

# **“INTEGRATED SOLVENT RECYCLE SYSTEM FOR EXTRACTING INDEGRIENTS OF BIOMASS”**

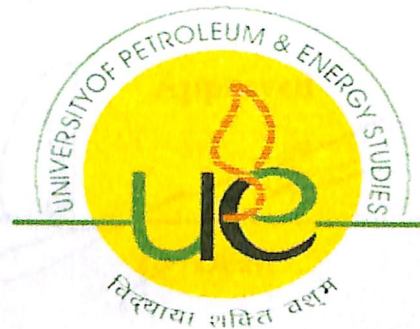
**A Thesis Submitted in Partial Fulfillment of the Requirement for the degree of**

**BACHELOR OF TECHNOLOGY  
(APPLIED PETROLEUM ENGINEERING)**

**By**

**MOHIT CHAUHAN (R240207034)  
MOHIT SINGH (R240207035)  
ROHIT CHAWLA (R240207046)**

**Under the Guidance of  
Dr. R.P.BADONI  
(Distinguished Professor)**



**College of Engineering**

**University of Petroleum and Energy Studies**

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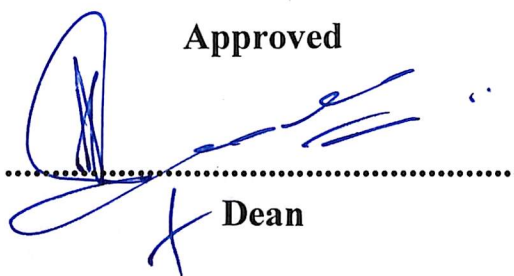
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**College of Engineering**

**University of Petroleum and Energy Studies**

**Dehradun**

**May, 2011**

## CERTIFICATE

This is to certify that Mohit Chauhan, Mohit Singh and Rohit Chawla have done their major project at University of Petroleum & Energy Studies during the fourth year of their academics.

The thesis titled **“INTEGRATED SOLVENT RECYCLE SYSTEM FOR EXTRACTING INDEGRIENTS OF BIOMASS”** is original work and has been carried out under supervision and has not been submitted elsewhere for a degree .



**Dr. R.P. BADONI**  
**Distinguished Professor,**  
**Chemical Engg. Department,**  
**UPES, Dehra Dun.**

## **ACKNOWLEDGEMENT**

Major Project in the final year is an indispensable part of any engineering curriculum. It provides the students with an opportunity to gain experience on the practical application of their technical knowledge and to study the various theoretical aspects as well.

We would like to thank our Project Guide **Dr.R.P.BADONI**, Distinguished Professor, Chemical Engg. Department, UPES, Dehra Dun for giving us this opportunity to work under his guidance on this project and for also providing us with all the necessary information. His technical help and goal oriented approach has been unique and a stepping stone towards the successful completion of the project.

We would also like to express our gratitude towards our parents for their kind co-operation and encouragement which helped us in completion of this project.

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**Mohit Chauhan**

Date-

**Mohit Singh**

**Rohit Chawla**

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## EXECUTIVE SUMMARY

In recent years biofuels have been look forward as a substitute for fossil fuel. Biofuels are obtained from biomass, like Jatropha, Castor which can be cultivated as crops. Biodiesel can be produced by Jatropha or Castor oil by transesterification process. Jatropha oil can be extracted from different methods and by using different solvents. In our project we have used carbon tetrachloride and methanol as solvents to extract active components from biomass and set up an apparatus named Integrated Solvent Recycle System. Solvent used is continuously recycled in the equipment by siphon affect. Crushed and dried form of jatropha seed sample is placed in the apparatus whose components are extracted from carbon tetrachloride and methanol one by one. The solvent is then regenerated using ASTM distillation. The extracted component is analyzed using mass balance and techniques like gas chromatography. The whole process is done again for Castor seed. Both solvents efficiency is compared with each other and also with other solvents used before by different researchers. Both biomass are also compared based upon their lipid content. Percentage recovery and quantitative analysis of extracted product is our most important aspect.

Today the primary energy source is oil. The world consumes 77 million barrels of petroleum daily, which makes 26 billion barrels annually. Oil accounts for 40% of all energy use, therefore continued access to this resource or an equivalent or an improved replacement is essential to continue the world as we know it. Alternate sources can be of several types like nuclear energy, tidal energy, geothermal energy etc. Biofuel which is derived from biomass is one such alternate source. It has been looked upon as substitute for petroleum since it first discovered in late 80s and much research has been done to make it a competitive fuel.

