentrate that may be used for a variety of purposes, it is necessary to remove the water content from the mineral during the last stage of mineral processing [15].

1.9. DESIGN OF EXPERIMENTS

A statistical tool called the design of experiments (DOE) is employed in a number of system, process, and product designs. It is a type of versatile instrument that can be used in a variety of circumstances, including:

1. Comparisons design
2. Modular screening
3. Identification of the transfer function
4. Optimization
5. Sturdy design

It is a widely used tool in a variety of scientific fields, including computer science, engineering, physics, biochemistry, and many more. It is a mathematical methodology for designing experiments, carrying them out, then analysing and interpreting the results. It is an effective instrument for enhancing the dependability and quality of products [5].

1.9.1. Taguchi method

The Taguchi DOE method, a novel approach with the goal of enhancing product and process quality, is utilised in this work. It is frequently called sturdy design. Dr. Genichi Taguchi created it to enhance the fundamental functionality of process and product, enabling adaptable designs [16].

The Taguchi method is a potent technique that can lower product costs while raising product quality. Depending on three things,

- Loss function: This function depicts the loss that people have caused.
- Orthogonal array - It is used to create and analyse experimental data from which more information may be gathered with regard to all affecting factors, using the fewest possible experiments.
- Robustness - It reduces the vulnerability of the system to different sources.

1.9.2 Taguchi method specifications

- It is an evaluation of two or more parameters or factors that affect the end product's results.
- Formulating the problem
- Identifying the parameters are the first two steps in adopting the Taguchi technique.
- Identifying the quantity of each factor's levels
- deciding on an orthogonal matrix.
- Arraying the variables in an orthogonal fashion.
- Applying the orthogonal array to the experiment
- Examining the results of the experiment
- Determining the best possible production quality

CHAPTER 2

AIM & SCOPE

2.1. AIM OF THE STUDY

The specific objectives of this study were

- Feasibility studies of oil extraction from cotton seeds
- To study and determine the optimum operating extraction parameters (temperature & feed (cotton seed) size)
- To characterize cotton seed extract
- Application of extracted oil as collector for non-coking coal flotation

2.2. SCOPE OF THE STUDY

- Usage of non-conventional collector reagent for the flotation of non-coking coal
- Feasibility studies of oil extraction from naturally occurring cotton seed using soxhlet method

CHAPTER 3

LITERATURE REVIEW

3.1. E.I. Bello, Aladesuru A.A et al, 2015 –proposed that cottonseed oil was extracted using soxhlet extraction process, yielding 20.2% oil content. The substance was transesterified without prior treatment using methanol and sodium hydroxide as catalyst because preliminary evaluation revealed little free fatty acid value. The oil had strong odour and was the colour of used black engine oil. Although the glycerol was brown , the biodiesel was bright yellow. The HP 6890 Gas Chromatography analyzer was used to assess fatty acid profile, which revealed that oil was 89.47% unsaturated. It was determined that biodiesel can be utilised as an alternative fuel for diesel engines after oil and biodiesel were characterised [2].

3.2. Manoj Kumar, Baohong Zhang et al, 2022 – has given a hypothesis on Cotton which is valuable annual fibre crop, and cottonseed oil is valuable byproduct that is derived from cottonseeds. Oil yield varies depending on cotton species, growing regions, season, and oil extraction techniques. This study gives a general summary of various chemical, biological, and mechanical processes for extracting CSO. Functional analysis and physicochemical analysis of CSO showed that it was of higher quality than other vegetable oils. Unsaturated fatty acids made up a higher percentage of fatty acid profile, which was proven to have positive health impacts. The current review covers number of physiochemical properties, such as iodine value, phosphorus content, moisture content, refractive index, specific gravity, saponification value, gossypol content, and antioxidants. The advantages of CSO for your health as well as its usefulness in cooking and other industrial applications are discussed. CSO is safe to consume and has a well-developed extraction technology, good fatty acid composition [6].

3.3. E. Rojo-Gutiérrez, J.J. Buenrostro-Figueroa¹ et al, 2020 – given a theory about cottonseed recovery during processing is lost in significant amounts. According to certain reports, cottonseed has significant lipid content and high-quality protein. In order to obtain maximum cottonseed oil extraction , the current study compared Soxhlet, mechanical, and ultrasound-assisted extraction techniques. Using Box-Behnken 34 experimental design, extraction efficiency

was evaluated based on influences of temperature, organic solvent, extraction time, and solvent:seed ratio. Using 10:1 ratio and Folch combination as operating system, ultrasound-assisted extraction obtained highest extraction yield (38.25%) from our tests at 45 °C after 1 hour. The primary leftovers in characterised CSO were palmitic, oleic, and linoleic acids, and used to make biodiesel. The goal of this study is to encourage use of this agricultural byproduct to enhance value of cottonseed [5].

3.4. Benjamin Durakovic et al, 2018 - A statistical tool used in different forms of system, process, and product design, development, and optimization is called design of experiments. It is versatile tool that may be applied in number of circumstances, including design for comparisons, variable screening, transfer function discovery, optimization, resilient design. This article examines development of DOE over time and presents state-of-the-art in its use. It also instructs researchers on how to construct, organise, and carry out experiments and how to evaluate and interpret results using examples. DOE applications are advancing quickly in both manufacturing and non-manufacturing areas. It has the most applicability in fields of medical, engineering, biochemistry, physics, and computer science. It is subfield of applied statistics that is utilised to carry out scientific investigations of system, process, or product where input variables (Xs) were changed to examine their impacts on measured response variable (Y). DOE has been highly helpful instrument for improving product quality and reliability over past 20 years. The use of DOE in decision-making processes for new product development, industrial process optimization, and other areas has grown across numerous industries [6].

3.5. N. Vasumathi, T. V. Vijaya Kumar et al., 2016 proposed a study on the beneficiation of non-coking coal by flotation technique and the concentrates produced can be used as a fuel in IGCC (integrated gasification combined cycle) plant for electricity production as the ash content of non-coking coal is reduced to less than 15% . The non-coking initially with 34.6% ash were reduced to 14.83% ash content by optimizing the flotation conditions. The collector used here is diesel (dosage - 2.87 kg/t) and MIBC as frother (dosage – 0.65 kg/t) [29].

CHAPTER 4

MATERIALS AND METHODS

4.1. MATERIALS

Cotton seeds were dried under UV light, ground and used as feed for soxhlet extraction studies. Used cotton seed oil extracted from cotton is taken as a collector reagent. Non-coking coal is taken as the feed for flotation.

4.2. CHARACTERISATION TECHNIQUES

4.2.1. Fourier transform infrared spectroscopy

FTIR is an analytical tool used for determining the structural details of a material and it does not destroy the sample, it is a faster technique, it is more precise and sensitive. It is based on the principle that when a sample is radiated by infrared radiation some of the radiation is passed through the sample and some radiation is absorbed by the sample. The IR radiation absorbed by the molecule at specific frequencies based on the type of atom or molecule present and the type of bond present between them.



Fig.4.1. FTIR absorption spectrometer

The radiation that is absorbed by the sample causes change in the vibrational energy level of molecule which is measured to obtain a spectrum. Each molecules have different structures forming various spectra. The spectra are like a fingerprint for the molecules from which the spectrum and the functional groups present in a particular molecule can be determined. It is a formidable tool for identifying the organic compounds having polar bonds [17].

