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Department of Petroleum Engineering

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Oil and Natural Gas Corporation Limited
Eastern Offshore Asset
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NO.KKD/EOA/HR/Internship/2023 Date: 13.04.2023

Certificate

This is to certify that Mr. **BIJJAM SRINIVAS REDDY** student of **4st Year B.Tech (Petroleum Engineering)** at **VIGNAN'S UNIVERSITY, GUNTUR** has successfully completed Internship entitled **OVERVIEW OF ODALAREVU ONSHORE TERMINAL** of ONGC, Eastern Offshore Asset, Kakinada during the Training, undergone from **01.03.2023** to **14.04.2023** During the training he took keen interest in the assigned work and his conduct is satisfactory.

We wish him all the success in future academic endeavors.

13/04/23.
Company Guide

13/4/2023
GM (HR) – Head (HR/ER)
R. GOVIND NAIK.
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CIN : U24110AP1989PLC009723

HETERO LABS LIMITED (UNIT-III)

Sy. No. : 120 & 128, 150 (PART), 150/1, 151/2, 158/1, N. Narasapuram (Village),
Nallamattipalem (V), Nakkapalli (Mandal), Visakhapatnam (Dist.) - 531 081, A.P., INDIA.
Tele Phone : +91-891-2877900, Fax : +91-891-2877933
E-mail : contact@heterodrugs.com. URL : http//www.heterodrugs.com.

Date: 15-05-2023
Place: Nakkapalli

TO WHOM IT MAY CONCERN

We are glad to inform you that Mr. **B. TARUN KUMAR** from Vignan' s Foundation for Science Technology and Research has successfully completed his internship at Hetero Labs Limited from 15-02-2023 to 15-05-2023.

During his internship, he was exposed to the various activities in Environment Health Safety.

We found him very inquisitive and hard working. He was very much interested to learn the functions of our core division and also willing to put his best efforts and get in to the depth of the subject to understand it better.

His association with us was very fruitful and we wish him all the best in his future endeavours.

For Hetero Labs Limited

Authorized Signatory

HETERO LABS LIMITED (UNIT-III) HETERO N.Narasapuram

CIN : U24110AP1989PLC009723

HETERO LABS LIMITED (UNIT-IX)

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Place: Nakkapalli

TO WHOM IT MAY CONCERN

We are glad to inform you that **Mr.TIRIVEEDHI VENKATA YASWANTH (191FA17007)** from **VIGNAN FOUNDATION FOR SCIENCE, RESEARCH AND TECHNOLOGY** Vadlamudi, Guntur has successfully completed his internship at ***M/R. Hetero Labs Limited*** from **15-02-2023 to 15-05-2023.**

During his internship, he was exposed to the various activities in **"Environment Health and Safety Department"** under the guidance of **Mr.CH. RAJASEKHARA REDDY**, Head of the Department.

We found his very inquisitive and hard working. He was very much interested to learn the functions of our division and also willing to put his best efforts and get in to the depth of the subject to understand it better.

His association with us was very fruitful and we wish him all the best in his future endeavours.

For Hetero Labs Limited

Authorized Signatory

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CH.RAJASEKHARA REDDY
HOD-EHS

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During his internship, he was exposed to the various activities in **"Environment Health and Safety Department"** under the guidance of **Mr.CH. RAJASEKHARA REDDY**, Head of the Department.

We found his very inquisitive and hard working. He was very much interested to learn the functions of our division and also willing to put his best efforts and get in to the depth of the subject to understand it better.

His association with us was very fruitful and we wish him all the best in his future endeavours.

For Hetero Labs Limited

Authorized Signatory

15/05/2023

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This is to certify that Mr. **MANDELA KIRAN TEJA** student of **4st Year B.Tech (Petroleum Engineering)** at **VIGNAN'S UNIVERSITY, GUNTUR** has successfully completed Internship entitled **SUB SEA PRODUCTION SYSTEM** of ONGC, Eastern Offshore Asset, Kakinada during the Training, undergone from **01.03.2023** to **14.04.2023** During the training he took keen interest in the assigned work and his conduct is satisfactory.

We wish him all the success in future academic endeavors.

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We wish him all the success in future academic endeavors.

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S.S. RAO
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We wish him all the success in future academic endeavors.

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A

PROJECT REPORT

ON

"EXTRACTION OF ANTIOXIDENTS FROM MEDICAINAL PLANTS"

Submitted in the partial fulfilment of the requirements for the award of the degree

BACHELOR OF TECHNOLOGY

In

Division of Petroleum Engineering

VIGNAN'S
Foundation for Science, Technology & Research
(Deemed to be University)
-Estd. u/s 3 of UGC Act 1956

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DEPARTMENT OF CHEMICAL ENGINEERING

VIGNAN'S FOUNDATION FOR SCIENCE, TECHNOLOGY AND RESEARCH

GUNTUR, VADLAMUDI-522213

MAY-2023

CERTIFICATE

This is to certify that the project entitled as, "**EXTRACTION OF ANTIOXIDANTS FROM MEDICINAL PLANTS** " submitted to the Vignan's Foundation for Science, Technology and Research (Deemed to be) University, Guntur, Andhra Pradesh for the award of the degree of Bachelor of Technology is a bonafide record of the research work done by him/her under my supervision. The contents of this thesis, in full or in part, have not been submitted to any other institute or university for the award of any Degree.

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Project Guide

Dr.P.Ashok kumar

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UNDERTAKING

This is to declare that the project entitled "**EXTRACTION OF ANTIOXIDANTS FROM MEDICINAL PLANT** " is an original work doneby undersigned, in partial fulfillment of the requirements for the degree "Bachelor of Technology in Chemical Engineering .

All the analysis, design and system development have been accomplished by theundersigned.Moreover,thisprojecthasnotbeensubmittedtoany othercollegeoruniversity.

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ABSTRACT

The phytochemicals present in plants are responsible for preventing disease and promoting health have been studied extensively to establish their efficacy and to understand the underlying mechanism of their action. Such studies have included identification and isolation of the chemical components, establishment of their biological potency both by in vitro and in vivo studies in experimental animals and through epidemiological and clinical-case control studies in man. Study findings suggest that phytochemicals may reduce the risk of coronary heart disease by preventing the oxidation of low density lipoprotein (LDL) cholesterol, reducing the synthesis or absorption of cholesterol, normalizing blood pressure and clotting, and improving arterial elasticity. Phytochemicals may detoxify substances that cause cancer. They appear to neutralize free radicals, inhibit enzymes that activate carcinogens, and activate enzymes that detoxify carcinogens. They observed that, the Ocimum sanctum Linn has also been suggested to possess, anticancer, antidiabetic, anti-fertility, antifungal, antimicrobial, cardio protective, analgesic, antispasmodic and adaptogenic actions. Eugenol (1-hydroxy-2methoxy-4-allylbenzene) is the active constituents present in Ocimum sanctum Linn. Indian traditional shrub tulsi (Ocimum sanctums): They unique medicinal plant. They observed that, Ocimum sanctum heals many diseases chronically due to its chemical constituent and believes that it has anti- ageing, immunomodulatory property along with antimicrobial and anticancer property. Phytochemical analysis of aqueous extract of Ocimum sanctum. They observed that, the plant is known to possess antiseptic, analgesic, anti-inflammatory, antimicrobial, antistress, immunomodulatory, hypoglycemic, hypertensive and antioxidant properties. The dried powder of Tulsi (100g) was placed in the thimble of Soxhlet apparatus.500 ml of distilled water was used as a solvent.

Keywords: Ocimum sanctum Linn; Phytochemical analysis; Methanol extract; Antioxidant activity

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2 REQUIRED PLANT

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1) INTRODUCTION :

India is well known as an "Emporium of medicinal plants". It possesses about 8% of the estimated biodiversity of the world with around 12600 species and is one of the 12 mega biodiversity centers with 2 hot spots of biodiversity in the Western Ghats and North-eastern region. It's also rich in ethnic diversity, there are about 67.37 million tribal people belonging to 537 tribal groups living in different geographical locations with various subsistence patterns . These tribal groups living in diverse, rich areas possess a wealth of knowledge and skills on the utilization and conservation of food and medicinal plants . According to the World Health Organization (WHO), almost 65% of the world's population has incorporated the value of plants as a methodology of medicinal agents into their primary modality of health care. It is often noted that 25% of all drugs prescribed today come from plants . This estimate suggests that plant derived drugs make up a significant segment of natural product– based pharmaceuticals .

Plants have long been used by men for their basic needs especially Ocimum sanctum. In essence have been in practices of medicinal plants are deeply rooted in the society of indigenous community . Medicinal plants begins an important aspect of various traditional medicine systems, have been used therapeutically all around the world . Although the various systems of traditional medicine in the world, e.g., Ayurveda, Chinese traditional medicine, Unani, Tibetan Medicine , Amazonian or African Medicine, may be based on different theoretical and cultural models, they all integrate phytotherapy into their doctrine . According to World Health Organization (WHO) estimates, more than 80% of the people in developing countries depend on the traditional medicine for their countries depend on the traditional medicine for their primary health needs . It is generally estimated that

over 6000 traditional plants in India are used in folk and herbal medicine, representing about 75% of the medicinal needs of the 3rd world countries Aromatic plants possess odorous volatile substances which occur as essential oil, gumexudate, balsam and oleoresin in one or more parts, namely root, wood, bark, stem, foliage, flower and fruit. .The characteristic aroma is due to a variety of complex chemical compound. The essential oil is concomitant to fragrance or perfumes because these fragrance are oily in nature and they represent the essence or the active constituents of the plant . They are called volatile or ethereal oils as they evaporate when exposed to air at ordinary temperatures. Essential oils are highly concentrated, low volume, high value products. Application of essential oils in agriculture as antifeedants, repellents, botanical insecticides, natural herbicides and growth booster are still open to fascinating realms of research . Essential oils of only about 500 species are known in some detail at present. Of these about 50 species find use as commercial source of essential oils and aroma chemicals, through the number of those having regular and large scale utilization hardly exceeds two drones Phytochemicals (from the Greek word phyto, meaning plant) are biologically active, naturally occurring chemical compounds found in plants, which provide health benefits for humans further than those attributed to macronutrients and micronutrients. They protect plants from disease and damage and contribute to the plant's color, aroma and flavor. In general, the plant chemicals that protect plant cells from environmental hazards such as pollution, stress, drought, UV exposure and pathogenic attack are called as phytochemicals. Recently, it is clearly known that they have roles in the protection of human health, when their dietary intake is significant. More than 4,000 phytochemicals have been catalogued and are classified by protective function, physical characteristics and chemical characteristics and About 150 phytochemicals have been studied in detail. In wide-ranging dietary phytochemicals are found in fruits, vegetables,

legumes, whole grains, nuts, seeds, fungi, herbs and spices. Broccoli, cabbage, carrots, onions, garlic, whole wheat bread, tomatoes, grapes, cherries, strawberries, raspberries, beans, legumes, and soy foods are common sources. Phytochemicals accumulate in different parts of the plants, such as in the roots, stems, leaves, flowers, fruits or seeds. Many phytochemicals, particularly the pigment molecules, are often concentrated in the outer layers of the various plant tissues. Levels vary from plant to plant depending upon the variety, processing, cooking and growing condition. Phytochemicals are also available in supplementary forms, but evidence is lacking that they provide the same health benefits as dietary phytochemicals

2) REQUIRED PLANTS:-

2.1 TULASI

- Its scientific name of Tulsi is Ocimumtenuiflorum . It is grown in warm and tropical areas.Tulasi grows fragrantly, erect and with several branches.
- Types of tulasi

    1.Krishna Tulsi (Brown color)

    2. Rama Tulsi (Green color)

- Tulsi has a unique combination of actions that include: Antimicrobial (anti -oxidant, anti -cataract, anti -inflammatory, anti -pyretic, anti -allergic, anti - thyroid, anti -fertility, anti -ulcer properties
- Tulsi is enriched with various phytochemicals. The leaves of the plant contains volatile oil 71% eugenol and 20 % methyl eugenol.
- Tulsi contain antioxidants like beta carotene that help in preventing cell damage

2.2 BUTTERFLY PEA

Clitoriaternatea, commonly known as Butterfly pea

- This plant is native to equatorial Asia, including locations in south Asia and southeast Asia but has also been introduced to Africa, Australia and the Americas.

Different colors :-

1. White
2. Pink
3. Blue

- It contains antioxidants like (kaempferol, p-coumaric acid, delphinidin-3,5-glucoside).
- It's rich in antioxidants and often used as an herbal tea and natural dye.

3) EXTRACTION METHODOLOGY :-

Basically three types of extraction methods :-

- Extraction method
- Soaking method
- Ultrasonication

Extraction method :

Extracted using heat by Soxhlet apparatus and also soaking method

Soaking method:

Soaking (Tulsi ,butterfly pea)powder in solvent without using heat for long period

Ultrasonication

By ultrasound cavities, which leads to increase temperature and pressure which enhance extraction by breaking the cell walls .

3.1 Experimental procedure:

Using method :- extraction method & ultrasonic associated extraction .

Types : Tulsi & Butterfly pea

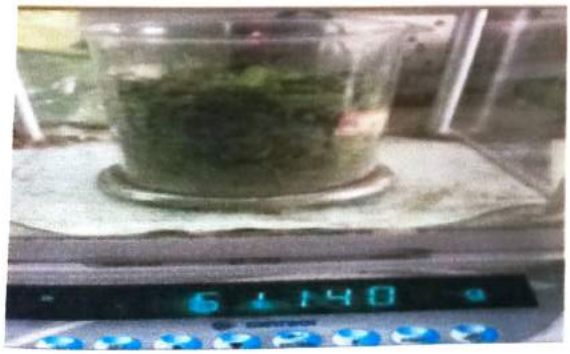
Portion requried : Leaves of Tulsii

Flower petals of butterfly pea

Apparatus : Soxhlet apparatus (condenser , distillation flask , sample holder , heater, round bottomed flask), ultrasonic cell crusher noise isolating chamber

Solvent used : Methanol , n hexane

1. First step in the experimental procedure is cleaning and it will done by collect fresh(Tulsi leaves OR Butterfly pea flowers) and wash them thoroughly with distilled water to remove any dirt.



2.Second step is drying the cleaned material and dry the leaves in a well-ventilated

area or in an oven at low temperature (below 50°C) until they are completely dry

3. Third step is size reduction grind the dried leaves using a mortar and pestle to obtain a fine powder.

3.2 By using soaking method :-

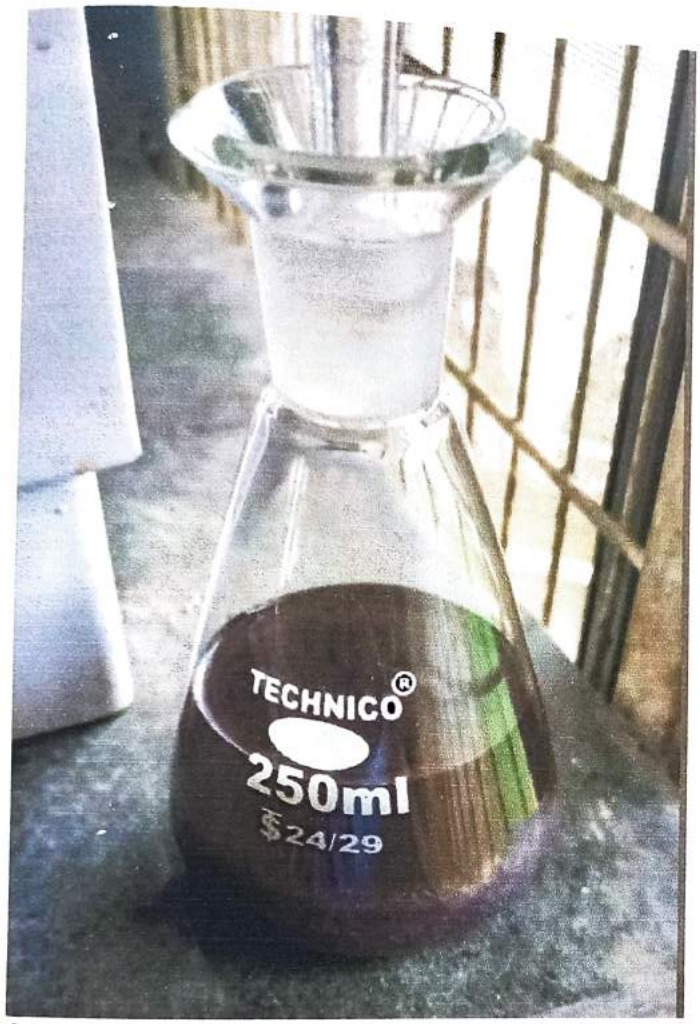
• Weight out about 25 g of the powdered Tulsi leaves OR Butterfly pea leaves and place them in a beaker or flask.

• Add 50 mL of ethanol or methanol to the beaker or flask and stir the mixture thoroughly.

• Cover the beaker or flask with aluminum foil to prevent evaporation and place it in a dark place for 24 hours to allow for the extraction of antioxidants.