Biofuels are mainly produced from biomass feed stocks or as a by-product from the industrial processing of agricultural or food products, or from the recovery and reprocessing of products such as cooking and vegetable oil. Biofuel contains no petroleum, but it can be blended at any level with petroleum fuel to create a biofuel blend. It can be used in conventional equipment or diesel engine with no major modification. Biofuel is very simple to use, biodegradable, non-toxic and nearly free of Sulphur and aroma.

Biomass has been looked forward since a long time for its components which can be used as fuel. But the exact or most appropriate technologies for extracting its components are in research phase. Also, composition of its ingredients is determined by various methods. In this context, Integrated Solvent Recycle System is an extraction process. In this method, compounds are absorbed in solvent according to their affinity towards the solvent.

### **Basis of Integrated solvent recycle system**

The main concept of Integrated Solvent Recycle System is leaching. In leaching there is a removal of materials by dissolving them away from solids and the process is known as extraction. Material which is insoluble can be separated by gravity or vacuum filtration, and soluble material is 'extracted' into the solvent. To separate complex mixtures into groups a sequence of solvents, of varying polarity or pH, can be used. The filtered solution can be used as a solution for further analysis. For example, to be injected into the GC system. The solvent can either be evaporated to recover the solute(s) in powder or crystalline form or regenerated using technique like distillation. The solvent can be hydrophilic or hydrophobic, acidic, neutral or basic.

In our project leaching plays an important role for separation of oil content from biomass like jatropha and Castor where biomass is solid phase and solvent is used as liquid phase.

## **Traditional Methods**

Traditional methods used in the rural and developing areas for extracting the oils are simple and the oil is extracted by hand using simple equipment.

In this process oil is extracted at high pressure which is called as hot oil extraction method. As in jatropha oil or in Castor oil operating temperature can be regulated so it is extracted using the hot oil extraction method.

Then the cold oil extraction method is easy to extract the oil from the hot oil extraction since the oil flows more easily due to higher viscosity. The remained press cake after extracting the oil also have less oil content which might be 3 to 7 % approximately. The above two reasons make the oil press method very interesting.

During the oil extraction method lots of stuffing of the seeds are converted into gum like substances and some non organic substances. These are unwanted products and so they have to be refined:

## **Objectives of Biomass Analysis**

Primary concern of the study is to evaluate and compare different solvents that can effectively and efficiently extract oil from biomass. Our study comprises of mainly four experiments to understand existing biofuel components .Different solvents are compared on the basis of oil recovery from jatropha and castor seeds. Jatropha and Castor seeds are also compared based upon there lipid content. Solvents are also compared based on their polarity and how much each (polar and non polar) solvent can extract oil from biomass. Percentage of moisture content in seeds and volatilities of solvents are main prerequisites to study.

**What is Leaching ?**

The main concept of Integrated Solvent Recycle System is leaching. In leaching there is a removal of materials by dissolving them away from solids. The chemical process industries use leaching but the process is usually called extraction, and organic solvents are often used. It is also known as solid-liquid extraction. The principle for the solid-liquid extraction is that the soluble compounds of a solid matter, existing of an inert matrix and the active agent, are extracted by a solvent. The extract can be included in the extraction matter in solid or liquid form. It can be included in cells like oil in oil seeds or as fine dispersion on the solid matter like caffeine in coffee. The practice and theory of leaching are well-developed because for many years leaching has been used to separate metals from their ores and to extract sugar from sugar beets. In our process we have separated lipids from their seeds

Following points are necessary for an economic extraction process:

The extraction matter has to be prepared in this way that the extract can be dissolved by the solvent in short time. This is achieved by grinding, milling or rolling.

Only the desired extract has to be solved and extracted. This is achieved by the selectivity of the solvent and the temperature.

Separation of the solvent from as well extract solution as extraction residual has to be economically.

A total solid-liquid extraction process includes the preparation of the extraction material, separation and recovery of the solvent from extract and separation and recovery of solvent from extraction residual.

**Solvent**

A solvent is a liquid, solid, or gas that dissolves another solid, liquid, or gaseous solute, resulting in a solution that is soluble in a certain volume of solvent at a specified temperature. Common uses for organic solvents are in dry cleaning, as nail polish removers, as a paint thinner and glue solvents, in spot removers, in detergents, to extract different component and in chemical synthesis. The use of inorganic solvents (other than H<sub>2</sub>O) is typically limited to research chemistry and some technological processes. Selection criteria for solvent are very important.