4.2.2. Gas chromatography- Mass spectrometry (GC-MS)

4.2.2.a. Instrumentation

The gas chromatograph and the mass spectrometer are the two main components that make up the GC-MS. The capillary column used by the gas chromatograph depends on the phase qualities as well as the column's dimensions (length, diameter, and film thickness). As the sample moves down the length of the column, the separation of molecules will be aided by the differences in chemical characteristics of the various molecules in the mixture and their varying affinities for the stationary phase of the column. The retention time of the molecules permits the mass spectrometer downstream to catch, ionise, accelerate, deflect, and detect the ionised molecules individually because the molecules are kept by the column and then elute (come off) from the column at distinct times. This is accomplished by the mass spectrometer by fragmenting each molecule into ionised parts and identifying these parts using their mass-to-charge ratios.

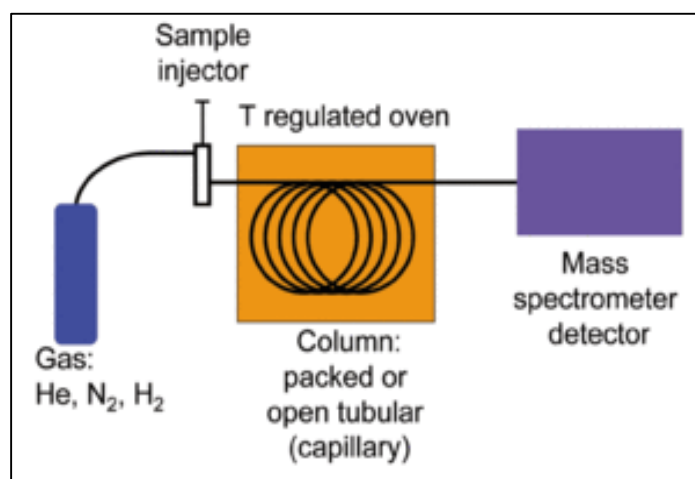


Fig.4.2. GC- MS instrument

When used combined, these two elements provide a far finer level of substance identification than when employed independently. Gas chromatography

or mass spectrometry alone cannot be used to accurately identify a specific chemical. While gas chromatography using a conventional detector (such as a Flame Ionization detector) is unable to distinguish between multiple molecules that just so happen to take the same amount of time to travel through the column (i.e., have the same retention time), this leads to two or more molecules that co-elute in the mass spectrometry process, which typically requires a very pure sample.

With a mass spectrometer, two distinct molecules can occasionally produce a same pattern of ionised particles (mass spectrum). Since it is highly rare that two different molecules will behave identically in a gas chromatograph and a mass spectrometer, combining the two techniques lessens the chance of error. Hence, it usually improves confidence that the target analyte is present in the sample when an identifying mass spectrum appears at a typical retention time in a GC-MS study.

A purge and trap (P&T) concentrator system may be used to introduce samples for the examination of volatile chemicals. Purging or sparging is the process of combining the sample with water and purging it with an inert gas (such as nitrogen gas) inside of an airtight chamber in order to recover the target analytes. Following their movement into the headspace above the water, the volatile chemicals are pulled out of the chamber along a pressure gradient (produced by the entry of the purge gas). Onto a "trap," the volatile substances are dragged along a hot line. The chemicals are held in the trap by being returned to the liquid phase in a column of adsorbent material that is at room temperature. The sample chemicals are then introduced to the GC-MS column by a volatiles interface, which is a split inlet system, once the trap has been heated. Volatile organic compounds (VOCs) and BTEX chemicals respond well to P&T GC-MS (aromatic compounds associated with petroleum).

The "purge-closed loop" technique is a quicker substitute. The inert gas is bubbled through the water in this system until the concentrations of organic molecules in the vapour phase and aqueous phase are in balance. The gas phase is then immediately analysed.

4.2.2.b. Types Of Mass Spectrometer Detectors

The quadrupole mass spectrometer, frequently referred to by the Hewlett-Packard (now Agilent 1200) brand name "Mass Selective Detector," is the most popular type of mass spectrometer (MS) used in conjunction with a gas chromatograph (GC) (MSD). The ion trap mass spectrometer is another widely used detector. A magnetic sector mass spectrometer may also be included, although these tools are pricey and large, and therefore are not frequently encountered in high-throughput service laboratories. Time of flight (TOF), tandem quadrupoles (MS-MS) (see below), or an ion trap MS, where n denotes the number of mass spectrometry stages, are examples of additional detectors that might be used.

4.2.2.c. Applications

An analytical technique called gas chromatography-mass spectrometry (GC-MS) combines the advantages of gas chromatography and mass spectrometry to identify various compounds in a test sample. Drug detection, fire investigation, explosives investigation, and sample identification are all applications of GC-MS. This includes the identification of unknown samples, such as those taken from the planet Mars during early 1970s probe expeditions. Moreover, GC-MS can be utilised at airport security checkpoints to find drugs on passengers or in their luggage. Moreover, items that were previously believed to have decomposed beyond identification can have trace components found in them. It enables examination and detection of even minute amounts of a chemical, similar to liquid chromatography-mass spectrometry.

Because it is utilised to conduct a 100% specific test that positively detects the presence of a specific chemical, GC-MS has come to be known as the "gold standard" for forensic substance identification. Any of the many chemicals in a category of substances may be detected by a nonspecific test. A non-specific test may statistically indicate the substance's identity, but this could result in erroneous positive identification [36].

4.3. SOXHLET EXTRACTION METHOD

It is a process of continuous liquid-solid extractions when the compound to be extracted has limited solubility and the impurities insoluble in the chosen solvent. It is used for the extraction of bioactive substances from natural products.

During the process, the vapours of the solvent flow above to the path of distillation and into the main chamber which contains the condenser. The vapour of the solvent gets condensed and drips into the feed material placed inside the thimble. Once the siphon tube is filled with the extracted compound it starts to drip down into the round bottom (RB) flask leaving behind the insoluble particles in the thimble and the cycle continues again till the feed is completely extracted [18].

4.3.1. Working principle

It is based on the principle of

- Evaporation – the solvent gets evaporated when heated to boiling.
- Condensation – the evaporated solvent gets condensed and drips down into the feed material.
- Percolation – the flow of the condensed solvent through the bed of feed substance to get extract [19].

4.3.2. Experimental conditions

- Weight of the feed (used cotton seed powder) is 50g.
- Amount of solvent (n-hexane) is 150ml.
- Temperature of solvent is maintained at 70°C.
- Pump rotation was set at 152 rpm

4.3.3. Working mechanism of soxhlet apparatus

- The feed sample which is dried and sieved is weighed 50 g.
- It is then transferred on the cotton bed placed at bottom of the thimble and another bed of cotton is placed on top of the feed and packed tightly.
- In the round bottom flask 150 ml of n-hexane based on 1:3 ratio of feed and solvent is measured and added.