• After 24 hours, filter the mixture through filter paper to obtain a clear extract.

• Evaporate the solvent from the extract to form the product



Soaking method

3.3 solvent extraction method :-

• 25 grams of powder should be wrapped in the Whatman filter paper. Insert the wrapped material in the extraction chamber .

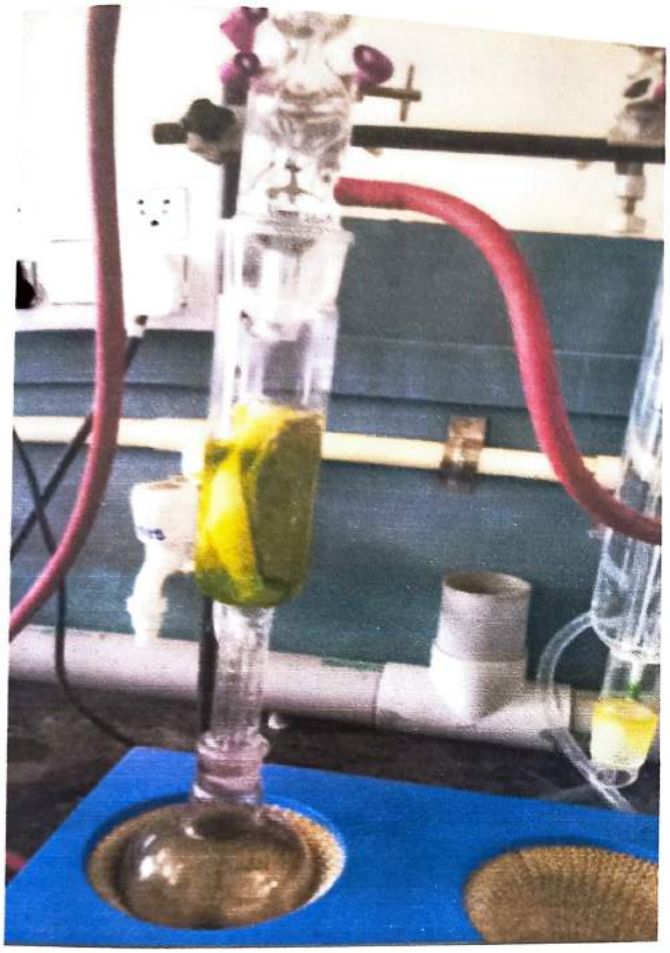
• Add appropriate solvent to cover it in the extraction chamber. Assemble the distillation apparatus, ensuring that the condenser is securely attached to the distillation flask.

• Make sure to run the condenser with running cool water to keep it cool during the distillation process. Heat the distillation flask slowly and steadily using a heating source.

• Allow the distillation process to continue until enough antioxidant-rich solvent has been collected in the receiving flask.

• Turn off the heating source and allow the apparatus to cool. Once cooled,

carefully remove the receiving flask and evaporate the excess solvent to obtain the product



Efficiency of soxhlet extraction for both plants :-

Antioxidants from Tulsi by Soxhlet extraction:

$$\text{Efficiency} = (3/25)*100 = 12\%$$

Antioxidants from butterfly pea by Soxhlet extraction:

$$\text{efficiency} = (4/25)*100 = 16\%$$

4) Identification of anti oxidants :

4.1 DPPH METHOD

There are several methods to identify the antioxidants, in those, one method is "DPPH"

- DPPH stands for 2,2-diphenyl-1-picrylhydrazyl, which is a chemical compound commonly used as a reagent to measure the antioxidant activity of substances. It is a stable free radical that reacts with antioxidants by giving up an electron, resulting in the formation of a stable diamagnetic molecule.
- When DPPH comes into contact with an antioxidant, such as a phenolic compound, the antioxidant donates an electron or hydrogen atom to the DPPH radical, causing the radical to be reduced and change color from purple to yellow.
- The change in color can be measured spectrophotometrically, to find the concentration of the antioxidants
- This method is used to determine the antioxidant potential of various samples, including food extracts, plant extracts, and pharmaceutical compounds.

Preparation of "DPPH"

- It is present in the solid form , it is prepared by using solvent methanol
- The standard solution for preparing of the DPPH is by adding 24mg in 100l of methanol.
- DPPH (violet) +antioxidant pale yellow/colorless

4.2 Photometric analysis :

This analysis is done to know the concentration of the antioxidants present in the product sample

• Photometric analysis is a technique used in various scientific fields to measure the intensity of light emitted, absorbed, or transmitted by a sample. It is widely used to determine the concentration of substances or to study various optical phenomena.

• The principal of photometric analysis is based on the measurement of light intensity using a photometer or spectrophotometer.

Procedure:

• Take 1ml of product sample and makeup with the appropriate solvent (methanol) to make into 10ml solution in a volumetric flask , it is considered as 1st main solution

• Now Take approximately 1ml from the 1st main solution and makeup to 10ml in volumetric flask .

• By using as a solvent (methanol) as a reference, find out the absorbance value by setting an appropriate wavelength (517 nm)

5) RESULTS :

5.1 YEILD :-

For tulasi :

SNO	Method	Yield (ML)	Efficiency
1	Soaking method	4 ml	16%
2	Soxhlet extraction	3 ml	12%
3	Ultrasonic associated extract	3 ml	12%

For butterfly pea :

SNO	METHOD	YIELD (ML)	Efficiency
1	Soaking method	5 ml	20%
2	Soxhlet extraction	4 ml	24%
3	Ultrasonic associated extract	6 ml	16%

5.2)CONCENTRATION OF SUBSTANCES :

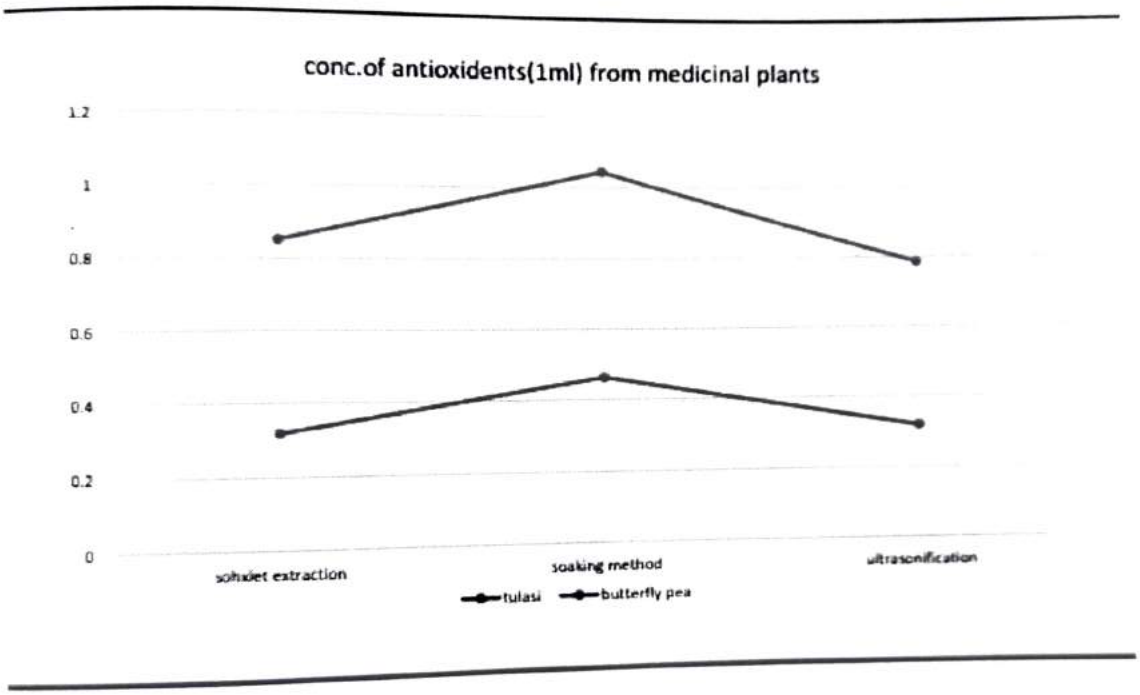
	Soxhlet extraction		Soaking		Ultrasonification	
	Abs	concentration	Abs	concentration	Abs	concentration
Tulasi	0.5704	0.8514	0.8419	1.0564	0.4086	0.7865
Butterfly pea	0.1570	0.3195	0.1892	0.4646	0.1564	0.3187

Sample calculation :

For Abs = 0.1564 ,

Concentration = $\left(\frac{0.1564}{715}\right) * 10 * 100 = 0.3187$ mg/ml

Graph for concentration :



6) APPLICATIONS OF ANTIOXIDANTS :-

Antioxidant supplements are compounds obtained either by extraction from natural foods or by chemical synthesis. Of course, they do not have the same composition as natural antioxidants in foods. Therefore, opinions are divided over whether or not antioxidant supplements offer the same health benefits as antioxidants in foods. So some of the benefits are below :-

6.1) applications of tulasi :

Skin health: They can help to protect the skin from damage caused by UV radiation, pollution, and other environmental stressors.

Immune system support: They can help to boost the immune system by neutralizing free radicals and reducing inflammation.

Respiratory health: They show anti-inflammatory properties that can help to reduce symptoms of respiratory conditions like asthma and bronchitis.

Digestive health: They reduce inflammation in the gut and protect against damage caused by free radicals. This can help to improve digestion and reduce symptoms of digestive disorders like ulcerative colitis.

Cardiovascular health: They can help to protect against oxidative stress, which can damage the cells lining blood vessels and increase the risk of heart disease.

6.2) applications of butterfly pea

Anti-aging benefits: They help to protect the skin from damage caused by free radicals, which can contribute to aging.

Brain health: They have been shown to have neuroprotective effects, which may help to reduce the risk of cognitive decline and improve memory and learning abilities

Stress and anxiety: They contains anxiolytic properties, which can help to reduce stress and anxiety levels.

Eye health: They may help to protect the eyes from damage caused by free radicals, which can contribute to the development of eye diseases such as cataracts and

macular degeneration.

Diabetes management: They have been shown to help regulate blood sugar levels, making it a potential natural treatment for diabetes.

Cardiovascular health: They may help to reduce inflammation and improve blood flow, which can have a positive effect on cardiovascular health.

7) CONCLUSION :

Tulasi and butterfly pea are the medicinal plants what we choose for the experiment. Tulsi is also known as "the elixir of life" since it promotes longevity. Different parts of plant are used in Ayurveda and Siddha Systems of Medicine for prevention and cure of many illnesses and everyday ailments like common cold, headache, cough, flu, earache, fever, colic pain, sore throat, bronchitis, asthma, hepatic diseases, malaria fever, as an antidote for snake bite and scorpion sting, flatulence, migraine headaches, fatigue, skin diseases, wound, insomnia, arthritis, digestive disorders, night blindness, diarrhea and influenza. This review will definitely help for the researchers as well as clinicians dealing with Ocimum sanctum to know its proper usage as this herb is seemed to be highly valuable, possessing many pharmacological/ medicinal properties. In conclusion, the present study phytochemical screening, total phenolic contents, total flavonoids contents and antioxidant properties have been done using Ocimum sanctum plant of methanol extracts. Among the two extract, the methanol was found to be the best extract the antioxidant.

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A

PROJECT REPORT

ON

EXTRACTION OF CAFFEINE FROM COFFEE SEEDS AND ITS APLLICATIONS

Submitted in the partial fulfillment of the requirements for the award of the degree

BACHELOR OF TECHNOLOGY

In

Chemical Engineering

VIGNAN'S
Foundation for Science, Technology & Research
(Deemed to be UNIVERSITY)
-Estd. u/s 3 of UGC Act 1956

(ACCREDITED BY NAAC WITH 'A'GRADE)

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MAY-2023

ACKNOWLEDGEMENT

I would like to thank my guide P. VIJETHA, Vignan's foundation for science technology and research, Vadlamudi. The interaction with her was really, fruitful and helped me in compiling this project report. My deepest thanks to Dr. M. Ramesh Naidu Head of Chemical Department VFSTR, Vadlamudi for guiding me and extending all possible help to me and always inspiring me to do my work on this project report with sincerity. Also, I would like to thank whose trust and enthusiasm was constant motivation during ongoing work. Finally, I would like to thankful to all staff members of Department of CHEMICAL Engineering, Vignan University, Vadlamudi and who directly or indirectly helped me in the completion of this project report.

UNDERTAKING

This is to declare that the project entitled "EXTRACTION OF CAFFEINE FROM COFFEE SEEDS AND ITS APPLICATIONS" is an original work done by undersigned, in partial fulfillment of the requirements for the degree "Bachelor of Technology in Chemical Engineering.

All the analysis, design and system development have been accomplished by the undersigned. Moreover, this project has not been submitted to any other college or university.

V. CHAITANYA

A.V. SASI

S. SAI

G. HARI

CERTIFICATE

This is to certify that the project entitled as, "EXTRATION OF CAFFEINE FROM COFFEE SEEDS AND ITS APPLICATIONS" submitted by to the Vignan's Foundation for Science, Technology and Research (Deemed to be) University, Guntur, Andhra Pradesh for the award of the degree of Bachelor of Technology is a Bonafide record of the research work done by him/her under my supervision. The contents of this thesis, in full or in part, have not been submitted to any other institute or university for the award of any Degree.

Project Guide

Dr. M. Ramesh Naidu
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ABSTRACT

This paper focuses upon extraction of a natural product, caffeine, from Coffee. It has been chosen since the starting ingredients are relatively easy to come by, and we will still find a reasonable level of challenge. Caffeine is a natural product found in coffee and tea. Efficient extraction of caffeine from Coffee relies heavily on the properties of caffeine & other components present in Coffee. This work will focus upon procedure (Batch) for Caffeine extraction and their working principles, its design aspect, various analytical methods for its separation/detection. It is to be noted that its scale up can be done according to industrial demand for its usage for manufacturing other products. By using Caffeine as raw material, we will prepare an Anacin – a pharmaceutical product which has several medicinal uses.

Keywords: Caffeine, Anacin, pharmaceutical.

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INTRODUCTION:

Coffee is one of the most consumed beverages in the world and, after petroleum, is the second traded product worldwide. In Western countries, a significant portion of the daily beverage is constituted by the different varieties of coffee. Coffee grows mainly in Africa and South America and nowadays, among many known species, only two varieties are successfully used in commercial cultivation: Coffee arabica. Arabica and Coffea canephore var. Robusta. Arabica is mainly cultivated in Brazil, Colombia, Costa Rica, Guatemala and India, whereas Robusta is mainly cultivated in Vietnam, Ivory Coast, Guatemala and India.

Coffee is second only to water as the most widely consumed beverage in the US and Europe and it is the main source of caffeine in daily consumption in adults, even if caffeine is contained also in tea, chocolate, and soft drinks. The two coffee bean varieties of worldwide importance differ considerably in price, quality and consumers' acceptance. Indeed, Arabica is preferable for the aroma effect and Robusta for taste and body; for these reasons, a good flavor is commonly obtained by blending the two varieties. Moreover, caffeine content of green coffee beans varies according to the species: Arabica beans contain about 1.0–1.2%, whereas the caffeine content in Robusta beans is about 1.6–2.5%.

Even if a moderate consumption of caffeine can have beneficial effects on adults' behavior, numerous studies, in recent years, reported the effect of caffeine consumption on cardiovascular diseases and on central nervous system, leading to an increasing consumption of decaffeinated coffee. Moreover, caffeine recovery is important, because it can be used in cola-type drinks or in combination with other active principles in the pharmaceutical field (in the treatment of headache and neuralgia), or as an ingredient in the cosmetic field (in the treatment of cellulitis and localized excess fat).

Four main methods are used worldwide for the decaffeination: in the solvent based methods, organic solvents (mainly methylene chloride and ethyl acetate) are employed, whereas in the non-solvent based methods, water or supercritical carbon dioxide (scCO2) are used for the

caffeine extraction. In all cases, coffee is decaffeinated in its green state; i.e., before the roasting operation. Until the mid-1970s, methylene chloride was considered the best solvent for extraction of caffeine with satisfactory results. However, subsequently, doubts arose about its risk to humans, due to the solvent high toxicity. Although the residual amount of methylene chloride in decaffeinated coffee was well below the limit of 10 ppm, established by the Food and Drug Administration, the suspected carcinogenicity of this solvent led to the choice of a less toxic solvent, such as ethyl acetate, a natural component detected in coffee aroma and found to occur naturally in different fruits. The use of ethyl acetate has two considerable drawbacks: it is highly flammable and has a fruity aroma. It must be handled carefully, increasing production costs, and it tends to pass on its characteristic aroma to the coffee, slightly altering the flavor. The decaffeination using water was developed in Switzerland, and constitutes a green process with respect to the product. Unfortunately, water is not a particularly selective solvent and, therefore, not only caffeine but also various flavors were removed from coffee beans using this method. As a result, a less flavorful brew with respect to other methods was obtained. The most selective process for removing just caffeine and not the other flavor precursors from coffee is based on the use of scCO2. This process was successfully developed on an industrial scale in the 1970s, based on two patents developed by Zosel: in the first one, the process was presented for the recovery of caffeine, whereas, in the second one, a detailed description aimed at obtaining decaffeinated coffee was proposed.