**Selection of the solvent**

- The solvent for extraction has to extract the active agent from a mixture.
- Selectivity:: Only the active agent has to be extracted and which means that a high selectivity is required.

- Miscibility::To achieve simple regeneration of the solvent the miscibility of solvent and primary solvent has to be low.
- Difference in density:: After extraction the two phases have to be separated in a separator and for this a high difference in density is positive.
- Recovery: :The solvent has to be separated from the extract phase easily to produce solvent free active agents. Corrosion: If the solvent is corrosive prices for construction increase.
- No or low toxicity
- Low price
- Flame temperature:: 25 °C higher than operating temperature
- Vapor pressure:: To prevent loss of solvent by evaporation a low vapor pressure at operating temperature is required.
- Viscosity:: A low viscosity of the solvent leads to low pressure drop and good heat and mass transfer.
- Chemical and thermal stability
- Regeneration of the solvent :-
- For all extraction processes the regeneration by further separation processes is necessary. By this way pure products are produced and the solvent can be recycled in the extraction process.
- Following possibilities for separating of the solvent are available:
- Rectification: The most common method
- Evaporation:: The evaporation of the solvent is used if the active agent is very high volatile. The solvent should have a low boiling temperature and a low heat of evaporation.
- Crystallization:: Cooling the solvent results in crossing the solubility and the active agent falls out and can be separated by mechanically separation processes.
- Extraction:: A further extraction step with another solvent can be used to separate the active agent from the first solvent. But the by this way produced extract has to be separated once again.

- **Boiling Point:** An important property of solvents is boiling point. This also determines the speed of evaporation. Small amounts of low-boiling solvents like dichloromethane, diethyl ether, or acetone will evaporate in seconds at room temperature, while high-boiling solvents like water or dimethyl sulfoxide need higher temperatures, an air flow, or the application of vacuum for fast evaporation.

Solvent		Boiling point
polar	Methanol	338 K
	Ethanol	351.1K
Non Polar	Hexane	342 K
	Carbon Tetrachloride	349.75 K

Table:1

- **Density:** Most organic solvents have a lower density than water, which means they are lighter and will form a separate layer on top of water. An important exception: most of the halogenated solvents like dichloromethane or chloroform will sink to the bottom of a container, leaving water as the top layer. This is important to remember when partitioning compounds between solvents and water in a separatory funnel during chemical syntheses. Often, specific gravity is cited in place of density. Specific gravity is defined as the density of the solvent divided by the density of water at the same temperature. As such, specific gravity is a unit less value. It readily communicates whether a water-insoluble solvent will float ( $SG < 1.0$ ) or sink ( $SG > 1.0$ ) when mixed with water.

## Solvent classifications

Solvents can be broadly classified into two categories: polar and non-polar. Generally, the dielectric constant of the solvent provides a rough measure of a solvent's polarity

## **Solvent Used**

We have used carbon tetrachloride and methanol as solvent. Carbon tetrachloride is non polar while methanol is polar.

## **Carbon Tetrachloride**

Carbon tetrachloride is a clear liquid that evaporates very easily. Carbon tetrachloride does not easily burn. Carbon tetrachloride has a sweet odor, and most people can begin to smell it in air when the concentration reaches 10 parts carbon tetrachloride per million parts of air (ppm). It produces poisonous vapours.

## **Methanol**

Methanol, also known as methyl alcohol is a chemical with the formula  $\text{CH}_3\text{OH}$ . It is the simplest alcohol, and is a light, volatile, colorless, flammable liquid with a distinctive odor very similar to, but slightly sweeter than, ethanol. At room temperature, it is a polar liquid, and is used as an antifreeze, solvent, fuel, and as a denaturant for ethanol. It is also used for producing biodiesel via transesterification reaction.

## **Commonly used solvent for biomass extraction**

Most commonly used solvents are Hexane and ethanol. Hexane give a recovery of 37% in case of jatropha and 40% in case of castor while ethanol gives recovery of 31% in case of jatropha and 36% in case of castor. Hexane is non polar while ethanol is polar due to its dipole moment.

## **Biomass**

All plants fix solar energy through the process of photosynthesis to produce biomass. The biomass thus produced passes through various cycles producing different forms of energy sources. For example, it is used as fodder for animals that in turn produce dung, agricultural waste for cooking. Currently availability of biomass in India is estimated at about 120-150 million MT per annum covering agricultural and forestry residues corresponding to a potential of 16,000 MW.