- The RB flask is then placed inside a water bath whose temperature is set at 70°C since the boiling point of n-hexane is 69°C.
- The Soxhlet extractor is then fitted to the RB flask tightly.
- The condenser is placed above the extractor and fitted firmly with the help of a burette stand.
- The opening of the condenser is sealed with cotton to avoid letting out any solvent vapours.
- The inlet and outlet tube of the condenser is dipped inside an ice bath to make it cool.
- The condenser should be maintained in cold condition so that the vapours can be easily condensed to liquid.
- The pump is set to rotate at 152 rpm to continuously circulate the water in and out of the condenser.
- As the solvent begins to boil it starts to evaporate and travels up through the distillation path into the extractor.
- Where the vapours get condensed into liquid and drips down into the thimble containing feed material.
- The solution extracted from the feed starts to fill into the siphon tube once it is filled it starts to drip down back into the RB flask.
- This complete process is referred as a cycle which involves evaporation, condensation, and percolation of the solvent.
- The process is repeated for several cycles until the extracted solution becomes light in colour and the insoluble feed are left back inside the thimble.
- After the complete extraction, RB flask is removed from the water bath.
- The extracted solution is transferred into a beaker to calculate the weight of extracted solution.
- The obtained solution contains the volatile solvent and non-volatile sample which needs to be separated.
- The sample is separated from the solvent using rotavapor to obtain used cotton seed oil which is used for flotation.



Fig.4.3. Soxhlet extraction setup

4.3.4. Experimental designing of soxhlet extraction

- The extraction process is done for feed materials with varying parameters such as feed size and temperature of the solvent.
- The feed sizes taken are 300, 600, 850 μm and 1 mm.
- Temperature is varied by 70 and 75°C.
- The orthogonal matrix that is chosen is L8 matrix.
- The input parameters are feed size and temperature where the output parameters are extracted solution and oil yield.
- The solution obtained after each extraction is poured into a separate beaker and measured to calculate the oil yield of each parameters fixed.
- After calculating the oil yield the extracted solution is taken for solvent and oil separation using rotavapor.

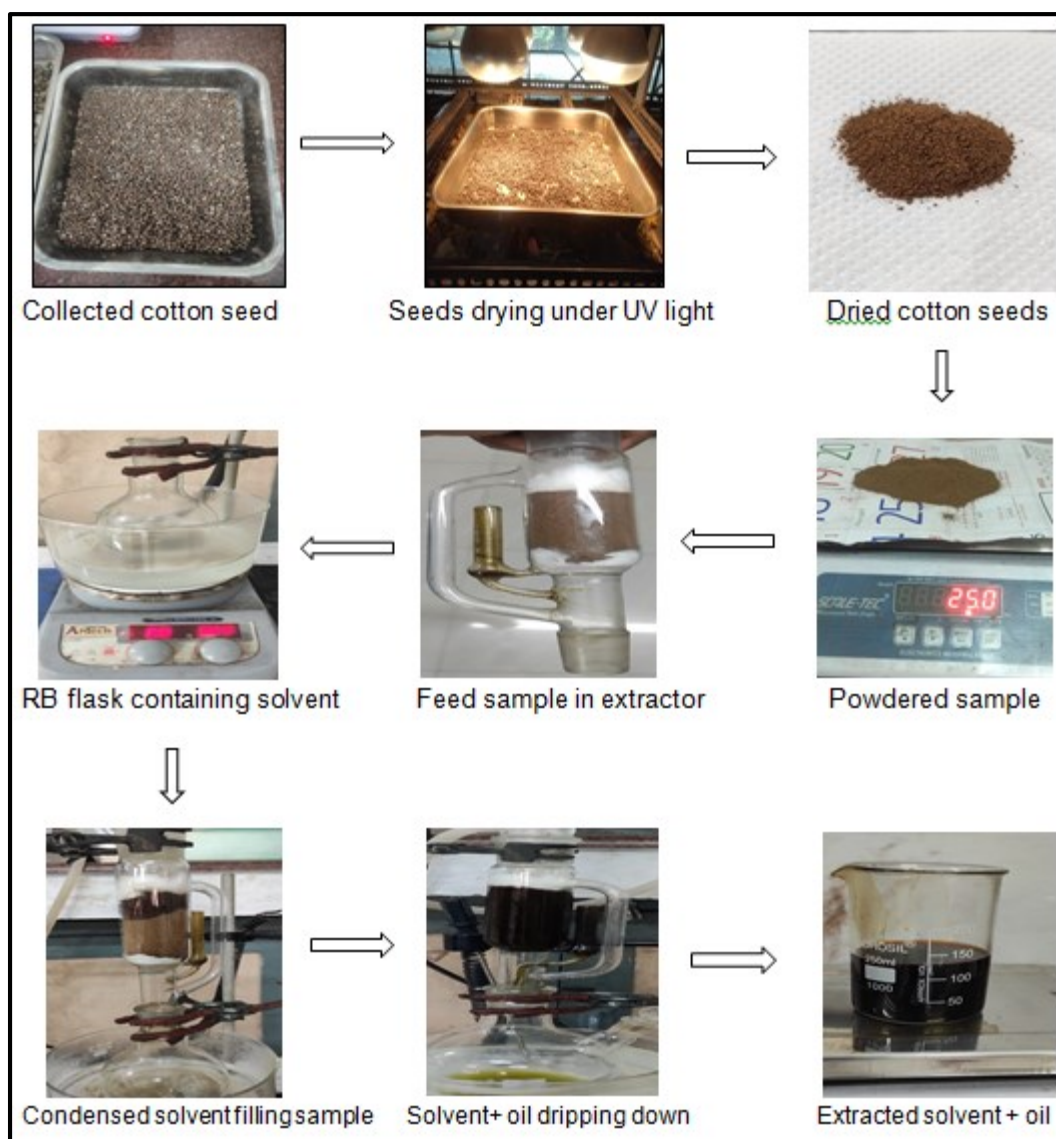


Fig.4.4. Flow chart of Soxhlet extraction

4.3.5. Rotatory evaporator

It is the instrument used to separate the oil of feed material and a solvent under reduced pressure or vacuum by evaporation. It is used to separate solvents with low boiling point like hexane. The reduced pressure allows the extracted solution to boil at a temperature lesser than normal boiling temperature [20]. Rotavapor is an efficient way to separate a non-volatile sample from a volatile solvent [21].

4.3.5.1 Principle

Rotavapor works on the principle of increasing the evaporation rate of solvent by:

- Lowering the pressure to reduce the solvent boiling point
- Rotation of sample for increasing the surface area
- Heating the water bath containing water

4.3.5.2. Rotavapor conditions

- Pressure – the pressure was set at 370 mbar.
- Temperature – the temperature was maintained at 40°C.
- Rotation – the rotation of flask containing the extracted solution was kept at level 3.



Fig.4.5. Rotary evaporator

4.3.5.3. Working mechanism of rotary evaporator

- The flask with the extraction sample and the round bottom flask for collecting the solvent are fitted to the rotavapor.
- The height of the ramp is then adjusted so that the flask does not touch the corners of the water bath.
- Then water is filled into the water bath till the flask dips into the water and the temperature of water bath is fixed at 40°C. The normal boiling point of n-

hexane is 69°C but due to the presence of reduced pressure its boiling point is reduced to 40°C.

- Now water is allowed to flow into the condenser and pressure is set at 370 mbar because it is the standard pressure of n-hexane.
- The flask is then rotated as the solvent starts to evaporate, if there is any occurrence of bubbles inside the flask the pressure is let out a little and then closed.
- As the solvent evaporates it gets condensed and drips down into the solvent flask.
- After the dripping stops which means that all the solvent present in the sample has been separated, the vacuum pump is switched off.
- Then the water flow into the condenser is stopped, the flask is removed carefully, and the yield of oil extracted is measured.
- The yield of oil is measured using the formula,

$$\% \text{ yield} = \frac{\text{Wt.of the oil extracted}}{\text{Wt.of the sample feed}} \times 100 \%$$

4.3.6. Sieve analysis

The dried feed material is separated into different micron sizes using sieves of size 300, 600 and 850 µm and 1 mm.