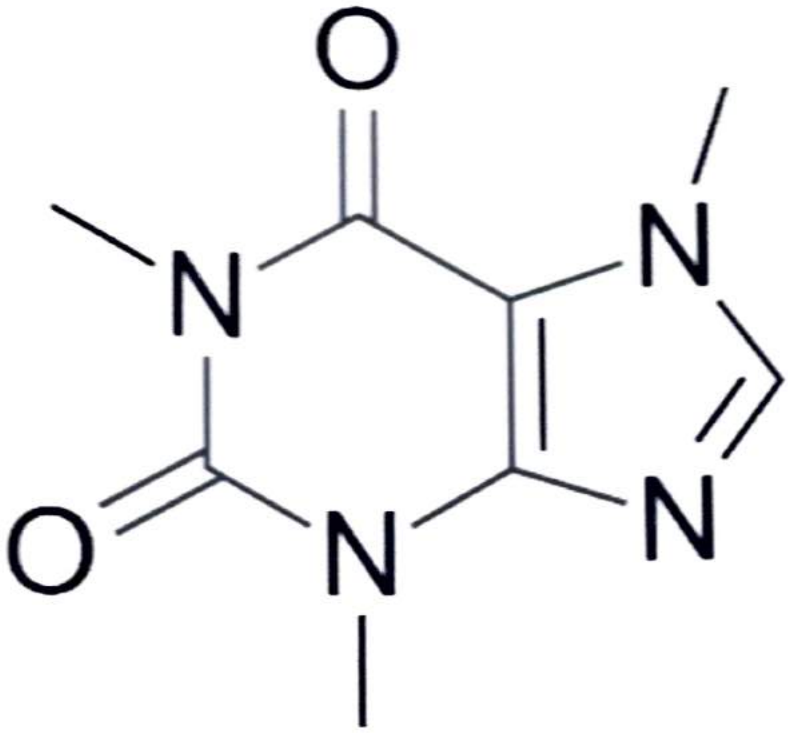
Supercritical fluids (SCFs) based techniques were proposed as an alternative to conventional processes, thanks to their specific characteristics, mainly, solvent power and liquid-like densities with gas-like transport properties that can be tuned by varying pressure and temperature. They were successfully applied in several fields, such as, for example, micronization, porous structures formation, adsorption. Among the different scCO2 based processes, one of the most studied one was supercritical fluids extraction (SFE), for the possibility of continuously modulating the solvent power/selectivity; this process was frequently used for the extraction of essential oils. SFE was used also for the extraction of caffeine from natural sources, such as coffee husks, coffee beans and tea leaves.

1. literature review

1.1 coffee

coffee the known evidence of coffee, knowledge and usage, dates to the 15th century. Ethiopian and Yemen cultures have cited the use of coffee in the 14th and 15th centuries. The beans obtained from the coffee shrub were used by the people to brew a concoction that energized them and helped them improve their concentration. The drink later reached Europe in the 17th century travelling through Cairo, the Middle East and Constantinople, where it became an instant hit among the people for its exquisite taste and aroma. Coffee comes from plants belonging to the genus Coffea. The two commercially important species are Arabica and Robusta out of the nearly 100 species that are estimated to exist. The optimum conditions required for these trees grow best include rich soil, mild temperatures, frequent rain and shaded sun. The coffee beans go through a series of steps between when they are planted and brewed to make a cup of coffee. These steps typically involve planting the shrubs, harvesting the cherries, processing them, drying the beans, milling them, roasting the coffee beans, grinding them and finally brewing the coffee. The caffeine in coffee has a lot of benefits when consumed by healthy adults within the daily-intake limit prescribed, which is generally about 400 mg, though it varies from person-to-person. The benefits include boosting metabolic rate, improving physical performance, providing essential nutrients like Riboflavin, Manganese, Magnesium, Potassium, Niacin, etc. Coffee has also been seen to reduce the risk of type II diabetes, Alzheimer's disease, Parkinson's disease and dementia among drinkers. Coffee also has a lot of antioxidants, thus reducing the risk of cancer. The caffeine content in coffee depends upon the specific type of bean being used.

1.2 Caffeine



Caffeine is an alkaloid (1,3,7-trimethylxanthine) and occurs naturally in tea leaves and coffee beans. Cocoa beans, from which chocolates are made, also contain a caffeine-like compound. It is the world's most consumed psychoactive drug. It is also artificially added to many types of sodas and energy drinks. This is because of how caffeine stimulates the Central Nervous System and keeps people alert and prevents tiredness. When consumed, 99% caffeine is absorbed into the bloodstream. It is lipophilic and crosses all biological barriers and is supplied to all body tissues. It also crosses the blood-brain barrier and the placenta.[8] It has been speculated that caffeine causes risk of heart diseases among its takers but there is no conclusive evidence of this. However, animal studies have indicated that it might be a weak teratogen (an agent that causes birth defects in an embryo or fetus), so pregnant women are advised to limit their intake of caffeinated beverages.

1.3. Effects of caffeine on body

Caffeine is probably the most frequently ingested pharmacologically active substance in the

world. When consumed at regular intervals, the body develops tolerance for caffeine and this depends upon the amount of consumed by individuals. Once accustomed to a particular amount, a cut down in the intake leads to caffeine withdrawal syndrome. The symptoms include headache, nausea, anxiety, restlessness and the intense urge to drink coffee. It interacts with various systems of the body and has the following results:

Central Nervous System: There is increase in vigilance and arousal when coffee is consumed. •Ingesting caffeine before sleep has shown increase in sleep latency, reduction in total sleeping time and an overall poorer quality of sleep.

Cardiovascular Effects: In hyper-tension prone drinkers, acute intake of caffeine has shown an increase in blood pressure. However, long term consumption has shown the development of tolerance and thus, no effect on blood pressure in the longer run.

Respiratory Effects: A primary increase in the respiratory rate can be observed and this is directly proportional to the plasma-caffeine level. •In patients with asthma, caffeine acts as a bronchodilator.

2. Material & Methods

2.1 Required seeds

Coffee is one of the most traded commodities, and is one of the most popular drinks in the world due to its unique flavor and characteristics. Two major species of coffee grown commercially are Robusta and Arabica. Robusta seeds were chosen because it has more content of caffeine 2kgs of Robusta coffee beans were bought an were performed required experimentation

2.2 Methods of caffeine extraction

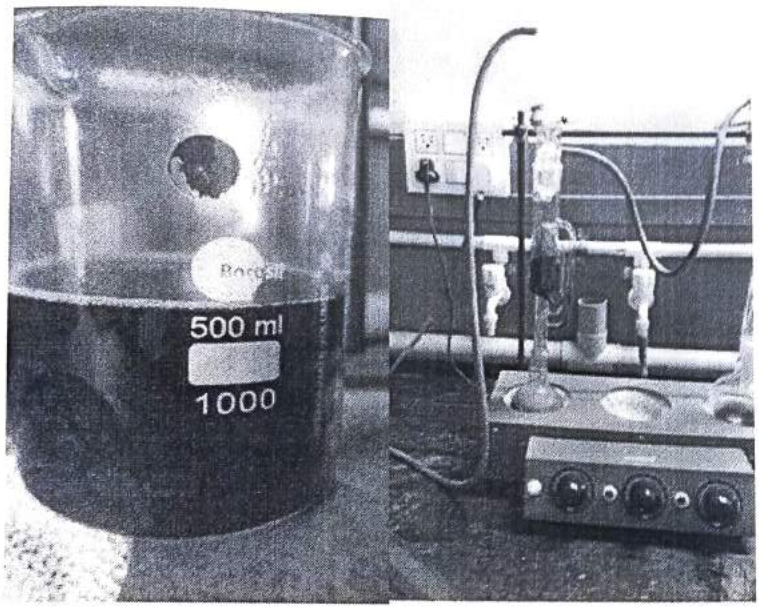
solvent	Solubility in water	Boiling point, C	Density (g/ml)
Ethyl acetate	Fairly soluble	76	0.902
Acetone	Fairly soluble	56	0.791

Sl.no	Decaffeination method	description
1	**water**	Non- toxic, complex process, remove little flavoring components
2	**Ethyl acetate**	Removes caffeine and little flavor compounds. Mildly toxic, removes some flavoring compounds.
3	**Dichloromethane**	Removes caffeine and little flavor compounds. Highly toxic, removes some flavoring compounds
4	**Supercritical CO2**	Selectively removes caffeine and very little flavor compounds, expensive

2.3 Process of Extraction of caffeine

2.3.1 Soxhlet extraction

1. Take the coffee beans and crush as small as possible
2. After crushing collect the powder form of coffee
3. Put the required amount of coffee powder into Soxhlet apparatus
4. Add the required amount of solvent, either Acetone or Ethyl acetate into to the Soxhlet apparatus.
5. Start heater for heating purpose and maintain the temperature
6. After prescribe time, collect the mixture and filtrate it by using filter paper for separating the solid content.

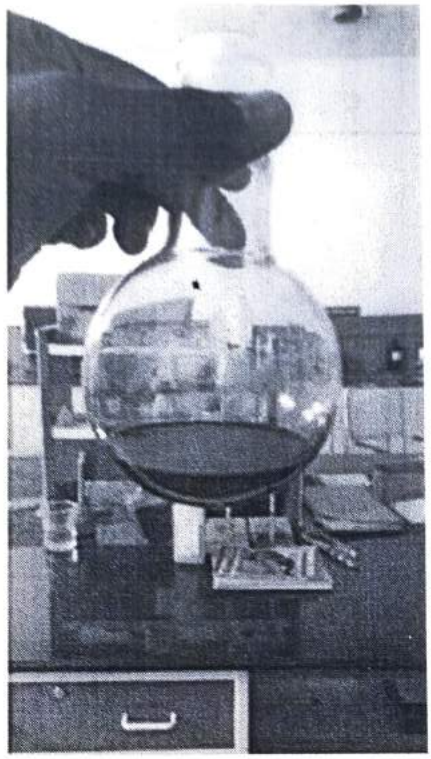


7. Put the liquid mixture into simple distillation apparatus for recover the solvent, so collect sample
8. Finally, samples are carried for analysis in GC Capillary column
9. Caffeine can further be gotten into powder form by putting the solution into the sublimator assembly as shown below

The equipment used for the caffeine extraction is sublimator and separating funnel

2.3.2 Powder based extraction

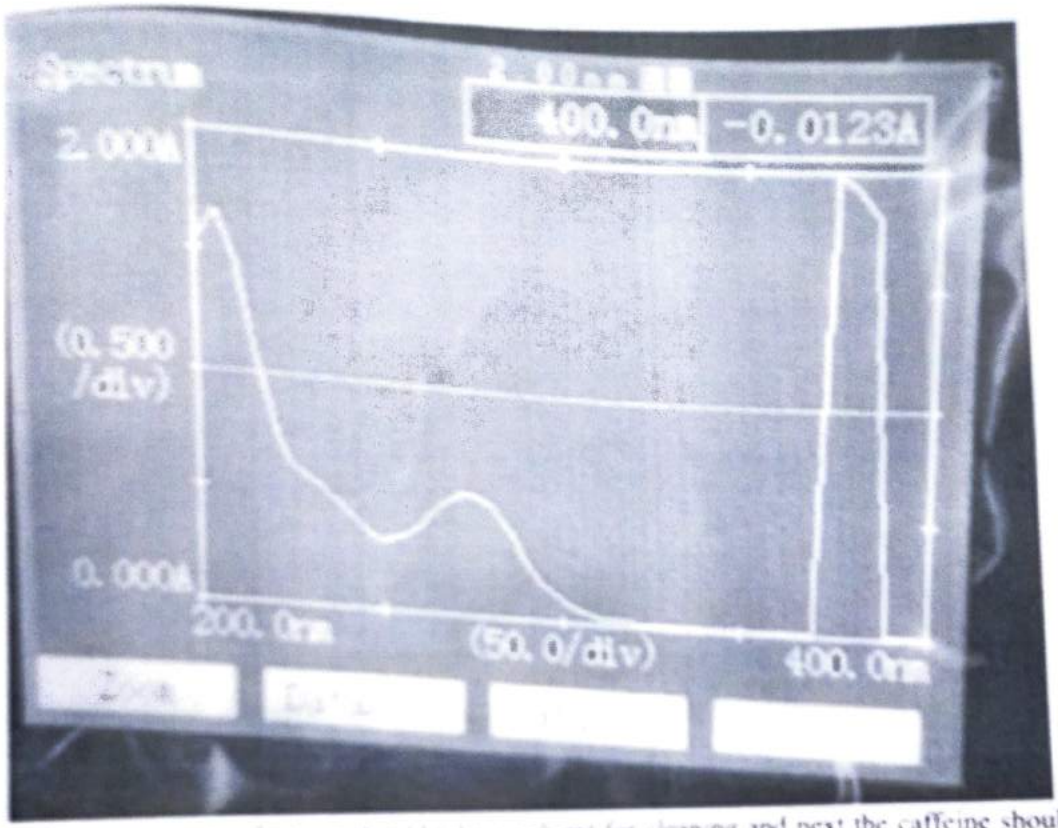
1. First take coffee beans of weight 200gms and make a fine powder of coffee beans by using mixer.
2. By using sieve analysis separate the coarse particles from the mixture
3. Now weigh 200gms of powder and take it into the 500ml beaker.
4. Add sodium carbonate of 60gms to the mixture this compound is added to the mixture because to increase the solubility of the caffeine.



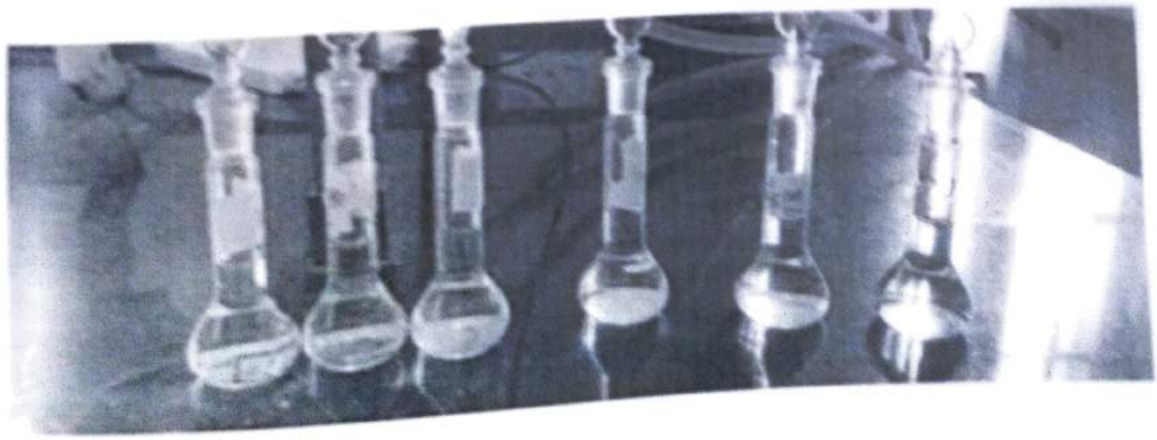
5. Water content of 400ml was added in a ratio of (1:2) ratio and was mixed thoroughly and was let to evaporation by application of heat (80C)
6. The liquid mixture was allowed to come until the 200ml mark so that liquid gets highly concentrated with caffeine.
7. Next, we take mixture into the separating funnel and add the solvent and is left for 12 hrs. and a layer gets separated by removing the bottom layer that is mixture of caffeine and evaporation is done to extract caffeine

3. IDENTIFICATION

In this the extracted caffeine was sent to the spectrometer UV analysis from this the wave length was obtained 274nm. When compared to the literature survey we found that caffeine should be of 260 to 275nm.

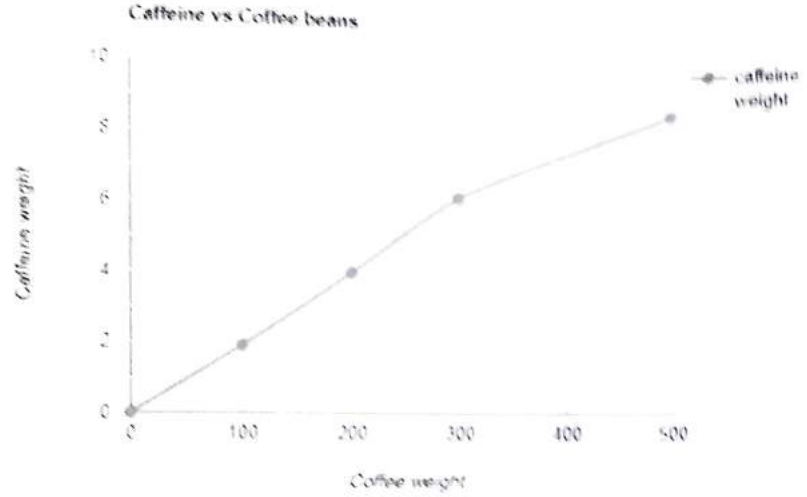


In this spectrometer first we should take a solvent for cleaning and next the caffeine should be dissolved into in the 100ml solution with a weight of 100mg so that the liquid contains 1mg/1ml. Next the standard solution was prepared with dissolving the 10 ml of this solution to the 100ml distilled water and then we prepare work solution which comprises of 0.01mg/1ml which is used for the UV spectrometer this test helps us to know about the compounds is correct or wrong product is obtained.