Biomass is an important source of energy accounting for about one third of the total fuel used in our country and in about 90% of the rural households. The widespread use of biomass is for household cooking and heating. Agricultural waste, wood, charcoal or dried dung are few of the types of biomass used. Biomasses are of several types like jatropha seeds, Jatropha, castor seeds etc. Biomass to be used in our project i.e. Integrated Solvent Recycle System is Jatropha seeds.

Jatropha (scientific name-Jatropha curcas ) is a drought-resistant perennial, growing well in marginal/poor soil. It is easy to cultivate, grows relatively quickly and lives, can produce seeds for 50 years. Jatropha the wonder plant produces seeds with 37% oil content. The oil can be combusted as fuel without being refined. This oil when burns give a clear smoke-free flame, tested successfully under specific conditions as fuel for simple diesel engine. Press cake, a by-product, is a good organic fertilizer, oil contains also insecticide. It is found to be growing in many parts of the country, rugged in nature and can survive with minimum inputs and easy to propagate.

### **Jatropha curcas**

Among most of the biomass Jatropha curcas has been identified as the most suitable oil seed bearing plant due to its various favorable attributes like short gestation period, adaptability in wide range agro-climatic conditions, hardy nature, quality of oil high oil recovery and etc. It can be planted on drought lands through Joint Forest Management (JFM), road sides, farmer's field boundaries, fallow lands and as agro-forestry crop

both sides of the railway tracks,. Its normal height is of 5 metres. Jatropha offers the following advantages. It is easy to establish; it require little care and grows quickly. It can grow in poor soils, in wastelands except waterlogged areas and flood. Reclamation of degraded land and wasteland is possible through its plantation. In fertile land, it gives higher yields. Hence, it promotes ensuring energy security besides rural economy besides. It is suitable for preventing soil erosion. It improves the soil fertility throughout the life cycle. It possesses medicinal value as well as other multiple uses.

Jatropha curcas is found in almost all the states in India and is generally grown, as a fence for protection against livestock as goats or cattle do not eat it since it is non-edible. It is a tropical species and can be grown well in subtropical conditions. It grows almost everywhere, even on gravely, alkaline soils, sandy, and acidic soils. Jatropha can easily be propagated on mass scale both stem cuttings by as well as seed. Humid and hot weather is preferred for good germination of seed. Cutting or Seeds can be directly planted in the main field.

The economic life of Jatropha is 30-40 years. The plant survives upto 50 years if root zone does not come in contact with rising water table and continues for longer time.

Jatropha plantation should not be taken as profitable cash crop if the value of by-products and other uses are not considered. Besides, raising of intercrops along with oil, other potentially high



value products such as seed cake and glycerin to make it more attractive should be effectively marketed and the environmental and socio-economic returns such as wasteland reclamation need to be adequately accounted .



**Figure 1. The Jatropha Plant**



**Figure 2. Dry Jatropha fruits**



**Figure 3. Jatropha seeds**



**Figure 4. Jatropha kernels**

### **Castor seeds:-**

The castor oil plant, Its seed is the castor bean which, despite its name, is not a true bean. Castor is indigenous to the Mediterranean Basin, Eastern Africa, and India, but is widespread throughout tropical regions (and widely grown elsewhere as an ornamental plant).

Castor seed is the source of castor oil, which has a wide variety of uses. The seeds contain between 40% and 60% oil that is rich in triglycerides, mainly ricinolein. The seed contains a toxin named ricin which is present in lower concentrations throughout the plant.

Castor oil dissolves easily in alcohol, chloroform, carbon sulfide, ether, glacial acetic acid, and benzene. Besides being used as a laxative, castor oil is widely used in the industrial field because of its many properties. In the textile industry, castor oil is used for moisturizing and removal of grease in fabrics, and for the manufacturing of waterproof fabrics. In the steel industry, it is used in cutting oils and lubricants for steel lamination at high temperatures and it is also used in other liquids that are necessary for steel work. The automotive industry uses castor oil for the production of high performance motor oil and braking fluids. Moreover, it is also employed as as oftener in the tanning industry and in the production of fluids for hydraulic devices, artificial leather, varnish, paint, linoleum, insulators, powder, fatty acids, enzymes, as a moisturizer for stationary and insecticides; additionally it can be used as a raw material for the fabrication of plastics.





**Figure 5: Castor Plant**



**Figure 6: Castor seeds**

## **Biofuel**

Biofuels have been around as long as cars have. At the start of the 20th century, Henry Ford planned to fuel his Model Ts with ethanol, and early diesel engines were shown to run on peanut oil.