Fig.4.6. Sieves

4.3.7. Ball milling

It reduces the size of a material by grinding it into a fine powder. The ball mill consists of a hollow cylinder which rotates about its horizontal axis . The cylinder consists of iron balls weighing approximately 2kg.

4.3.7.1. Principle

Ball mill depends on two main principles impact and attrition which are responsible for size reduction of materials by rapidly moving the steel balls.

- Impact – It is the pressure put forth by two heavy objects.
- Attrition – It is the reduction of material size when they hit with the steel balls.

4.3.7.2. Main parts in ball mill

- Cylinder – A hollow metal whose length is larger than its diameter.
- Balls – It is made up of stainless steel whose size depends upon the diameter of the cylinder. It covers about 50-30% area inside the hollow cylinder [22].

4.3.7.3. Grinding method

- Sample weight – A sample of weight about 300gm of grade E non coking coal.
- Grinding time – The sample is grinded for 40 minutes.
- Water – About 165 ml of water is added to grind the sample.



Fig.4.7. Ball mill

4.3.7.4. Working mechanism of grinding mill

- It is first filled with water and then steel balls were added to it and it is made to run for 5 to 10 minutes to remove any substances present inside the cylinder.
- Then the water and balls are removed by placing the opening of cylinder facing downwards.
- Now the sample is fed into the cylinder carefully, then steel balls and water are added to it then the lid is tightly closed.
- It is then made to run for 40 minutes to grind the sample into a fine powder.
- The sample is then collected from the cylinder and cylinder is washed well with water to remove any sample present in it.

4.4. FROTH FLOTATION PROCESS

The non-coking sample obtained after ball mill grinding is left to settle for a day and then decanted to remove excess of water. The slurry is then transformed into the flotation cell and then placed in the column.

4.4.1. Floatation conditions

- Collector dosage – 4 ml of used tea powder oil
- RPM (rotation per minute) – 900 rpm
- AFR (air flow rate) – 3.5 lpm (liquid per minute)

4.4.2. Working mechanism

- The flotation cell is switched on setting the rpm at 900.
- After 5 minutes, the mentioned amount of collector and frother is added to the slurry.
- A little amount of water is added to make the slurry level to $\frac{3}{4}$ th of the flotation cell.
- Now, air pressure is set to 3.5 lpm and the slurry starts to form froth which is collected in a cleaned and labelled tray.
- The timer is started as soon as the collection of froth has been started and this process is called roughing.

- The collection of froth is continued till the formation of froth is stopped completely.
- After collecting all the froth, the cell is switched off and air pressure is brought back to zero.
- The timer is then stopped, and the collection time is noted.
- The slurry in the cell is the primary tailings (PT) which is transformed into a labelled tray and allowed to settle down.
- The froth produced from roughing is again poured into the cell and the cell is turned on and air pressure is kept at 3,5 lpm.
- The froth has started to form, and timer is switched on. The froth is collected till it stops to form and the collection time is noted.
- Now the slurry in the cell is the cleaner 1 tailing (CL 1T) it is transferred into the tray and allowed to settle.
- The collected froth from cleaner 1 tailing is again transferred into the cell and the frothing process is repeated.
- It is then transformed into a tray labelled as cleaner 2 tailing (CL 2T) and left to settle.
- Likewise 3rd (CL 3T) and 4th (CL 4T) tailing happen in same manner.
- The froth present in the tray after 4th cleaning is the final concentrate (FC).
- The trays are then kept in oven for drying after removing excess water from the trays.



Fig.4.8. Froth obtained from flotation of non-coking coal

4.4.3. Sampling methods

Sampling method is a technique used to obtain a small weight of sample representing a bulk of sample. This method is based on probability and care must be taken since the sample represents the whole product. In this work the sampling method that is used is coning and quartering method.

4.4.3.1. Coning and quartering method

- It is mostly used in laboratories for the sampling of less bulk samples.
- This method is inexpensive and can be used for any type of ores.
- It is the primary method used for sampling.
- It is an old Cornish method applied for dividing the material samples.
- The sample is transformed into a clean paper after it has been scrubbed off using a big size spatula from the tray used for drying.
- Then it is weighed, and the weight of the sample is noted.
- The sample is then mixed well and made into a conical heap.
- Using a Petri dish, the cone is pressed slowly to form a circle with equal thickness.

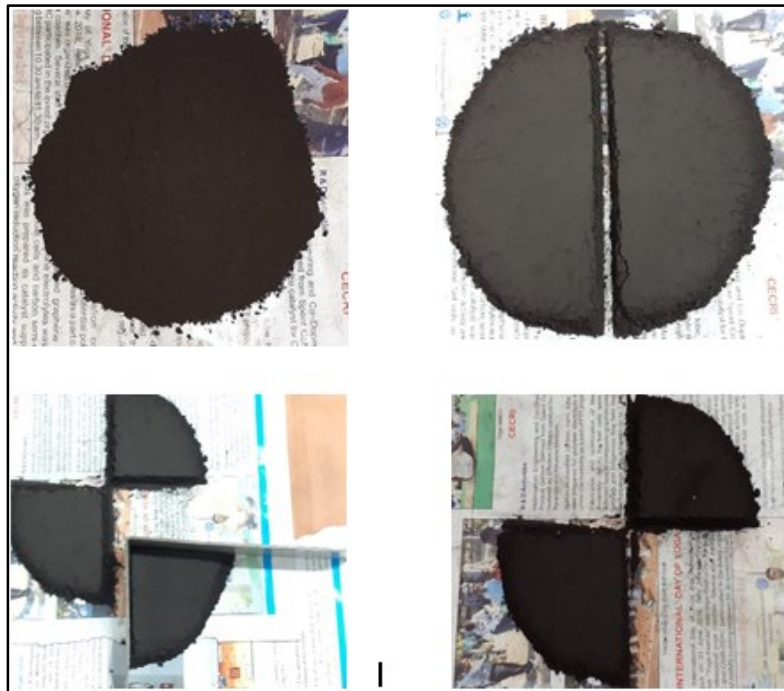


Fig.4.9. Cone and quartering process

- The circle is then cut into four halves using a cross cutter of 90° angle.
- Then the two opposite samples are taken out in a separate paper and kept in a zip lock cover.
- The remaining two halves is again coned and then quartered till the sample of required weight is obtained [23].

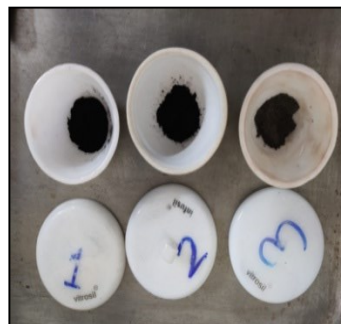
The products are dried well and sampling – coning and quartering method is done.

4.4.4. Ash analysis

For ash analysis porcelain crucibles are used as they can withstand elevated temperature. The crucibles are washed and dried well. Then the products obtained after sampling is weighed approximately 0.5 gm in the crucibles and kept in furnace for about 2.5 hours at a temperature of 900°C. After the crucibles are cooled, weight is taken, and ash percentage is calculated.