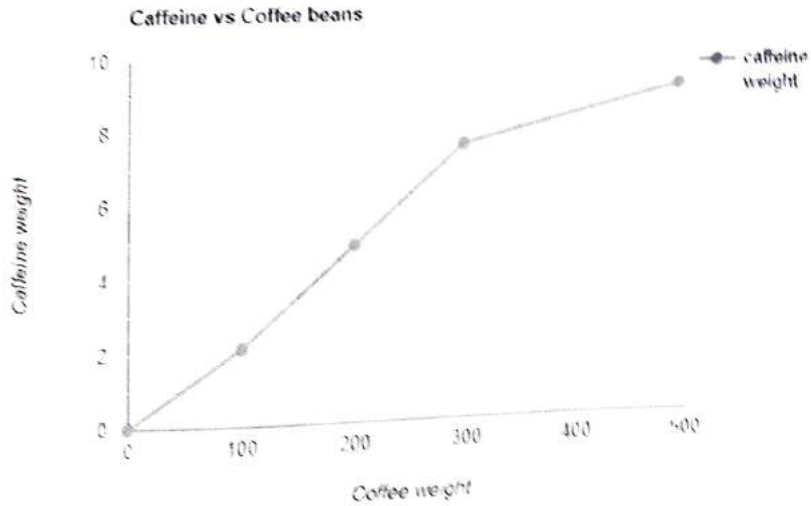


4. Yield analysis

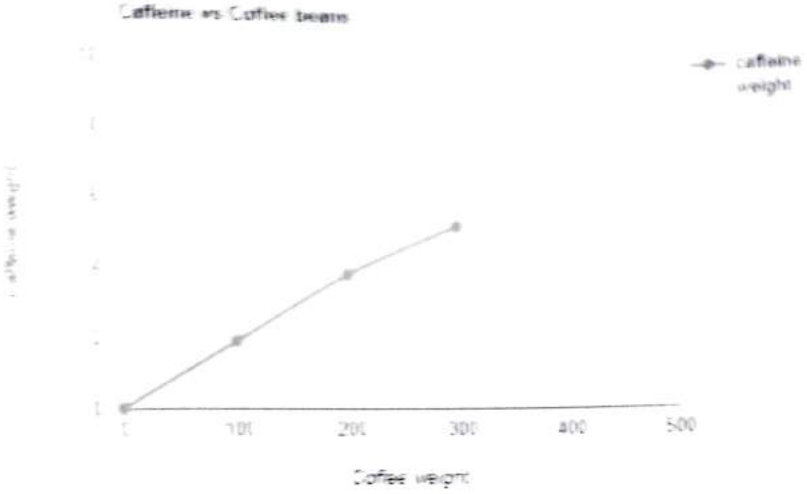
When caffeine was extracted yield ratio should also be checked for better experimentation the following diagrams explains the weight of coffee powder to the amount caffeine was extracted



The above graph explains about the yield obtained for the coffee powder to the solvent acetone (gm vs gm)



The above graph explains about the yield obtained for the coffee powder to the solvent ethyl acetate (gm vs gm)



The above graph explains about the yield obtained for the coffee powder to the solvent water (gm vs gm)

5. Drug powder preparation

1. Initial preparation of starch mucilage (5% v/v):

In 10 mL of water, disperse 1 g of starch powder in a beaker. Take another 10 mL of water in another beaker and keep for heating. When the temperature is raised to 60 – 65oC, add the starch dispersion from the first beaker to the water under heating. Mix it continuously until the formation of a sticky viscous solution. Then remove from heating and allow to cool to room temperature

2. Preparation of tablets:

Pass the all the excipients through sieve #80. According to the formula given in the table, weigh specified quantities of Aspirin and other excipients. Transfer Aspirin, lactose and starch into a clean and dry mortar and mix thoroughly. Now add starch mucilage drop-wise to the powder and mix. Continue the addition and starch mucilage and mixing until

granulation dough mass. Then take a sieve and dry sieve no 12 and pass small amounts of the dough mass through the sieve to obtain wet granules. Keep these granules in a hot air oven at 60°c until dried sufficiently. After drying remove the granules from the hot air oven and check the weight of dried granules. Add proportional quantities of lubricant and other extra granular excipients to the obtained dry granules in a polythene bag and mix for 5min. Compress these lubricated granules in a tablet compression machine to obtain tablets of 800mg each.

S no	Ingredient	Quantity per 1 tablet (mg)	Quantity per 20 tablets
1	Aspirin	400	2 gm
2	Caffeine	32	640 mg
3	Starch mucilage	Q.S	

6. Gel preparation:

6.1 definition:

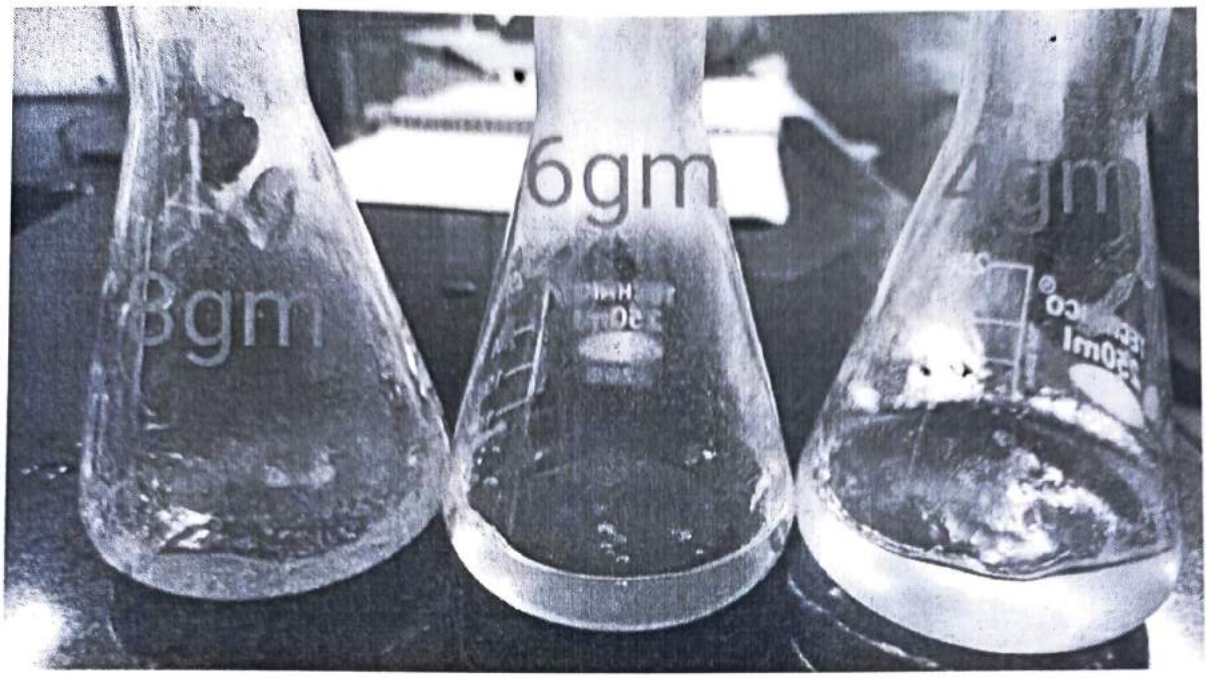
A gel is a solid or semisolid system of at least two constituents, consisting of a condensed mass enclosing and interpenetrated by a liquid.

6.2 Preparation of gels

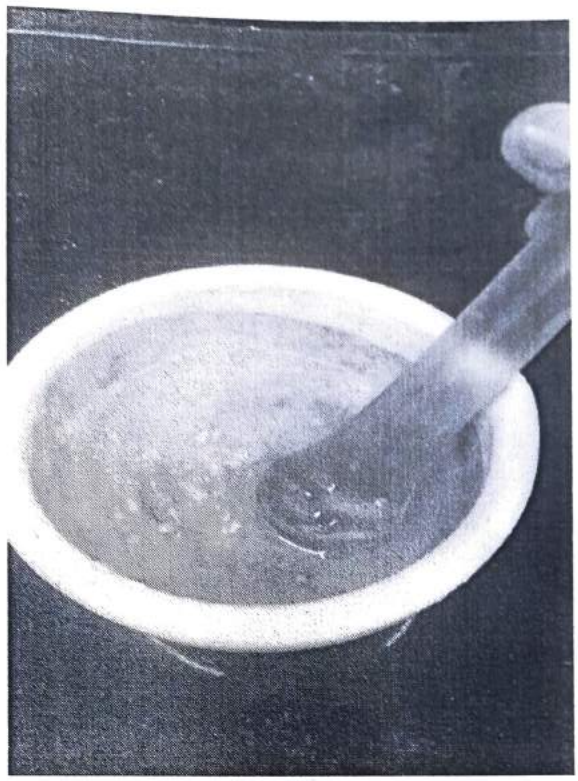
The gel can be prepared in 3 different types of method they are

1. Cold method
2. Fusion method
3. Dispersion method

In this case dispersion method was chosen since it was the most effective and efficient it includes the stirring the gelling agent at 1200rpm for 30 minutes.



The gel was prepared by using mortar and pestle and using the chemicals carboxy methyl cellouse which is a semi synthetic polymer was used as gelling agent. In this process three samples were taken which are 4, 6, 8 grams respectively into conical flask. Here the three samples were taken to adjust the viscosity according the patient satisfaction. At first the extracted caffeine was taken which is 5gm in each beaker was dissolved in the distilled water and was added with CMC and was kept in shaker 30 minutes at 1200 rpm.



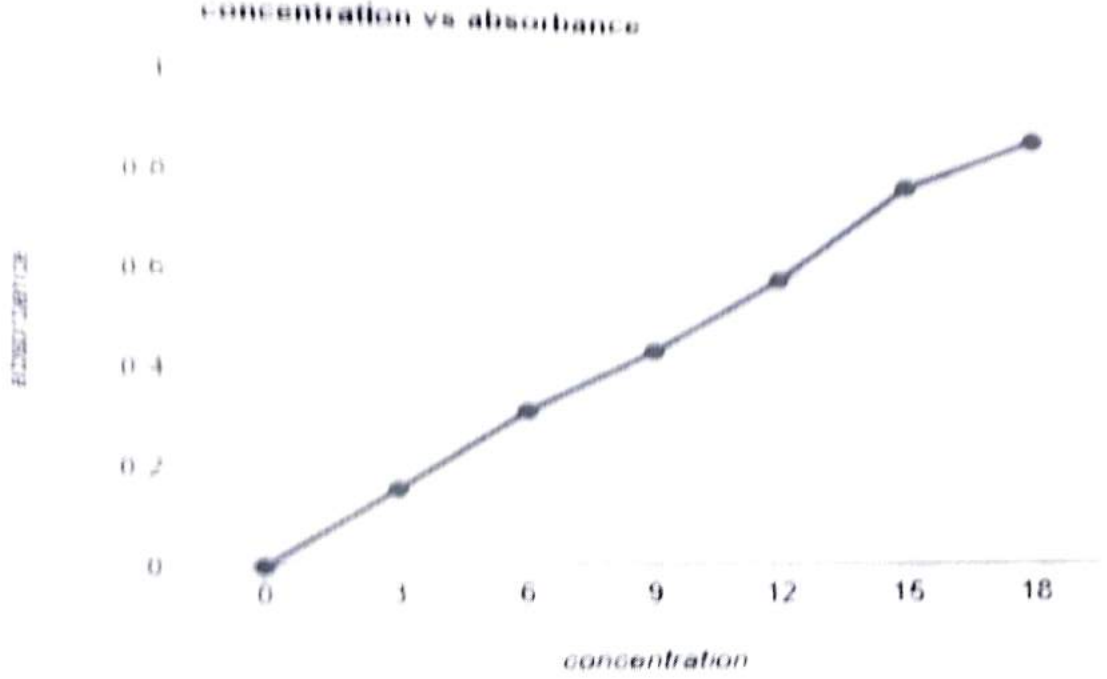
7. Results and conclusion

The total caffeine obtained is mentioned in the following table

Sl.no	Solvent used	Weight of coffee beans used (kg)	Weight of caffeine extracted (gm)
1	Acetone	1.1	20.7
2	Ethyl acetate	1.1	22
3	water	0.6	10.5

As we can see, the highest amount caffeine can be extracted by using ethyl acetate.

The absorbance test was conducted to know the light absorbance so that we can know the penetration of the drug molecule. The results are as follows


concentration vs absorbance
1
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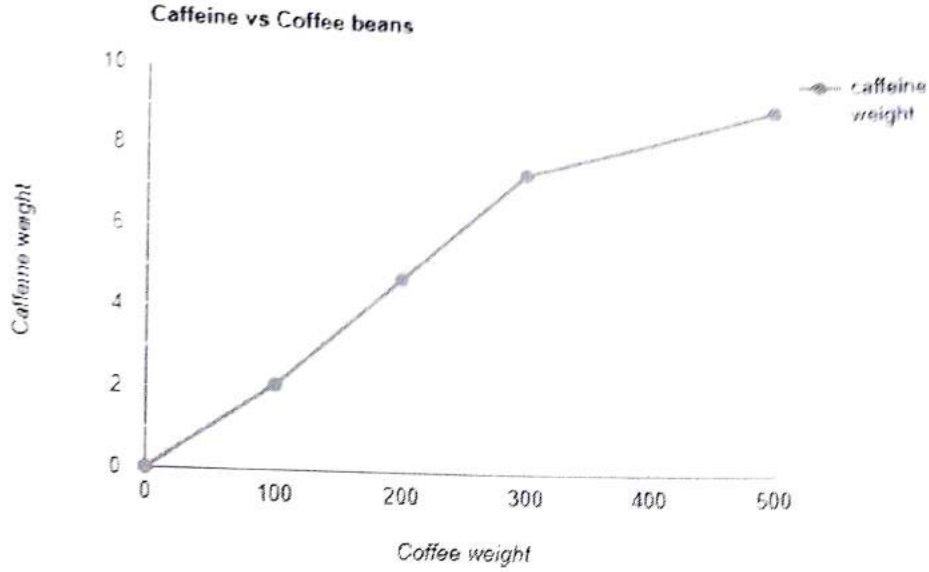
0.4
0.2
0
0
3
6
9
12
15
18
concentration



The above graph explains the amount caffeine extracted to the amount of coffee beans used Units of (gm vs gm).

Conclusion:

Caffeine is a natural stimulant found in coffee, tea, chocolate, and other food and drinks. Caffeine is defined as a drug because it stimulates the central nervous system. It affects kids and adults similarly and, at lower levels, can make people feel more alert and energetic. Foods and drinks with caffeine are everywhere, but it's wise to keep caffeine consumption to a minimum, especially in younger kids. From the result of the performed experiment, it can be concluded that coffee and caffeine lead to an increase in the natural acidity of the stomach. This is because caffeine has a direct effect on the gastric acid and pepsin secretion. This, in the long run, can lead to peptic ulcers in regular drinkers. This effect is predominant when coffee is ingested into an empty stomach and leads to acute heartburn

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A

PROJECT REPORT

ON

"BIODIESEL PRODUCTION FROM COTTON SEED OIL"

Submitted in the partial fulfilment of the requirements for the award of the degree

BACHELOR OF TECHNOLOGY

In

Division of Petroleum Engineering

Submitted by :-

DESIDERIO BOI ELIA MONSTAFA	201FA17008
MOHAMED FAIZAN RAZA	201FA17006
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UNDER THE GUIDANCE:

Mr. ABHISHEK KUMAR

DEPARTMENT OF CHEMICAL ENGINEERING

VIGNAN'S FOUNDATION FOR SCIENCE, TECHNOLOGY AND RESEARCH

GUNTUR, VADLAMUDI-522213

MAY-2023

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I would like to thank my mentor **MR ABHISHEK** Vignan's foundation for science technology and research, Vadlamudi. The interaction with her was really, fruitful and helped me in compiling this project report.

My deepest thanks to **DR.M. RAMESH NAIDU** Head of Chemical Department VFSTR. Vadlamudi for guiding me and extending all possible help to and always inspiring me to do my work on this project report with sincerity.

Also I would like to thank **MS P. CHANDANA** SRI whose trust and enthusiasm was constant motivation during on-going work. Last but not least I would like to thankful to all staff members of Department of Petroleum Engineering, Vignan University, Vadlamudi and who directly or indirectly helped me in the completion of this project report.

UNDERTAKING

This is to declare that the project entitled "**Biodiesel Production from Cotton Seed Oil**" is an original work done by undersigned, in partial fulfillment of the requirements for the degree "Bachelor of Technology in Chemical Engineering.

All the analysis, design and system development have been accomplished by the undersigned. Moreover, this project has not been submitted to any other college or university.