Fig.4.10. Furnace with samples



4.5. DESIGN OF EXPERIMENTS VIA TAGUCHI-ANOVA METHOD

The MINITAB18 Statistical Software is sophisticated statistics software that can conduct regressions, ANOVA, control charts, DOE, and a variety of other statistical analyses. It is particularly helpful for developing and analyzing findings for DOE projects and allows building DOEs using the factorial, response surface, mixed, and taguchi methods. In this study, the design of experiments was incorporated using taguchi method [32].

The Taguchi technique entails decreasing variation in a process through firm experiment design. This technique was devised to study how various parameters influence the mean as well as the variance of a process performance characteristic that determines how well the process in being effective. Taguchi's experimental design calls for the use of orthogonal grids to arrange the process parameters and the levels at which they should be examined. The Taguchi technique examines pairs of combos rather than all potential combinations, as the factorial design does. This enables the collection of data required to determine which variables most influence the product quality with the least amount of experimentation, saving both time and resources.

Table 4.1. Ranges and levels of input parameters in Taguchi design

Process parameters	Parameter designation	Levels and Range			
		1	2	3	4
Feed size (μm)	A	350	600	850	1000
Temperature ($^{\circ}\text{C}$)	B	70	75		

A conventional design of experiments employs numerous experiments and prioritizes on how sources influence the outcomes. The Taguchi technique, on the other hand, requires far fewer experimental tests to understand the impact of various input parameters on the resultant product. In this research, with four levels of two process parameters: feed size (μm) and temperature ($^{\circ}\text{C}$) as mentioned in Table 1. The results of all trails were then assessed statistically. ANOVA analysis was also carried out to determine the importance of the input factors [26]

CHAPTER 5

RESULT AND DISCUSSION

As a result of this review, the extraction of oil from cotton seed was performed by Soxhlet extraction technique. This technique is done utilizing a chosen natural dissolvable which is n-Hexane and boundaries like time, temperature and decrease of the molecule size. The outcomes are organized and the diagrams are plotted for every boundary. The assurance and portrayal of oil from cotton seed are additionally illustrated in this review.

5.1 DESIGN OF EXPERIMENT (DOE) OF SOXHLET EXTRACTION

The cotton seed sample with different micron sizes like 300 μm , 600 μm , 850 μm and 1mm are subjected to Soxhlet extraction with n-hexane as a solvent for extraction. The yield obtained for the extraction solution obtained from 50 g of used cotton seed powder and 150 ml of n-hexane as solvent has been given in Table 5.1.

Table.5.1. Tabulation of the calculated oil yield

Run no.	Feed size (μm)	Temperature ($^{\circ}\text{C}$)	Oil extracted (gm)	Oil yield (%)
1	300	70	14.13	28.26
2	300	75	10.46	20.92
3	600	70	5.08	16.93
4	600	75	14.3	20.42
5	850	70	9.65	19.3
6	850	75	8.95	17.9
7	1000	70	9.82	19.64
8	1000	75	11.13	22.26

5.2. FTIR RESULTS

The FTIR spectra of the extracted cotton seed oil was recorded in the absorbance mode in the range of 4000 –500 cm^{-1} . The strong band at 2922 cm^{-1} indicated the stretching

vibration of alkanes (-C-H) group [33]. The peak at 2853 cm^{-1} represents the -CH symmetric stretching of the methyl group in aliphatic compounds [31]. The strong band at 1743 cm^{-1} is due to the -C=O stretching vibrational mode [27]. The medium peak at 1452 cm^{-1} represents the presence of amine group. The weak band at 1377 cm^{-1} denotes the -C-CHO bending vibrational mode [30]. The strong band at 1161 cm^{-1} is due to the stretching vibration of -C-O-C. The peak at 1098 cm^{-1} is due to the presence of ether and ester (-C-O) stretching vibrational mode [33]. The peak at 722 cm^{-1} corresponds to the -C-C-, CH_3 and CH_2 functional groups [21].

X-axis denotes the wavenumber (cm^{-1}) and Y-axis denotes the absorbance (%).

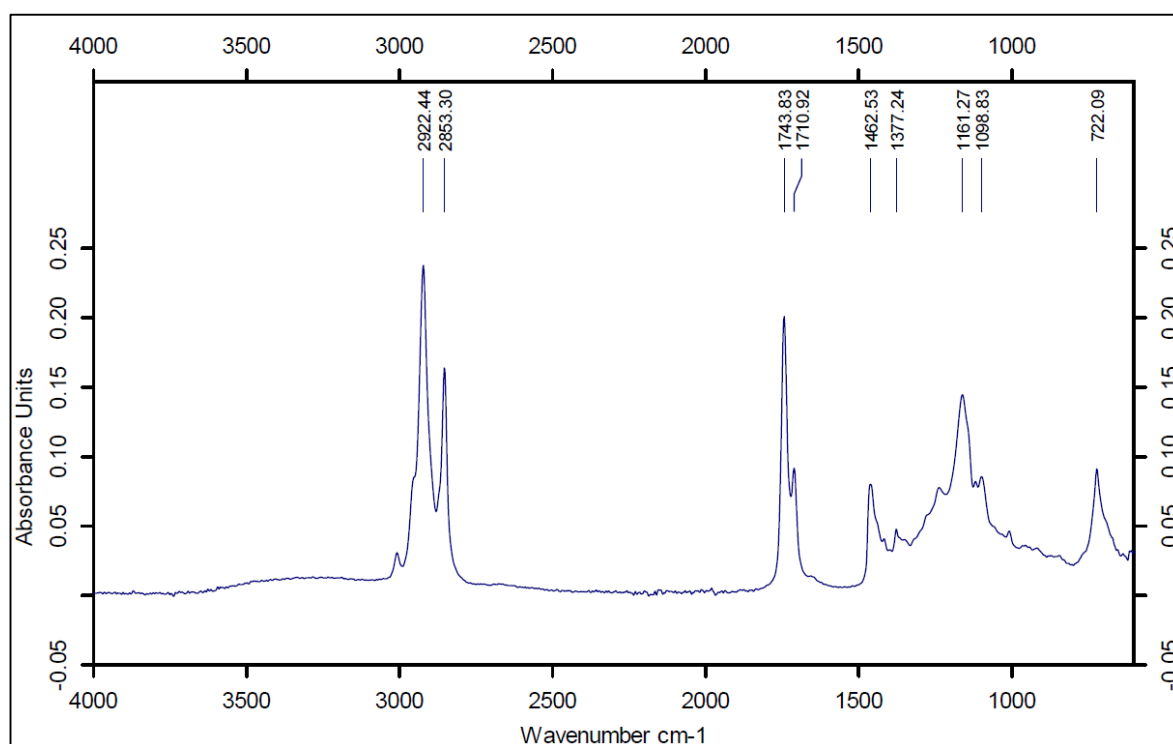


Fig.5.1. Graphical representation of FTIR

5.3. GAS CHROMATOGRAPHY (GC)

The GC results of the extracted Cucurbita pepo seed oil is observed from the above graph. The free fatty acid composition in the extracted oil mainly consists of C14-myristic acid with 0.297%, C16- Palmitic acid with 23.438%, C16.1 – Palmitoleic acid with 0.398%, C18.0 – Stearic acid with 2.527%, C18.1 – Oleic acid with 25.781%, C18.2 – Linoleic acid with 46.08%, C18.3 – Linolenic acid with 0.138%, C20 – Arachidic acid with 0.576%, C20.1 – Gadoleic acid with 0.16%, C22 – Behenic acid with 0.419% and

C24- Lignoceric acid with 0.185% in which the percentage of linoleic acid and oleic acid were higher, the oil was used as the bio-collector in the froth flotation process.

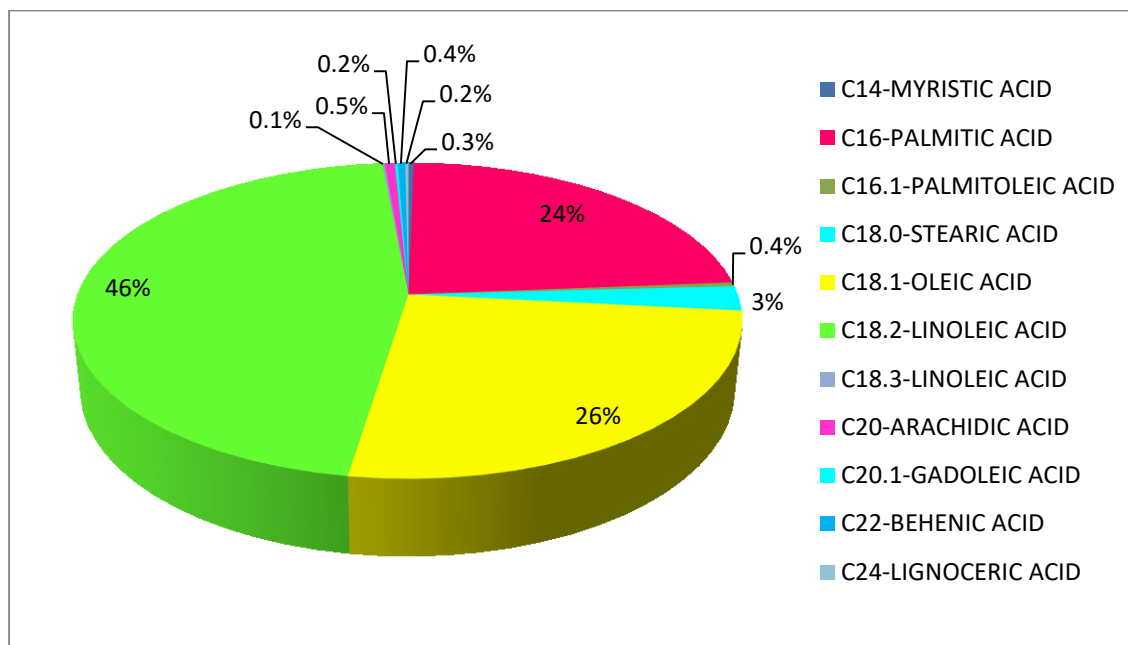


Fig.5.2. GC graph of the extracted oil

Table.5.2. FFA composition of the cotton seed oil

Sl. No.	FFA composition	Retention time (min)	Area%
1	C14 – Myristic acid	5.202	0.297
2	C16 – Palmitic acid	6.862	23.438
3	C16.1 Palmitoleic acid	7.295	0.398
4	C18.0 Stearic acid	9.017	2.527
5	C18.1 – Oleic acid	9.553	25.781
6	C18.2 – Linoleic acid	10.437	46.08
7	C18.3 – Linolenic acid	11.383	0.138
8	C20 – Arachidic acid	11.538	0.576
9	C20.2 – Gadoleic acid	11.800	0.16
10	C22 – Behenic acid	14.265	0.419
11	C24 – Lignoceric acid	16.992	0.185

Table.5.2. FFA composition of the cotton seed oil

5.4. FLOTATION PROCESS

The oil extracted is used as collector reagent for the flotation of non-coking coal. The products obtained after flotation are calculated for their weight and ash percentage

Table.5.3. Results of non- coking coal flotation

Reagent	Sample	Weight %	Ash %	Ash distribution %
Cotton seed oil	FC	42.26	9.32	11.66
	CL- 4T	6.04	17.71	3.17
	CL- 3T	4.83	24.77	3.55
	CL- 2T	5.89	35.20	6.14
	CL- 1T	10.87	53.30	17.16
	PT	30.11	30.11	58.33
Head calculated				33.77

5.5. TAGUCHI RESULTS

The Taguchi method is primarily concerned with improving the impairment of its function. Essentially, the deviation of a value from its distinctive nominal value is maximized in this method. It primarily provides a summary of the unpredictable variables referred to as noise in this method and the parameters accountable for the deviation of experimental findings. As a result, it provides an array of various runs (trials) for performing the experiments, ensuring that the design of tests generated is sufficiently durable to provide optimum responses . The system is enhanced by minimizing the noise by managing the mean variables. This design employs a two-step optimization process [Source: MINITAB18] .

Step 1: The signal-to-noise ratios are taken into account in order to optimize the variables considered in the trial design. The loss functions proposed by Taguchi are used to determine the signals to noise (SN) ratios. In the present study, the condition “Larger is Better” is used to calculate the SN ratio in order to assess the impact of design variables [34] on the flotation studies of the sample as most significant of the outcomes were desirable. The “Larger is better” type was chosen in this research to target the outcomes, which are ash rejection, combustible recovery, and separation efficiency [34]. They can be calculated using the following arithmetic expression:

$$\frac{S}{N_{Bigger}} = -10 \log \left(\frac{\sum \frac{1}{y^2}}{n} \right)$$

Where, n is the experimental run number, Y is the quality characteristics, $\bar{Y}$ is the mean quality characteristics and s is the sum of variance.

The mean of S/N ratio against all three input factors at different states is disclosed in the Table 5.4. The mean of S/N ratio is the average of each run's S/N ratio and the overall number of trials. Higher the S/N ratio, greater is the response rate.

Table 5.4. Response table 1 for signal to noise(S/N) ratios

Input parameters	Mean S/N ratio				Max-Min (Δ)	Rank
	Level 1	Level 2	Level 3	Level 4		
Yield % (Larger is better)						
A: Feed size (μm)	27.72	25.39	25.38	26.41	2.33	1
B: Temperature (°C)	26.29	26.15			0.41	2

Step 2: Identifying the control variables that assist the mean to reach the desired value while having minimal effect on the S/N ratio.

The outcome for means is shown in Table 5.5. It provides the mean of means value of all three control variables at each level. The variation in the input parameters can be identified by comparing the means and surmised by examining the variance. Because the target is to optimize the responses, factor values with the largest mean value chosen.

Table 5.5. Response table for mean of means

Input parameters	Mean S/N ratio				Max-Min (Δ)	Rank
	Level 1	Level 2	Level 3	Level 4		
Yield % (Larger is better)						
A: Feed size (μm)	24.59	18.68	18.60	20.95	5.99	1

B: Temperature (°C)	21.03	20.38			0.65	2
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According to Table 5.4 and 5.5, the order of significance of control variables in the instance of yield% is as following order below:

Table 5.6. Order of significance and optimal conditions of single response characterization

Responses	Order of significance	Optimal condition
Yield %	Feed size > Temperature	A1- B1

5.5.1. Effect of input parameters on yield %

Figure 5.3 depicts the mean yield value at all levels corresponding to the various control factors. Higher the mean value, greater is the response traits. The mean value of yield ranges from 18 to 25%. The feed size (A) has the highest and the lowest mean value. The optimum mean value for yield was achieved at 350µm feed size with temperature at 70°C.