Student 1 DESIDERIO BOI ELIA :-

Student 2 Mohammad Faizan Raza faizan

Student 3 Mohammed Suliman Elhag - M. Sulima

CERTIFICATE

This is to certify that the project entitled as, **PRODUCTION OF BIODIESEL FROM COTTON SEED OIL**" submitted by '201FA17008, 201FA17002, 201FA17006' to the Vignan's Foundation for Science, Technology and Research (Deemed to be) University, Guntur, Andhra Pradesh for the award of the degree of Bachelor of Technology is a bonafide record of the research work done by him/her under my supervision. The contents of this thesis, in full or in part, have not been submitted to any other institute or university for the award of any Degree.

Project Guide

Dr.M. Ramesh Naidu
Head of the Department
Dept. Of Chemical Engineering

ABSTRACT

Biodiesel, a promising substitute as an alternative fuel has gained significant attention due to the predicted shortness of conventional fuels and environmental concern. The utilization of liquid fuels such as biodiesel produced from Cotton seed oil by Transesterification process represents one of the most promising options for the use of conventional fossil fuels. The Cotton seed oil is converted into COTTON SEEDS OIL methyl ester known as bio- diesel prepared in the presence of homogeneous acid catalyst. The physical properties such as density, flash point, Kinematic viscosity, Cloud point and Pour point were found out for Cotton seed oil and Cotton seed methyl ester. The same characteristics study was also carried out for the diesel fuel for obtaining the base line data for analysis. The value obtained from the Cotton seed methyl ester is closely matched with the values of conventional diesel and can be used in the existing diesel engine without any modification.

Keywords: Cotton seeds oil, Transesterification, Biodiesel parameters

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INTRODUCTION

Biodiesel is an alternative fuel made from renewable biological sources such as vegetable oils both (edible and non-edible oil) and animal fats. Vegetable oils are usually ester glycol with differential chain length and degree of saturation. It may be seen that vegetable contains substantial amount of oxygen in their molecules. Practically the high viscosity of vegetable oils (30- 200 Centistokes) as compared to that to Diesel (5.8- 6.4 Centistokes) leads to oils unfavourable pump, inefficient mixing of fuel with air contributes incomplete combustion, high flash point result in increased carbon deposit formation and inferior coking. Due to these problems, vegetable oil needs to be modified to bring the combustion related properties closer to those of Diesel oil. The fuel modification is mainly aimed at reducing the viscosity and increasing the volatility. One of the most promising processes to convert vegetable oil into methyl ester is the Transesterification, in which alcohol reacts with triglycerides of fatty acids (vegetable oil) in the presence of catalyst. Cotton seed vegetable oil is one of the prime non edible sources available in India. The vegetable oil used for biodiesel production might contain free fatty acids which will enhance saponification reaction as side reaction during the Transesterification process.

All countries are at present heavily dependent on petroleum fuels for transportation and agricultural machinery. The fact that a few nations together produce the bulk of petroleum has led to high price fluctuation and uncertainties in supply for the consuming nations. This in turn has led them to look for alternative fuels that they themselves can produce. Among the alternatives being considered are methanol, ethanol, biogas and vegetable oils. Vegetable oils have certain features that make them attractive as substitute for Diesel fuels.

Vegetable oil has the characteristics compatible with the CI engine systems. Vegetable oils are also miscible with diesel fuel in any proportion and can be used as extenders. India highly depends on import of petroleum crude and nearly two third of its requirement is met through imports. Moreover the gases emitted by petrol, diesel driven vehicles have an adverse effect on the environment and human health.

LITERATURE REVIEW

Introduction to work

2.1 Mechanism

2.1.1 Transesterification:

Is the process of chemically reacting a fat or oil with an alcohol in a presence of a catalyst. Alcohol used is usually methanol or ethanol Catalyst is usually sodium hydroxide or Potassium hydroxide. The main product of Transesterification is biodiesel and the co-product is glycerine

2.2 Reaction:-

Neutralization:

The vegetable oil contains about 14- 19.5 % free fatty acids in nature; it must be freed before taken into actual conversion process. The presence of about 14% of free Fatty acid makes Cotton seed oil inappropriate for industrial biodiesel production.

The dehydrated oil is agitated with 4 % HCl solution for 25 minutes and 0.82 gram of NaOH was added per 100 ml of oil to neutralize the free fatty acids and to coagulate by the following reaction. $RCOOH + NaOH \rightarrow RCOONa + H_2O$ the coagulated free fatty acid (soap) is removed by filtration. This process brings the free fatty acid content to below 2 % and is perfect source for biodiesel production

SNO	PARAMETERS	OPTIMIZATION CONDITION
1	Catalyst Amount	2 grams
2	Temperature	60-90 Degrees Celsius
3	Reaction Time	50-60 minutes
4	Methanol/Oil Ratio,	1:3
5	Biodiesel Yield	68%

Factor effecting

Scarcity, insecurity, and severe environmental impact of fossil fuel-based energy consumption have enthused the production and utilization of alternative energy resources. Biodiesel is identified as promising renewable energy that can substitute the petrol diesel consumption with numerous advantages. However, more than 95% of biodiesel is produced from edible oil crops, which jeopardizes the food supplies. As a result, exploring inexpensive and non-edible oil-bearing energy crops such as Cotton seed curses (Cotton seed) has been the target of governments, researchers, industries, and policymakers.

However, sustainable biodiesel production from this plant is not achieved yet due to various ecological, socioeconomic, legislative, and technological factors. Previous reports showed that the individual impact of those factors; however, all factors are strongly correlated, and the impact of one factor is significantly affected by the situation of other factors. Therefore, the present review is devoted to critically examine and discuss the sole and interactive effect of various factors affecting the cultivation of Cotton seed for sustainable biodiesel production by reviewing more than 185 published articles.

Various oil extraction and biodiesel production technologies and factors affecting the physicochemical properties of Cotton seed oil and biodiesel were profoundly investigated.

Moreover, the performance, combustion, and emission characteristic of diesel engines fuelled with Cotton seed biodiesel were carefully reviewed and compared with petrol diesel. In conclusion, factors affecting the sustainable biodiesel production potential of Cotton seed vary across growing Regions due to variation in determinants and the performance and emission characteristic of Diesel engines fuelled with Cotton seed biodiesel slightly differed from petrol diesel.

Introduction to Cotton plant and cottonseed production

Cottonseed oil is extracted from cotton plant. India being the second largest producer of cotton, cottonseed oil is a promising source for the production of biodiesel.

Cotton is a natural fibre that grows on a plant. The plant is a shrub native to tropical and subtropical regions around the world, including the China, India, Americas, and Pakistan. There are up to 52 species of cotton in the Gossypium genus. The cotton plant is a leafy, green shrub related to the Hibiscus. The cotton plant briefly produces cream and pink flowers that once pollinated are replaced by fruit, better known as cotton bolls. Cotton is grown

Physical Properties

- Cottonseed oil has a mild taste
- It is an odourless pale yellow liquid. The amount of colour depends on the amount of refining.
- Cottonseed oil has a smoke point of about 450 °F (232 °C).
- Density ranges from 0.917 g/cm to 0.933 g/cm
- It is less dense than water and insoluble in water.
- Cotton seed oil is sensitive to heat and light.
- Cotton seed oil is combustible.

1.2.1.3 Chemical Properties

- Moisture Content 7.21 %
- Refractive index 1.464
- Specific gravity 0.92
- PH value 4.82.

The oil content is 25-30% in the seed. The oil contains 21% saturated fatty acids and 79% Unsaturated fatty acids. These are some of the chemical elements in the seed, cursing, which is poisonous and render the oil not appropriate for human consumption. Oil has very high Saponification value and being extensively used for making soap in some countries.

Nitrogen, Phosphorous and Potassium and can be used as organic manure. Thermodynamic Conversion process, pyrolysis, useful products can be obtained from the cotton seed oil cake.

The liquid, solid (char), and gaseous products can be obtained. The liquid can be used as fuel in furnace and boiler. It can be upgraded to higher grade fuel by Transesterification process.

It is significant to point out that, the non-edible vegetable oil of cotton seed oil has the requisite potential providing a promising and commercially viable alternative to diesel oil since it has desirable physical chemical and performance characteristics comparable to diesel. Cars could be run with cotton seed without requiring much change in design. Cotton seed oil expelled from seeds and filtered through filter press can replace kerosene or oil lamp. Cotton seed oil can be used as liquid fuel for lighting and cooking. It will also be used in big Diesel engine based electricity generating sets, pump sets, heavy farm machinery, where the viscosity of oil is not an issue.

The seeds of cotton seed contain (50% by weight) viscous oil which can be used for manufacture of candles and soap, in the cosmetic industry, for cooking and lighting by itself or as a Diesel /paraffin substitute or extender

The latter use has important implications for Meeting the demand for rural energy services and also exploring practical substitute for Fossil fuels to counter greenhouse gas accumulation in the atmosphere. Also, oil is used as an illuminant in lamps as it burns without emitting smoke. It is also used as fuel in place of, or along with kerosene stoves.

Cotton seed as an energy source: There are number of variety Of Cotton seed. Best among these are cotton seed. Cotton seed oil is an important product from the Plant for meeting the cooking and lighting needs of the rural population, boiler fuel for Industrial purpose or as a viable substitute for Diesel.

About one- third of the energy in the fruit of cotton seed can be extracted as oil that has a similar energy value to Diesel fuel. Cotton seed oil can be used directly in Diesel engines added to Diesel fuel as an extender or Trans esterified to a bio-diesel fuel. There are some technical problems to Using cotton seed oil directly in Diesel engines that have yet to be completely overcome.

Moreover, the cost of producing cotton seed oil as a Diesel substitute is currently higher than the cost of Diesel itself.

Use as jet fuel: Aviation fuels may be more widely substituted with biofuels such as cotton Oil than fuels for other forms of transportation. On December 30, 2008, Air New Zealand flew the first successful test flight with a Boeing 747 running one of its four Rolls-Royce engines on a 50:50 blend of cotton seed oil and jet A-1 fuel. Subsequently, Air New Zealand and Houston based Continental Airlines have run tests in Jan. 2009, further demonstrating the viability of cotton seed oil as a jet fuel.

MATERIALS AND METHODS

Materials

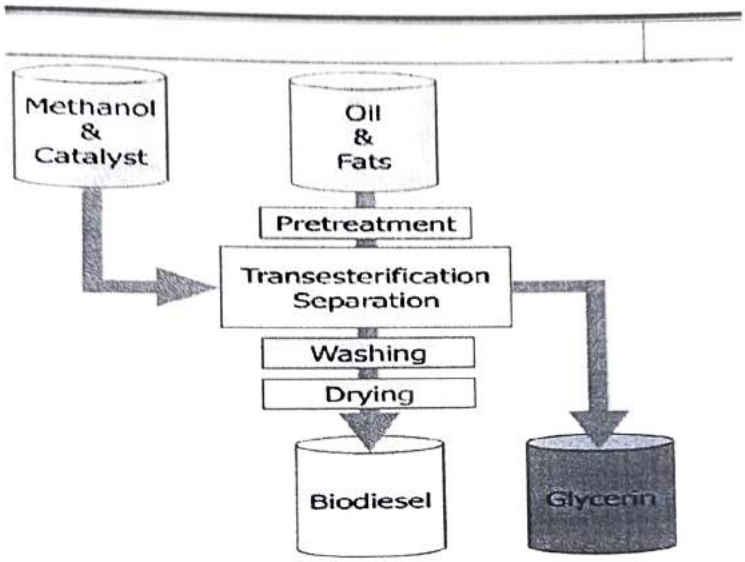
Materials and apparatus used in the production of the biodiesel are as follows: -

S.NO	MATERIALS
1	Distilled Water, Methanol, And Cotton seed Oil.
2	Retort Stand, Separating Funnel, Magnetic Stirrer, Oven, Conical Flask, Digital Weighing Balance, Stop Watch, Hot Plate
3	Pipette ,Thermometer
4	Measuring Cylinder, Conical Flask, Digital Weighing Balance.

Methods.

Steps in biodiesel production: two steps are used in the production of the biodiesel as followed by:-

(i) Transesterification.-



The step by step approach used in the production of the biodiesel is given below.

(i) 100 mL of cotton seed oil was measured and poured into 250 mL conical flask and heated to a temperature of 50°C. Seen in fig 1

Preheating of cotton seeds oil (fig 1)

(ii) A 50mL of methanol was poured in a round bottom flask and soxhlet apparatus, and the heater was turned on. This was done to purify the methanol.

(iii)The sodium hydroxide pellet was placed in the weighing balance to get exactly 2 grams

Fig.2 Mixing of NaOH and Methanol

(v) The dissolved mixture in a separate vessel and was poured into the heated cotton seed oil round in bottom flask while stirring the mixture continuously. The mixture was maintained at atmospheric pressure and 60°C for 60 minutes.

(vi) After completion of Transesterification process, the mixture is allowed to settle under Gravity for 24 hours in a separating funnel. (Fig.3)

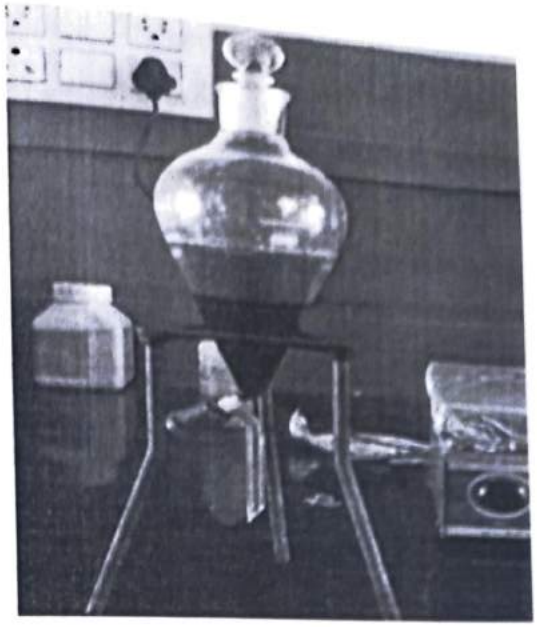


Fig.3 Heterogeneous Phase Separation

(vii) The products formed during Transesterification were Cotton seed oil methyl ester and Glycerine.

(viii) The bottom layer consists of Glycerine, excess alcohol, catalyst, impurities and traces of unreacted oil. The upper layer consists of biodiesel, alcohol and some soap

(ix)After separation of glycerine we will get the biodiesel (fig 4)

(x) After filter we will get purified biodiesel (fig 5)

Fig.4 Separated Biodiesel

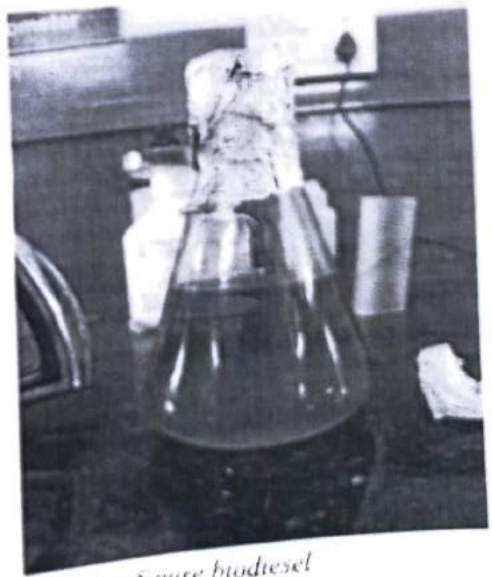


Fig. 5 pure biodiesel

YIELD PERCENTAGE

Yield of biodiesel (%) =mass of biodiesel obtained / mass of oil used*100.

Mass of biodiesel obtained= **324 grams**

Mass of oil used= **400 grams**

The yield (%) was found to be **68%**

Parameters of biodiesel

Determination of Specific Gravity: -

Density bottle was used to determine the density of the oil. A clean dry bottle of 25 mL was weighed (w0) and then filled with the oil; a stopper was inserted and then reweighed to give (w1). The oil substituted with water after washing and drying and weighed to give (w2) as

Specific gravity :- **(w1-w0)/ (w2-w0)**

Determination of Moisture Content:

The oil sample was weighed and the mass taken as (w1) thus was then dried in the oven and the weight after drying was taken as (w2). The percentage moisture in the oil was then calculated using the formula below:

%moisture content = (w1-w2)/w1 *100

Flash point:

150 ml of blended oils was poured into a metal container and heated at a controlled rate over certain temperature after, which, the flame being passed over the surface of the blended oils was observed at a regular intervals of 5 sec to 1 min. The flash point was determined using pesky marten's equipment.

Flame point:

150 ml of blended oils was poured into a metal container and heated at a control rate over certain temperature after, where the continuous flame being observed over the surface of the blended oils was observed at regular intervals of 5 sec to 1 min. The flame point was determined.