Graph

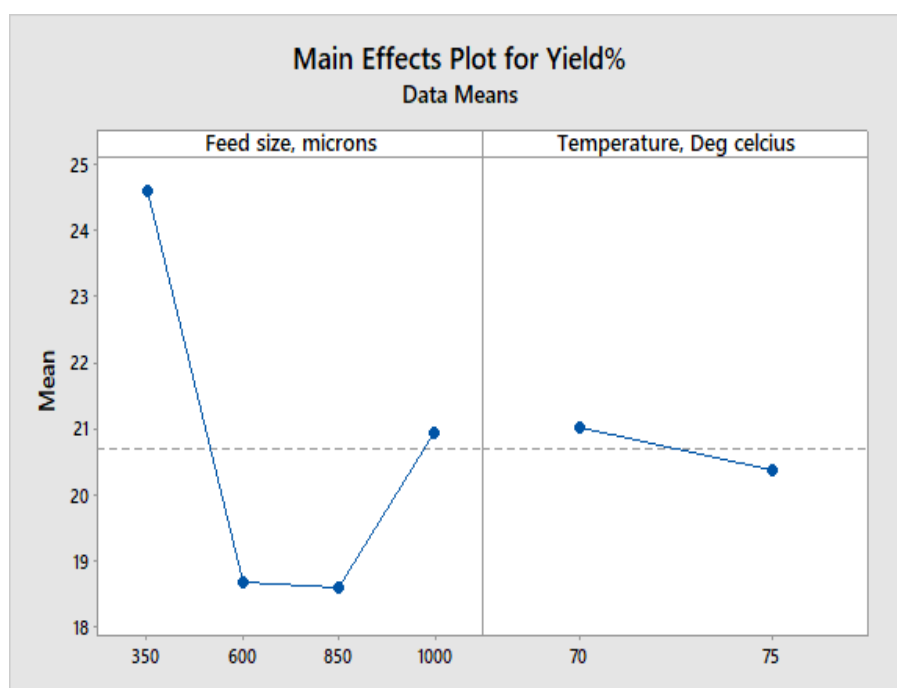
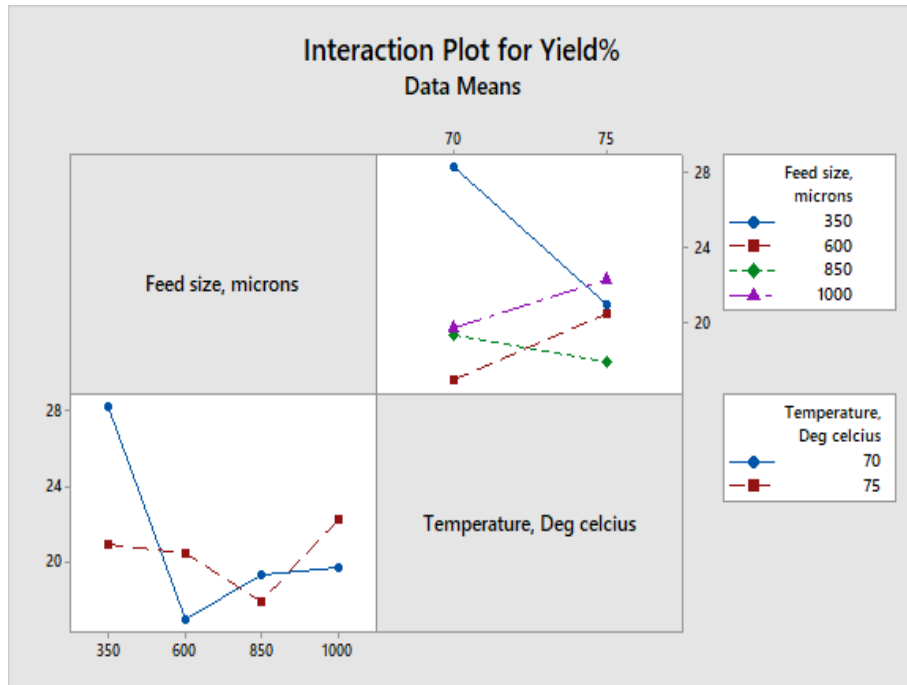


Fig.5.3. Input parameters versus mean yield%

The main effect plot for yield% shows that the effect of feed size on yield is high whereas, the impact of the temperature doesn't affect the mean yield%.

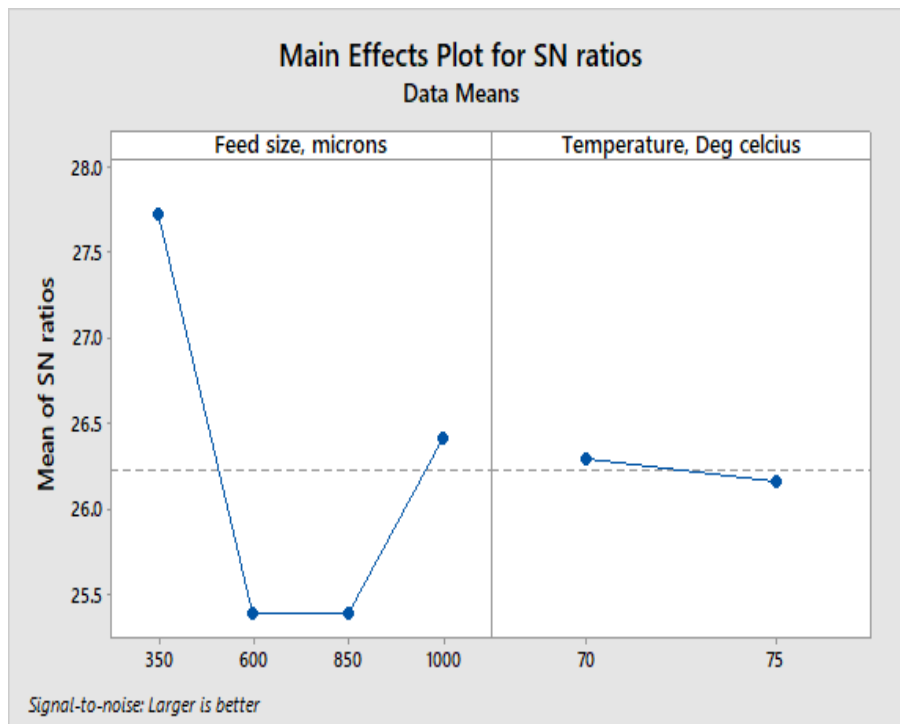


Graph

Fig.5.4.