Determination of Saponification Value:-

The oil sample was filtered to remove any impurities and last traces of moisture. 5 g of the sample was then weighed into a flask and 5 mL of alcoholic KOH was added from burette allowing it to drain for the same duration of time. A reflux condenser was connected to the flasks and allowed to boil gently for one hour. After the flask and condenser get cooled, they are rinsed down the inside of the condenser with a little distilled water and then the condenser was removed About 1 mL of indicator was added and titrated against 0.5 m HCL until the pink colour vanish.

Characterization of parameters:

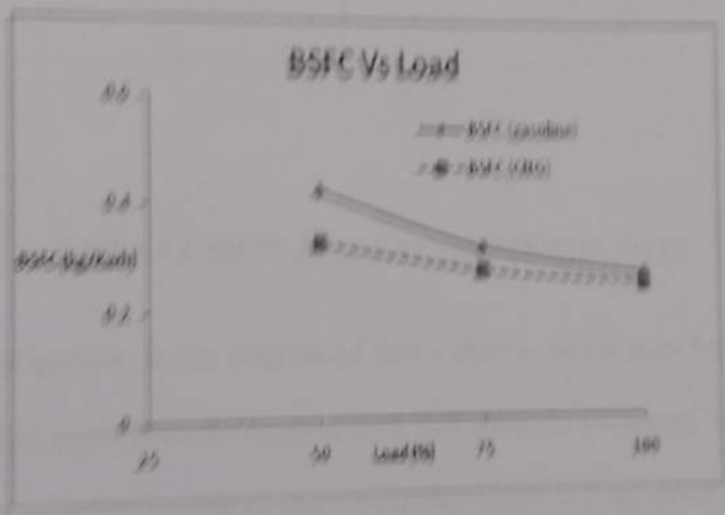
S.NO	Properties	Units	Result
1	Specific gravity	Kg/L	0.88
2	Flash point	°C	48
3	Fire point	°C	72
4	Total glycerine	%mass	%0.22
5	Saponification	Mg kOH/g	190
6	PH	Unit less	0.20
7	Moisture content	unit less	5.63
8	Density	g/cc	0.875

4.2. Results/Optimizations

Engine Performance:

Based on Diesel

S.NO	LOAD	SPEED(rpm/s)	TIME(sec)	BTH (%)	BSFC	BP(KW)
1	3 kg	1500	44	10.93	0.815	0.883
2	6 kg	1500	33	15.58	0.514	1.748
3	9 kg	1500	30	19.146	0.42	2.563



Based on Biodiesel

S.NO	LOAD	SPEED	TIME(Sec)	BTH (%)	BSFC	BP(g/kw)
1	3 kg	1500	44	10.14%	0.8064	0.883
2	6 kg	1500	36.2	15.14%	0.538	1.692
3	9 kg	1500	33.4	18.5%	0.395	2.423

Parameters comparison study

PROPERTIES	DIESEL	BIODIESEL
Flash Point	65	48
Flame Point	70	72
Specific Gravity	0.79	0.88
Density	0.85	0.875

CONCLUSION AND RECOMMENDATION

In the current investigation, it has confirmed that cotton seed oil may be used as resource to obtain biodiesel. The experimental result shows that alkaline-catalysed Transesterification is a promising area of research for the production of biodiesel in large scale. Materials for use in the production of biodiesel are readily available without the need for special equipment or scarce chemicals. Effects of different parameters such as temperature, time, reactant ratio, and catalyst concentration on the biodiesel yield were analysed. The best combination of the parameters was found as 8: 1 molar ratio of methanol to oil, 1.0% KOH catalyst, 60°C reaction temperature and 60 minutes of reaction time. This optimum condition yielded 90% of biodiesel. The viscosity of Cotton seed oil reduces substantially after Transesterification and is comparable to diesel. Biodiesel characteristics like density, flash point, specific gravity are comparable to diesel.

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A

PROJECT REPORT

ON

"EXTRACTION OF ANTIOXIDENTS FROM MEDICAINAL PLANTS"

Submitted in the partial fulfilment of the requirements for the award of the degree

BACHELOR OF TECHNOLOGY

in

Division of Petroleum Engineering

Submitted by :-

A. VISHNU 191FA17001

UNDER THE GUIDANCE:

Mr. PRATHAMESH SAPALE

DEPARTMENT OF CHEMICAL ENGINEERING

VIGNAN'S FOUNDATION FOR SCIENCE, TECHNOLOGY AND RESEARCH

GUNTUR, VADLAMUDI-522213

DEC-2022

CERTIFICATE

This is to certify that the Thesis entitled as **"PREPARATION OF HYDROGEL FOR CONFORMANCE CONTROL IN THE EOR APPLICATION"** submitted by **B.TARUN KUMAR(191FA17001)** to the Vignan's Foundation for Science, Technology and Research (Deemed to be) University, Guntur, Andhra Pradesh, India, for the award of the degree of Bachelor of Technology (B. Tech) is a bonafide record of the research work done by him under my supervision. The contents of this thesis, in full or in part, have not been submitted to any other institute or university for the award of any degree.

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ABSTRACT

The synthesis and characterization of Polyacrylamide/clay nanocomposites for the development of hydrogel system used in enhanced oil recovery is described. The synthesized nanocomposite copolymer was crosslinked with Chromium (III) acetate to form the hydrogel which exhibited an acceptable gel strength, gelation time and gel stability. The nanocomposite gels prepared with low crosslinker concentration (2000 ppm chromium acetate) showed higher gel strength and required longer gelation time than the conventional Polyacrylamide (PAM) gel; these are desirable properties for the effective placement of gel uring enhanced oil recovery operations.

1. INTRODUCTION

Water production from the economically capable hydrocarbon reservoir restricts the actual hydrocarbon production. To overcome this problem we have so many mechanical and chemical solutions that works effectively. In case of chemical solutions we have polymer injection method which is widely used method for water shutoff jobs around the world. In thismethod polymers shows reduction in swelling and mechanical toughness of the gel. Nanocomposite hydrogel is one of the solution to overcome this disadvantages and perform inan effective manner to control waterproduction fromoil or gas wells. Nanoclay is introduced in the synthesis of the nanocomposite hydrogel to improve the mechanical properties of the gel.

Hydrogels are widely used in many applications, such as hygiene, cosmetics, agriculture, medicine, biotechnology, and petroleum recovery treatments of mature reservoirs. Hydrogels which is prepared by the polyacrylamide having cross-inker polymers enhance hydrogels mechanical strength, but preparing a hydrogel having large amount of cross-inkers results in the reduction of swelling capability and mechanical toughness. To overcome the weakness and limitations of the conventional hydrogels, preparation of new type of hydrogels, like nanocomposite hydrogels, has recently attracted more attention. These nanocomposite hydrogels have excellent properties, such as good mechanical toughness, large deformability, high swelling/deswelling rates, and high transparency.

The internal actives takes plays in the preparation of the nanocomposite hydrogel are the acrylamide chains are bound to the surface of the clay particles due to hydrogen bonds between the oxygen atoms of clay and the amide protons of the acrylamide as well as due to complex formation between the metal ions on the clay surface and the carbonyl oxygen of the acrylamide. Nanocomposite hydrogel is prepared by the synthesized by free-radical

polymerization/gelation ofthe monomers in the presence of the nanoclays

2 EXPERIMENTAL EQUIPMENTS

2.1 Scanning electron microscope

- Principle
- SEM sample preparation
- Advantages
- Disadvantages
- Applications

2.1.1Principle

The basic principle is that a beam of electrons are generated by a suitable source, typically a tungsten filament or a field emission gun. The electron beam is accelerated through a high voltage and passes through a system of apertures and electromagnetic lenses to produce a thin beam of electrons, then the beam scans the surface of the specimen. Electrons are emitted from the specimen by the action of the specimen beam and collected by a suitable position detector.

2.1.2Working

The SEM uses electrons instead of light to form an image. A beam ofelectrons is produced at the top of the microscope by heating of a metallic filament. The electron beam follows a vertical path through the column of the microscope. It makes its way through electromagnetic lenses which focus and direct the beam down towards the sample. Once it hits the sample, other electrons (backscattered or secondary) are ejected from the sample. Detectors collect the secondary or backscattered electrons, and convert them to a signal that is sent to a viewing screen similar to the one in an ordinary television, producing an image.

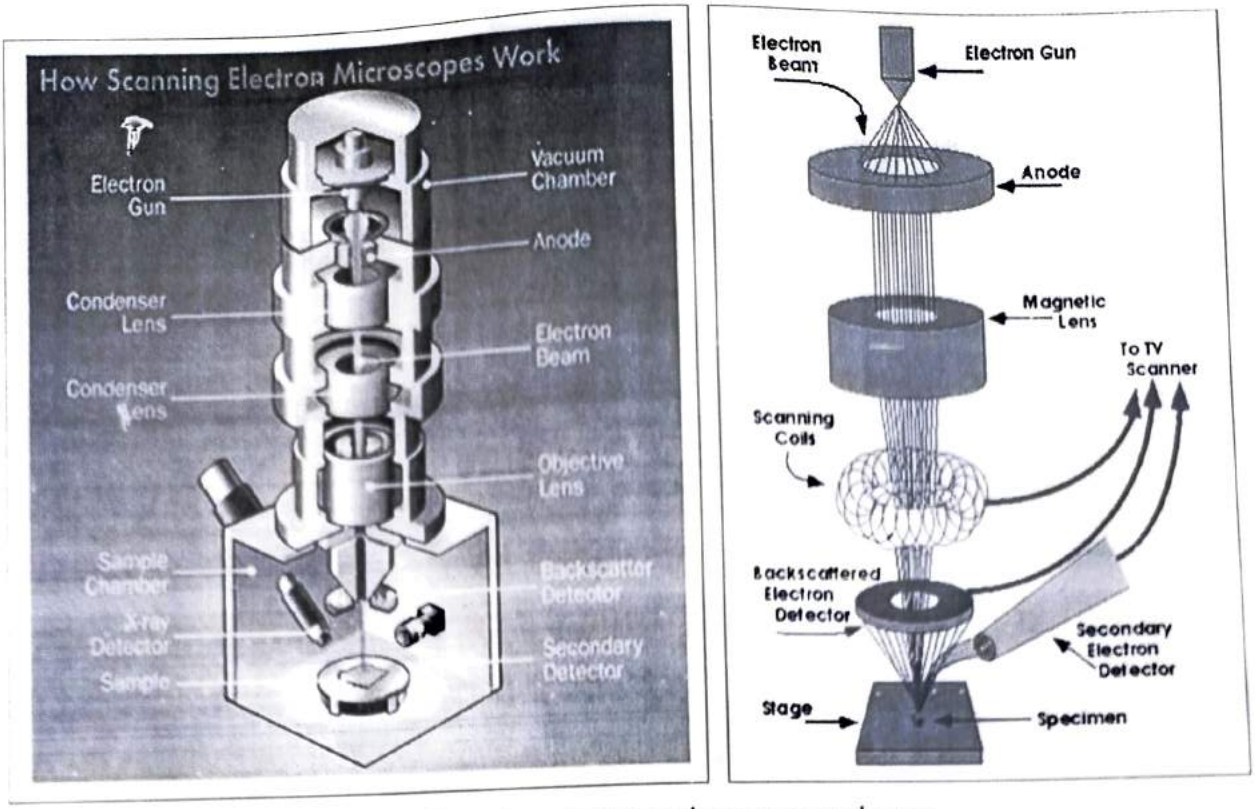


Figure 2.1 : Scanning electron microscope and parts.

2.1.3Electron guns Electron guns are typically one of two types:

- Thermionic guns
- Field emission guns

2.1.3.1Thermionic guns

Which are the most common type, apply thermal energy to a filament to coax electrons away from the gun and toward the specimen under examination. Usually made of tungsten, which has a high melting point.

2.1.3.2Electron guns field emission guns

Create a strong electrical field to pull electrons away from the atoms they are associated with. Electron guns are located either at the very top or at the very bottom of an SEM and fire a beam of electrons at the object under examination. These electrons don't naturally go where they need to, however, which gets us to the next component of SEMs.

2.1.4Condenser lenses

Just like optical microscopes, SEMs use Condenser lenses to produce clear and detailed images. The Condenser lenses in these devices, however, work differently. For one thing, they aren't made of glass. Instead, the Condenser lenses are made of magnets capable of bending the path of electrons. By doing so, the Condenser lenses focus and control the electron beam, ensuring that

the electrons end up precisely where they need to go.

2.1.5Chamber

The sample chamber of an SEM is where researchers place the specimen that they are examining. Because the specimen must be kept extremely still for the microscope to produce clear images, the sample chamber must be very sturdy and insulated from vibration. In fact, SEMs are so sensitive to vibrations that they are often installed on the ground floor of a building.

2.1.6Detectors

Various types of detectors are there in SEM. These devices detect the various ways that the electron beam interacts with the sample object. For instance, Everhart-Thornley detectorsregister secondary electrons, which are electrons dislodged from the outer surface of a specimen.These detectors are capable of producing the most detailed images of an object's surface. Other detectors, such as backscattered electron detectors and X-ray detectors, can tell researchers about the composition of a substance.

2.1.7Secondary electron detector

(Everhart-Thornley) Backscattered electron detector: (Solid-State Detector) Secondaryelectrons: Everhart-Thornley Detector Backscattered electrons: Solid State Detector X-rays: Energy dispersive spectrometer (EDS).

2.1.8Sample preparation

Cleaning the surface of the specimen- The proper cleaning of the surface of the sample is important because the surface can contain a variety of unwanted deposits, such as dust, silt, and detritus, media components, or other contaminants, depending on the source of the biological material and the experiment that may have been conducted prior to SEM specimen preparation.

- Stabilizing the specimen- Stabilization is typically done with fixatives. Fixation can be achieved, for example, by perfusion and microinjection, immersions, or with vapours using various fixatives including aldehydes, osmium tetroxide, tannic acid, or thiocarbohydrazide.
- Rinsing the specimen- After the fixation step, samples must be rinsed in order to remove the excess fixative.
- Dehydrating the specimen- The dehydration process of a biological sample needs to be done very carefully. It is typically performed with either a graded series of acetone orethanol.
- Drying the specimen - The scanning electron microscope (like the transmission electron microscope) operates with a vacuum. Thus, the specimens must be dry or the sample will be destroyed in the electron microscope chamber. Many electron microscopists consider a procedure called the Critical Point Drying (CPD) as the gold standard for SEM specimen drying. Carbon dioxide is removed after its transition from the liquid to the gas phase at the critical point, and the specimen is dried without structural damage.

- Mounting the specimen- After the sample has been cleaned, fixed, rinsed, dehydrated, and dried using an appropriate protocol, specimens must be mounted on a holder that can be inserted into the scanning electron microscope. Samples are typically mounted on metallic (aluminum) stubs using a double-sticky tape. It is important that the investigator firstdecides on the best orientation of the specimen on the mounting stub before attaching it. A re-orientation proves difficult and can result in significant damage to the sample.
- Coating the specimen- The idea of coating the specimen is to increase its conductivity in the scanning electron microscope and to prevent the build-up of high voltage charges on the specimen by conducting the charge to ground. Typically, specimens are coated with a thin layer of approximately 20 nm to 30 nm of a conductive metal (e.g., gold, gold-palladium, or platinum).A spider coated in gold, having been prepared for viewing with a scanningelectron microscope.

2.1.9Advantages

Advantages of a Scanning Electron Microscope include its wide-array of applications, the detailed three-dimensional and topographical imaging and the versatile information garnered from different detectors. SEMs are also easy to operate with the proper training and advances in computer technology and associated software make operation user-friendly. Although all samples require minimal preparation actions.

2.1.10Disadvantages

The disadvantages of a Scanning Electron Microscope start with the size and cost. SEMs are expensive, large and must be housed in an area free of any possible electric, magnetic or vibration interference. Maintenance involves keeping a steady voltage, currents to electromagnetic coils and circulation of cool water. SEMs are limited to solid, inorganic samples small enough to fit inside the vacuum chamber that can handle moderate vacuum pressure.

2.1.11Applications

SEMs have a variety of applications in a number of scientific and industry-related fields, especially where characterizations of solid materials are beneficial. In addition to topographical, morphological and compositional information, a Scanning Electron Microscope can detect and analyze surface fractures, provide information in microstructures, examine surface contaminants, reveal spatial variations in chemical compositions, provide qualitative chemical analyses and identify crystalline structures

2.2 Thermo-Gravimetric Analysis

- Introduction
- Types
- Principle

- Instrumentation
- Factors affecting results
- Advantages
- Limitations
- Applications

2.2.1Thermo-Gravimetric Analysis

Thermo-Gravimetric Analysis or Thermal Gravimetric Analysis (TGA) is a method of thermal analysis in which the mass of a sample is measured over time as the temperaturemust be prepared before being placed in the vacuum chamber, most SEM samples changes.

Three types of thermo-gravimetry

- Isothermal or static thermo-gravimetry: In this technique the sample weight is recorded as function of time at constant temperature.
- Quasistatic thermo-gravimetry: In this technique the sample is heated to constant weight at each of series of increasing temperatures.
- Dynamic thermo-gravimetry: In this technique the sample is heated in an environment whose temperature is changing in a predetermined manner generally at linear rate.

2.2.2Principle

In thermo-gravimetric analysis, the sample is heated in a given environment (air, N2, CO2, He, Ar, etc.) at controlled rate. The change in the weight of the substance is recorded as a function of temperature or time.

This plot of weight change against temperature is called thermo-gravimetric curve or thermo-gram. this is the basic principle of TGA.

2.2.3Instruments

- Recording balance
  - Sample holder
  - Furnace
  - Temperature programmer /controller (thermocouple)
  - Recorder

2.2.4Recording balance

A microbalance is used to record a change in mass of sample/ substance. An ideal microbalance must possess following features. It should provide electronic signals to record the change in mass using a recorder. The electronic signals should provide rapid response to change in mass.

2.2.5Sample holder

The sample to be studied is placed in sample holder or crucible. It is attached to the weighing arm of microbalance. There are different varieties of crucibles used. Some differ in shape and size while some differ in materials used. They are made up from platinum, aluminum, quartz or alumina and some other materials like graphite, stainless steel, glass etc

- There are different types of crucibles. They are: Shallow pans(used for volatile substances)
- Deep crucibles (Industrial scale calcination)
- Loosely covered crucibles (self generated atm. Studies)
- Retort cups (Boiling point studies)

2.2.6Furnace

The furnace should be designed in such way that it produces a linear heating range. It should have a hot zone which can hold sample and crucible and its temperature corresponds to the temperature of furnace. There are different combinations of microbalance and furnace available

2.2.7Temperature programmer/controller

Temperature measurement is done in no. of ways thermocouple is the most common technique. The position of the temperature measuring device relative to the sample is very important. The major types are

- The thermocouple is placed near the sample container and it has no contact with the sample container. This isn't a good arrangement where low-pressure are employed.
- Temperature measurement is done in number of ways, thermocouple is the most common technique. The position of the temperature measuring device relative to the sample is very important.

2.2.8Recorder

The recording systems are mainly of 2 types

- Time-base potentiometric strip chart recorder.
- X-Y recorder.

In some instruments, light beam galvanometer, photographic paper recorders or one recorder with two or more pens are also used. In the X-Y recorder, we get curves having plot of weights directly against temperatures

2.2.9Factors affecting TGA

Factors affecting the TG curve The factors which may affect the TG curves are classified into two main groups.:

- Instrumental factors
    1) Furnace heating rate
    2) Furnace atmosphere.
- Sample characteristics includes
    1) Weight of the sample
    2) Sample particle size.

2.2.10Advantages of TGA

A relatively small set of data is to be treated. Continuous recording of weight loss as a function of temperature ensures Equal weightage to examination over the whole range of study. As a single sample is analyzed over the whole range of temperature, the variation in the value of the kinetic parameters, if any, will be indicated.

2.2.11Limitations of TGA

The Chemical or physical changes which are not accompanied by the change in mass on heating are not indicated in thermo- gravimetric analysis. During TGA, Pure fusion reaction, crystalline transition, glass transition, crystallization and solid state reaction with no volatile product would not be indicated because they provide no change in mass of the specimen.

2.2.12Applications of TGA

From TGA, we can determine the purity and thermal stability of both primary and secondary standard. TGA is used to study the kinetics of the reaction rate constant. Used in the study of catalyst: The change in the chemical states of the catalyst may be studied by TGA techniques. (Zn- ZnCrO4) Zinc-Zinc chromate is used as the catalyst in the synthesis of methanol. Analysis of the dosage form. Oxidative stability of materials. Estimated lifetime of a product.

2.3X-Ray Diffraction

- Introduction
- Generation of x-rays
- Principle

- Methods
- Applications

2.3 Introduction

X rays were discovered by Wilhelm Roentgen who called them x-rays because the nature at first was unknown so, x rays are also called Roentgen rays. X-ray diffraction in crystals was discovered by Max von Laue. The wavelength range is 10-7 to about 10-15 m. The penetrating power of x rays depends on energy also, there are two types of x rays.

- Hard x rays: which have high frequency and have more energy.
- soft x rays: which have less penetrating and have low energy Max.

2.3.2X-Rays

- X-rays are short wave length electromagnetic radiations produced by the deceleration of high energy electrons or by electronic transitions of electrons in the inner orbital of atoms.
- X-ray region 0.1 to 100 A° 3.Analytical purpose 0.7 to 2 A°.

2.3.3Principle

X-ray diffraction is based on constructive interference of monochromatic x-rays and a crystalline sample. These x-rays are generated by a cathode ray tube, filtered to produce monochromatic radiation, collimated to concentrate and directed towards the sample. The interaction of incident rays with the sample produces constructive interference when conditions satisfy Bragg's law.

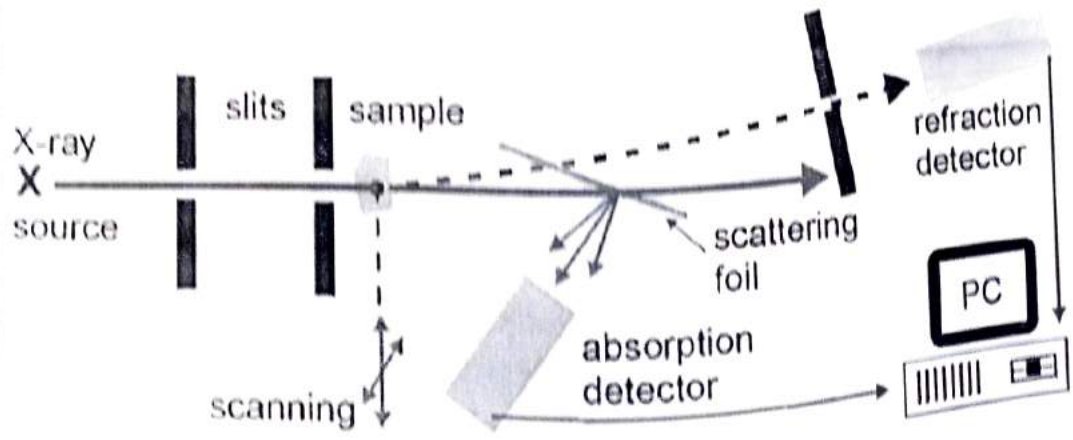


Figure 2.3 : X-Ray diffraction principle.

2.3.4X-Ray Diffraction methods

These are generally used for investigating the internal structures and crystal structures of various solid compounds. They are

- Laue's photographic method
  a) Transmission method
  b) Back reflection method
- Bragg's X-ray spectrometer method
- Rotating crystal method 4 Powder method

2.3.5Applications of XRD

- Structure of crystals
- Polymercharacterisation
- State of anneal inmetals
- Particle size determination
  a) Spot counting method
  b) Broadening of diffraction lines
  c) Low-angle scattering
- Applications of diffraction methods to complexes
  a) Determination of cis-trans isomerism
  b) Determination of linkage isomerism
- Miscellaneous applications

2.4Fourier Transform Infrared Analysis

FT-IR stands for Fourier Transform Infrared, the preferred method of infrared spectroscopy. In infrared spectroscopy, IR radiation is passed through a sample. Some of the infrared radiation is absorbed by the sample and some of it is passed through (transmitted). The resulting spectrum represents the molecular absorption and transmission, creating a molecular fingerprint of the sample. Like a fingerprint no two unique molecular structures produce the same infrared spectrum. This makes infrared spectroscopy useful for several types of analysis.

- It can identify unknown materials.
- It can determine the quality or consistency of a sample.
- It can determine the amount of components in a mixture.

2.4.1Superiority of FT-IR

- Fourier transform infrared spectroscopy is preferred over dispersive or filter methods of infrared spectral analysis for several reasons:

- It is a non-destructive technique.
- It provides a precise measurement method which requires no external calibration.
- It can increase speed, collecting a scan every second, It can increase sensitivity.
- It has greater optical throughput.
- It is mechanically simple with only one moving part.

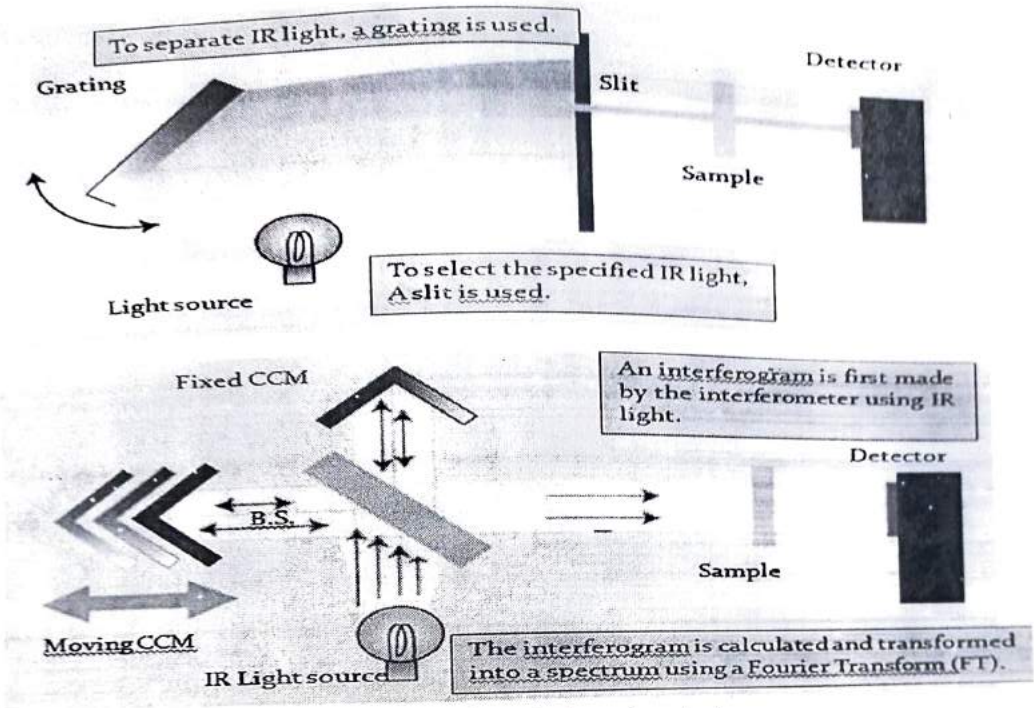


Figure 2.4 : Fourier transform infrared analysis.

2.4.2Sampling techniquesLiquidSamples

- Neat sample Diluted solution Liquid cell

Solid Samples

- Neat sample Cast films Pressed films KBr pellets Mull

Gas Samples

- Short path cell Long path cell

2.4.3The Sample Analysis Process The normal instrumental process is as follows:

2.4.4The Source

Infrared energy is emitted from a glowing black- body source. This beam passes through an aperture which controls the amount of energy presented to the sample (and, ultimately,to the detector).

2.4.4.1The Interferometer

The beam enters the interferometer where the "spectral encoding" takes place. The resulting interferogram signal then exits the interferometer.

2.4.4.2The Sample

The beam enters the sample compartment where it is transmitted through or reflected off of the surface of the sample, depending on the type of analysis being accomplished. This is where specific frequencies of energy, which are uniquely characteristic of the sample, are absorbed.

2.4.5The Detector

The beam finally passes to the detector for final measurement. The detectors used are specially designed to measure the special interferogram signal.

2.4.6Advantages of FT-IR

2.4.6.1Speed

Because all of the frequencies are measured simultaneously, most measurements by FT-IR are made in a matter of seconds rather than several minutes.

2.4.6.2Sensitivity

Sensitivity is dramatically improved with FT-IR for many reasons

2.4.6.3Mechanical Simplicity

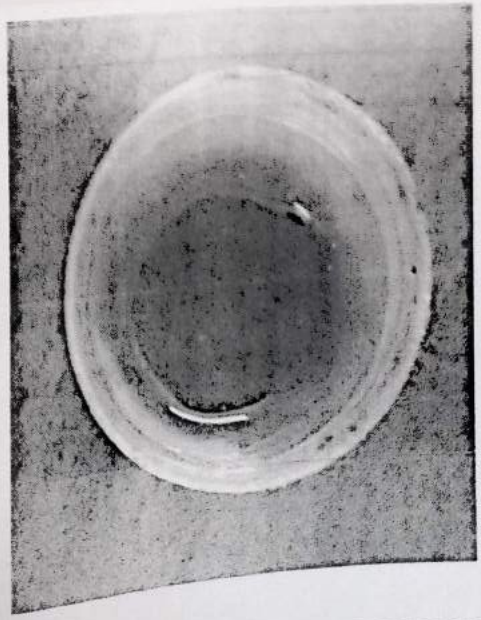
Thus, there is very little possibility of mechanical breakdown.

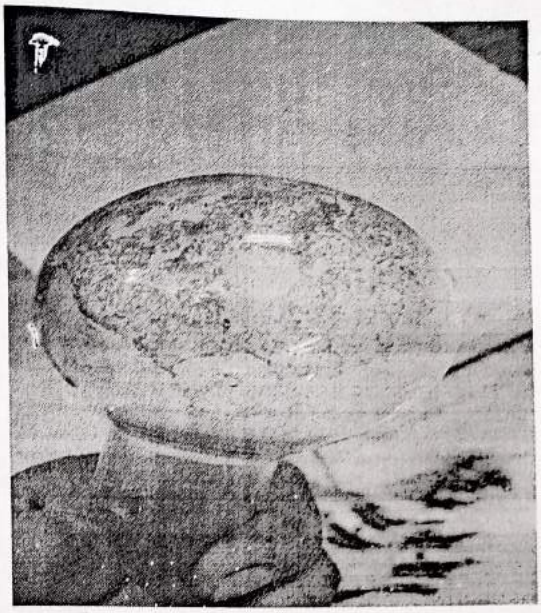
2.4.6.4Internally Calibrated

These instruments employ a HeNe laser as an internal wavelength calibration standard. These instruments are self-calibrating and never need to be calibrated by the u

Preparation of Sample:

- The sample we prepared is using 200mg PHPA + 25ml H_2O + 125mg Chromium Acetate and stir it for five hours using magnetic stirrer and the pellet.
- We don't need to disturb the gel in between the stirring time, and we will give it for analysis.
- In this we will take a 100ml beaker we will weigh all the chemicals using weighing machine as per the requirement and in the PHPA and Chromium acetate we will add distilled water and mix it up well and stir for 5 hours.
- After stirring we will use High Vaccum Pump and Rotary Evaporator to dry the sample then we will get the powder form of the sample, XRD, SEM , FTIR, TGA.
- After the preparation of the sample we have to store it in a plastic bottle.



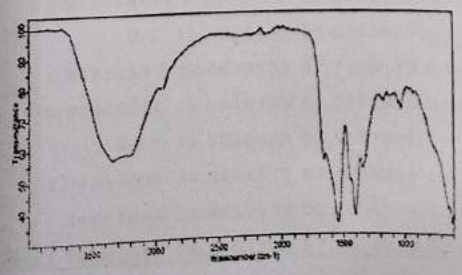


The FTIR Result of sample (Fourier transform infrared spectroscopy):

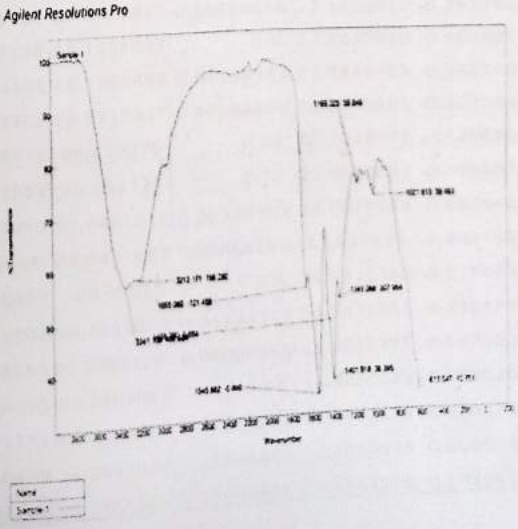
VIGNAN'S
Foundation for Science, Technology & Research
(Deemed to be University)
-Estd. u/s 3 of UGC Act 1956

Sample ID:Sample-1
Sample Scans:64
BackgroundScans:64
Resolution:4
System Status:Good
File Location:C:\Program Files (x86)\Agilent\MicroLab PC\Results\2018\Textile\20.04.2018\Sample-1_2022-04-22T12-07-23.a2r

Method Name:ATR-600-4000
User:V F S T R
Date/Time:04/22/2022 12:07:22 PM
Range:4000 -600
Apodization:Triangular