5.5.2. Effect of input parameters on s/n ratio

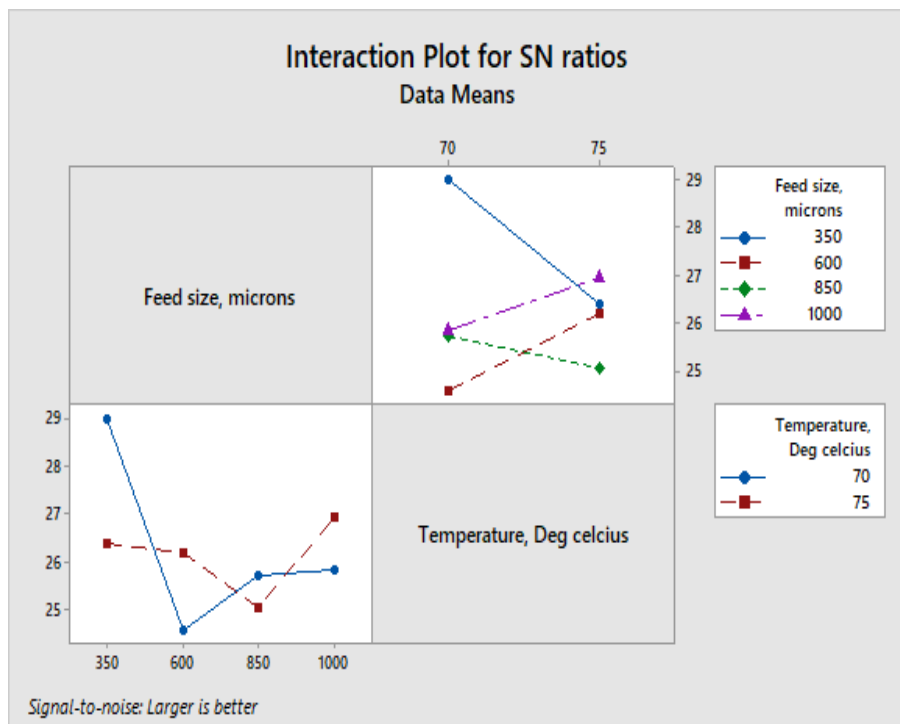
Figure 5.5 depicts the mean yield value at all levels corresponding to the various control factors. Higher the mean value, greater is the response traits. The mean value of yield ranges from 25 to 28 %. The feed size (A) has the highest and the lowest mean value. At level one, the feed size (A) has the greatest mean yield value of 27%, while the other control factors (i.e.) temperature (B) has the highest mean yield value of 26% respectively. As a result, the optimal control factor values for the finest yield was identified as A1-B1. The optimum mean value for yield was achieved at 350 μ m feed size with temperature at 70°C.



Graph 5..5

Fig.5.5. Input parameters versus mean of means

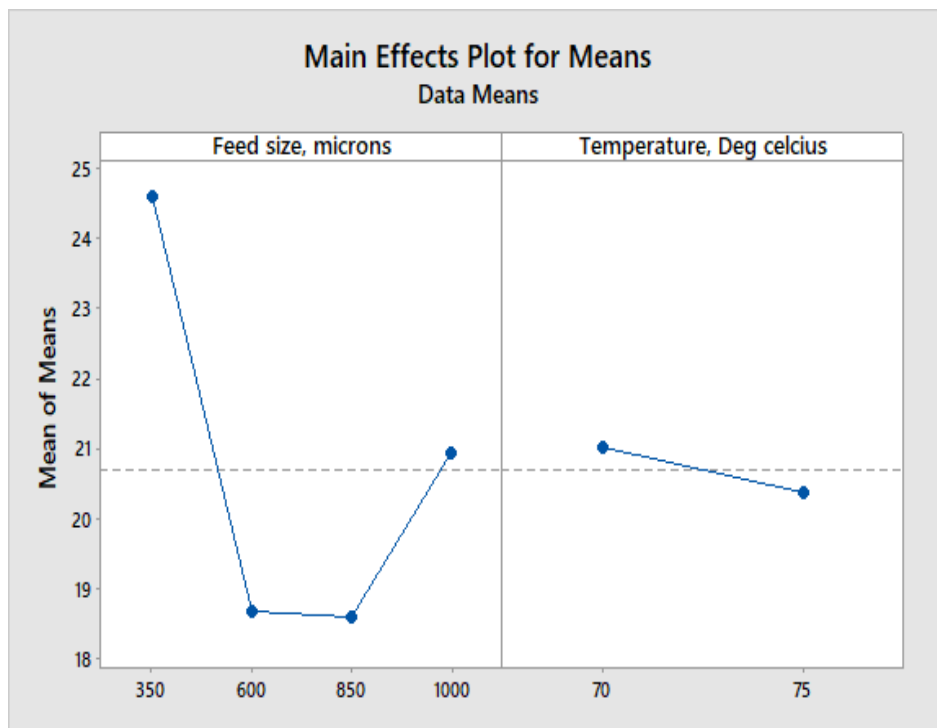
The main effect plot for yield% shows that the effect of feed size on yield is high whereas, the impact of the temperature doesn't affect the mean yield%.



Graph Fig.5.6.

5.5.3. Effect of input parameters on mean and means

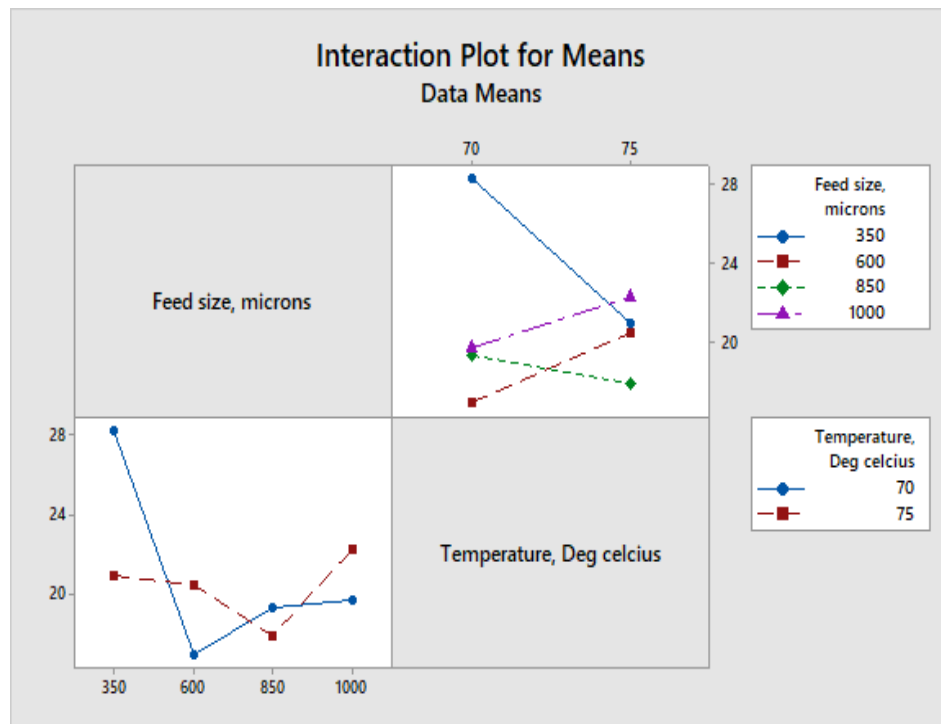
Figure 5.7 depicts the mean yield value at all levels corresponding to the various control factors. Higher the mean value, greater is the response traits. The mean value of yield ranges from 18 to 25 %. The feed size (A) has the highest and the lowest mean value. At level one, the feed size (A) has the greatest mean yield value of 24%, while the other control factors (i.e.) temperature (B) has the highest mean yield value of 21% respectively. As a result, the optimal control factor values for the finest yield was identified as A1-B1. The optimum mean value for yield was achieved at 350 μ m feed size with temperature at 70°C.



Graph 5.7

Fig.5.7. Input parameters versus mean of means

The main effect plot for yield% shows that the effect of feed size on yield is high whereas, the impact of the temperature doesn't affect the mean yield%.



Graph Fig.5.8.

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