Agilent Resolutions Pro

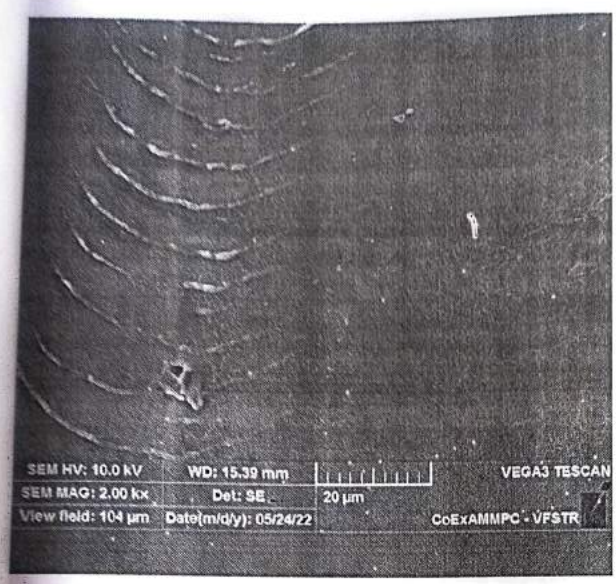


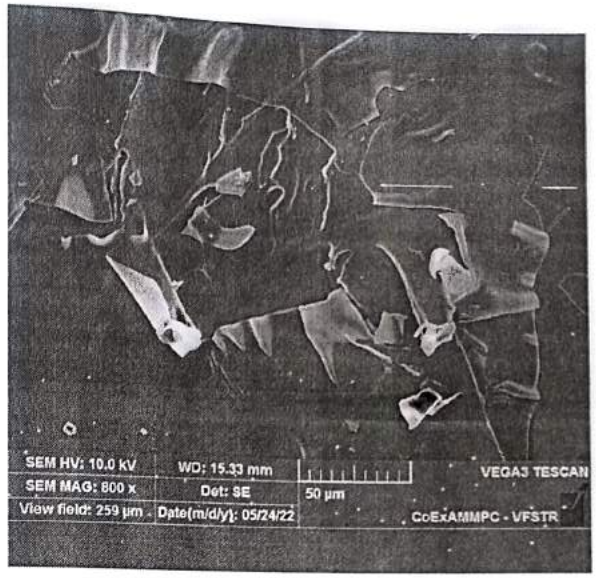
	A	B	C
1	XLabel	Wavenumber	
2	YLabel	%Transmittance	
3	FileType	%Transmittance	
4	DisplayDir	20300	
5	PeakDirect	20311	
6	600.1014	35.29524	
7	600.5673	35.71785	
8	601.0332	36.00062	
9	601.4991	36.16704	
10	601.9651	36.24088	
11	602.431	36.23709	
12	602.8969	36.16318	
13	603.3628	36.02764	
14	603.8287	35.84873	
15	604.2946	35.65745	
16	604.7606	35.49211	
17	605.2265	35.38656	
18	605.6924	35.35768	
19	606.1583	35.39829	
20	606.6242	35.47944	
21	607.0902	35.56099	
22	607.5561	35.6055	
23	608.022	35.58914	
24	608.4879	35.50549	
25	608.9538	35.36197	
26	609.4197	35.17219	
27	609.8857	34.94902	
28	610.3516	34.70195	

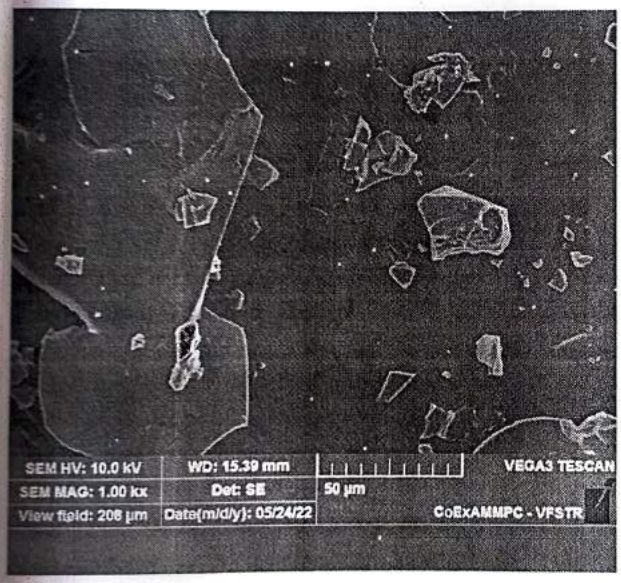
The TGA Result of the sample (Thermo gravimetric analysis):

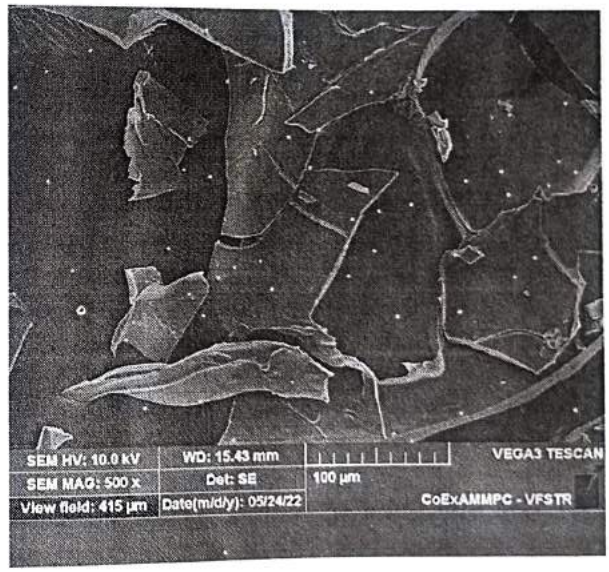
	A	B	C	D	E	F	G	H	I
1	Time	Temp	TG	Time	Temp	DTG	Time	Temp	DTA
2	min	Cel	%	min	Cel	ug/min	min	Cel	uV
3	0	21.1122283	99.8681481	0	21.1122283	75.515625	0	21.1122283	-0.05217003
4	0.04	21.1755065	99.7861052	0.04	21.1755065	75.6679687	0.04	21.1755065	-0.08040583
5	0.08333333	21.2075099	99.6969316	0.08333333	21.2075099	76.0195312	0.08333333	21.2075099	-0.11132776
6	0.12666666	21.2824764	99.6072543	0.12666666	21.2824764	77.2617187	0.12666666	21.2824764	-0.13987457
7	0.17	21.3547058	99.5151648	0.17	21.3547058	79.6289062	0.17	21.3547058	-0.16716206
8	0.21333333	21.4144706	99.4198147	0.21333333	21.4144706	81.6796875	0.21333333	21.4144706	-0.19359540
9	0.25666666	21.5058116	99.3230598	0.25666666	21.5058116	82.4296875	0.25666666	21.5058116	-0.21540868
10	0.3	21.5829601	99.2257746	0.3	21.5829601	83.0976562	0.3	21.5829601	-0.23793148
11	0.34333333	21.6768837	99.1276942	0.34333333	21.6768837	83.5546875	0.34333333	21.6768837	-0.25811505
12	0.38333333	21.7446861	99.0368241	0.38333333	21.7446861	83.6015625	0.38333333	21.7446861	-0.27713465
13	0.42666666	21.8116092	98.9386112	0.42666666	21.8116092	83.4257812	0.42666666	21.8116092	-0.29883217
14	0.47	21.9118328	98.8406103	0.47	21.9118328	83.8476562	0.47	21.9118328	-0.31657927
15	0.51333333	22.0268859	98.7412575	0.51333333	22.0268859	84.515625	0.51333333	22.0268859	-0.33419077
16	0.55666666	22.1596927	98.6418518	0.55666666	22.1596927	84.5507812	0.55666666	22.1596927	-0.35198128
17	0.6	22.3017520	98.5423134	0.6	22.3017520	85.1835937	0.6	22.3017520	-0.36959350
18	0.64333333	22.4346485	98.4414497	0.64333333	22.4346485	86.2382812	0.64333333	22.4346485	-0.38824927
19	0.68666666	22.5753803	98.3394991	0.68666666	22.5753803	86.8710937	0.68666666	22.5753803	-0.40723931
20	0.73	22.7310371	98.2371244	0.73	22.7310371	86.859375	0.73	22.7310371	-0.42568187
21	0.77	22.8662052	98.1430202	0.77	22.8662052	86.953125	0.77	22.8662052	-0.44388735
22	0.81333333	23.0079650	98.0403009	0.81333333	23.0079650	87.6445312	0.81333333	23.0079650	-0.46744143
23	0.85666666	23.1512928	97.9368394	0.85666666	23.1512928	88.0429687	0.85666666	23.1512928	-0.49475586
24	0.9	23.3936462	97.8331923	0.9	23.3936462	88.59375	0.9	23.3936462	-0.51744341
25	0.94333333	23.6473503	97.7285113	0.94333333	23.6473503	89.6132812	0.94333333	23.6473503	-0.53872954
26	0.98666666	23.8015155	97.6222929	0.98666666	23.8015155	90.8203125	0.98666666	23.8015155	-0.56730210
27	1.03	23.9830093	97.5147491	1.03	23.9830093	92.0507812	1.03	23.9830093	-0.59852695
28	1.07333333	24.2171573	97.4055883	1.07333333	24.2171573	93.1640625	1.07333333	24.2171573	-0.62947857
29	1.11666666	24.4850139	97.2954997	1.11666666	24.4850139	94.3242187	1.11666666	24.4850139	-0.66055417
30	1.15666666	24.7164268	97.1924358	1.15666666	24.7164268	95.2851562	1.15666666	24.7164268	-0.69169139

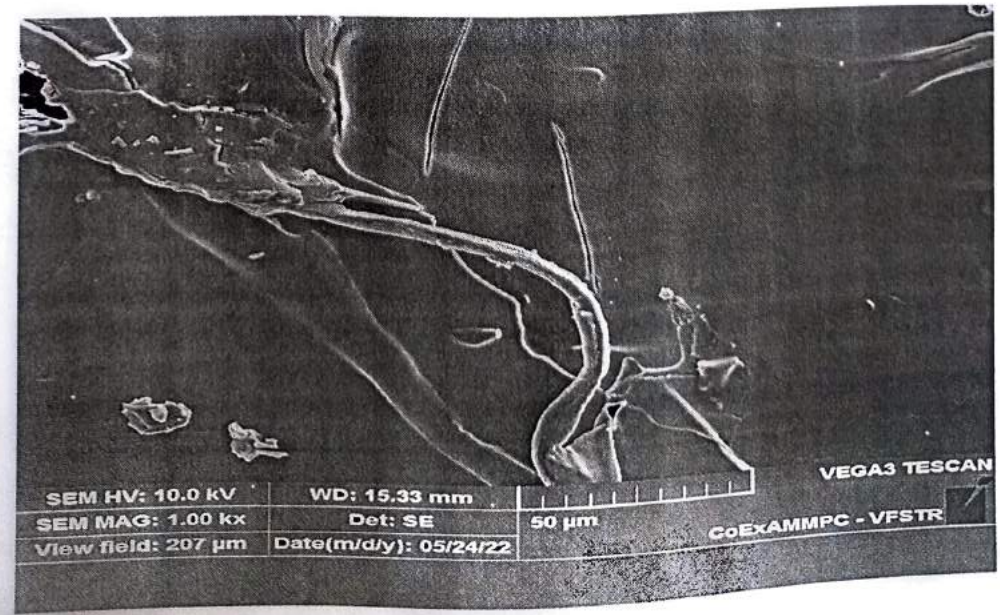
he SEM Result of the sample (Scanning electron microscope):











e XRD Result of the sample (X-ray powder diffraction):

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SAMPLE          =  Sample-1-1
COMMENT         =
FNAME           =  Sample-1-1_Theta_2-Theta.ASC
DATE            =  27-May-22 15:09

GROUP_COUNT     =  1
GONIO           =  MiniFlex 300/600, 150
ATTACHMENT      =  Standard Sample Holder
ASC             =  0, 0, 0.000000, 0.000000
FILTER          =  K-beta(Ni)
C_MONOCHRO      =  None, 0.000000
SLIT_NAME       =  0, IHS
SLIT_NAME       =  1, DS
SLIT_NAME       =  2, SS
SLIT_NAME       =  3, RS
COUNTER         =  SC-70, 0
POS_FORMAT      =  0
SCAN_AXIS       =  2theta/theta
MEAS_MODE       =  Continuous Scanning
TARGET          =  Cu
KRAY_CHAR       =  K-ALPHA
WAVE_LENGTH1    =  1.54059
WAVE_LENGTH2    =  1.54441
THICKNESS       =  0, 0.000000
MU              =  0, 0.000000
SCAN_MODE       =  2Theta/Theta
SPEED_DIM       =  sec./Step
XUNIT           =  deg.
YUNIT           =  counts
SCALE_MODE      =  1
REP_COUNT       =  0
PK_COUNT        =  0
STD_MATERIAL    =  Unknown, Unknown, 0.000000, 0.000000, 0.000000, 0.000000, 0.000000, 0.000000
LATT_CONS       =  0, Unknown, Unknown, 0.000000, 0.000000, 0.000000, 0.000000, 0.000000, 0.000000
SEC_COUNT       =  1
TSPEC_SIZE      =  0
EXTRA_SIZE      =  0
MEMO            =  Sample-1-1
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#Attenuator_coefficient=1.000000
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10.1000  54.0000
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10.1400  37.0000
10.1600  49.0000
10.1800  47.0000
10.2000  37.0000
10.2200  50.0000
10.2400  46.0000
10.2600  60.0000
10.2800  58.0000
10.3000  39.0000
10.3200  50.0000
10.3400  46.0000
10.3600  43.0000
10.3800  57.0000
10.4000  41.0000
10.4200  49.0000
10.4400  52.0000
10.4600  31.0000
10.4800  52.0000
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10.5200  47.0000
10.5400  51.0000
10.5600  45.0000
10.5800  42.0000
10.6000  46.0000
10.6200  45.0000
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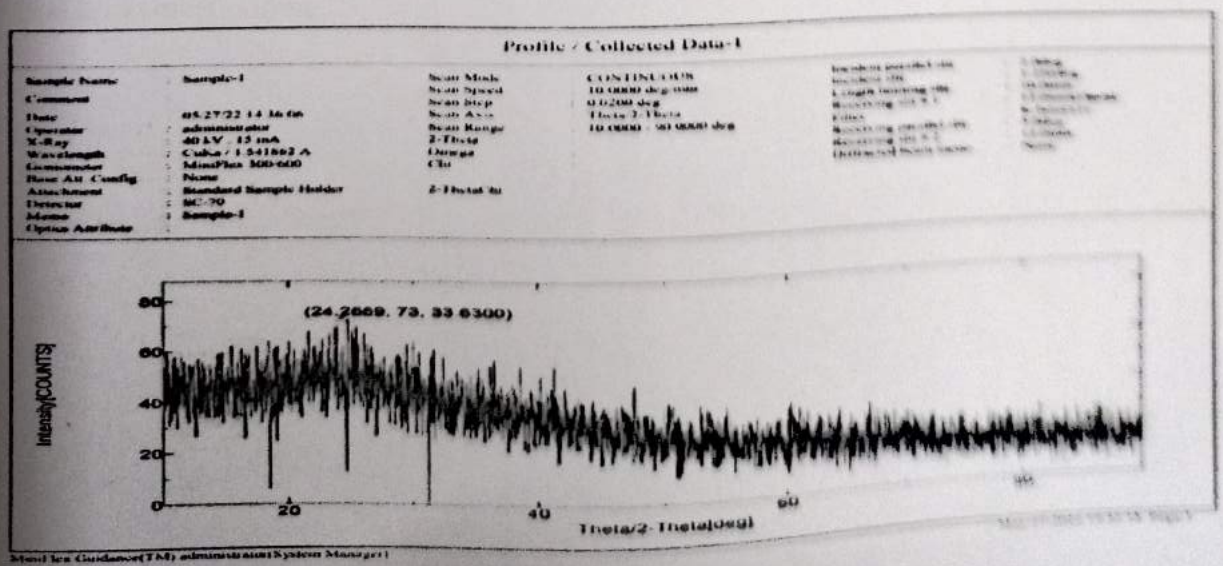

3. CONCLUSION

A degradable nanocomposite hydrogel has been synthesized using laponite XLG asnanomaterial, and evaluated for mobility control and fracture-plugging applications in mature reservoirs. It was observed that gel strength increased with increasing nanomaterial concentration. It was also observed that longterm thermal stability of hydrogels was directly proportional to nanomaterial concentration. The higher the nanomaterial concentration, the longer the thermal stability of the hydrogels. Post-degraded nanocomposite hydrogel viscosity measurements were 26 times higher than those of hydrogels without nanomaterial. After degradation, LXLG nanocomposite hydrogel had a post-degradation viscosity (4437 cp), whereas hydrogel without nanomaterial had a post-degradation viscosity of 170 cp. LXLG nanocomposite hydrogel can be used for conformance control applications because they have higher strengths and longterm thermal resistance than hydrogels without nanomaterial.For secondary polymer flooding-mobility control applications, we recommend using degradable LXLG nanocomposite hydrogels, since they have a high post-degradation viscosity under anaerobic conditions. This product, when injected into the reservoir, will initially act as a conformance control agent by plugging water thief zones and channels, thereby directing injected water to sweep out oil from low permeability oil rich zones. After an extended time period, this product degrades into a highly viscous polymer solution which then moves deeper into the reservoir, mixes with flood water and increases its viscosity, and by so doing improves water and polymer flooding processes by increasing water sweep efficiency, thereby enhancing oil production. Thus, with a single product, we can

- correct reservoir heterogeneity and improve conformance control.
- improve water flooding process.
- Boostpolymerflooding.