Biofuels are predominantly produced from biomass feed stocks or as a by-product from the industrial processing of agricultural or food products, or from the recovery and reprocessing of products such as cooking and vegetable oil. Biofuel contains no petroleum, but it can be blended at any level with petroleum fuel to create a biofuel blend. It can be used in conventional heating equipment or diesel engine with no major modification. Biofuel is simple to use, biodegradable, non-toxic and essentially free of Sulphur and aroma.

But discoveries of huge petroleum deposits kept gasoline and diesel cheap for decades, and biofuels were largely forgotten. However, with the recent rise in oil prices, along with growing concern about global warming caused by carbon dioxide emissions, biofuels have been regaining popularity.

Gasoline and diesel are actually ancient biofuels. But they are known as fossil fuels because they are made from decomposed plants and animals that have been buried in the ground for millions of years. Biofuels are similar, except that they're made from plants grown today.

Much of the gasoline in the United States is blended with a biofuel—ethanol. This is the same stuff as in alcoholic drinks, except that it's made from corn that has been heavily processed. There are various ways of making biofuels, but they generally use chemical reactions, fermentation, and heat to break down the starches, sugars, and other molecules in plants. The leftover products are then refined to produce a fuel that cars can use.

Countries around the world are using various kinds of biofuels. For decades, Brazil has turned sugarcane into ethanol, and some cars there can run on pure ethanol rather than as additive to fossil fuels. And biodiesel—a diesel-like fuel commonly made from palm oil—is generally available in Europe.

On the face of it, biofuels look like a great solution. Cars are a major source of atmospheric carbon dioxide, the main greenhouse gas that causes global warming. But since plants absorb carbon dioxide as they grow, crops grown for biofuels should suck up about as much carbon dioxide as comes out of the tailpipes of cars that burn these fuels. And unlike underground oil reserves, biofuels are a renewable resource since we can always grow more crops to turn into fuel.

Unfortunately, it's not so simple. The process of growing the crops, making fertilizers and pesticides, and processing the plants into fuel consumes a lot of energy. It's so much energy that there is debate about whether ethanol from corn actually provides more energy than is required to grow and process it. Also, because much of the energy used in production comes from coal and natural gas, biofuels don't replace as much oil as they use.

For the future, many think a better way of making biofuels will be from grasses and saplings, which contain more cellulose. Cellulose is the tough material that makes up plants' cell walls, and most of the weight of a plant is cellulose. If cellulose can be turned into biofuel, it could be more efficient than current biofuels, and emit less carbon dioxide.

### **What is Transesterification ?**

The process of converting biomass oil into biodiesel fuel . Transesterification refers to a reaction between an ester of one alcohol and a second alcohol to form an ester of the second alcohol and an alcohol from the original ester, as that of methyl acetate and ethyl alcohol to form ethyl acetate and methyl alcohol. Chemically, transesterification means taking a triglyceride molecule or a complex fatty acid, neutralizing the free fatty acids, removing the glycerin and creating an alcohol ester. This is accomplished by mixing sodium hydroxide with methanol to make sodium methoxide. This liquid is then mixed into vegetable oil. The entire mixture then settles. Glycerin is on the bottom and methyl esters, or biodiesel, is left on top. The glycerin can be used to make and the methyl esters is washed and filtered.

### **Liquid – Liquid Extraction**

Liquid-liquid extraction, also known as solvent extraction and partitioning, is a method to separate compounds based on their relative solubility's in two different immiscible liquids, usually an organic solvent and water. It is an extraction of a substance from one liquid phase into another liquid phase. Liquid-liquid extraction is a basic technique in chemical laboratories, where it is performed using a separatory funnel. This type of process is commonly performed after a chemical reaction as part of the work-up.

### **Simple distillation**

In simple distillation all the hot vapors produced are immediately channeled into a condenser that cools and condenses the vapors. Therefore, the distillate will not be pure - its composition will be identical to the composition of the vapors at the given temperature and pressure, and can be computed from Raoult's law.

As a result, simple distillation is usually used only to separate liquids whose boiling points differ greatly or to separate liquids from in volatile solids or oils. For these cases, the vapor pressures of the components are usually sufficiently different that Raoult's law may be neglected due to the insignificant contribution of the less volatile component. In this case, the distillate may be sufficiently pure for its intended purpose.

**Experiment****METHODOLOGY**

Isolation of compounds in a pure state from natural sources is the most important, yet can be a difficult and time-consuming. In this context, Integrated Solvent Recycle System is a solid-liquid integrated extraction process. In this method, compounds are absorbed in solvent according to their affinity towards the solvent. The basic procedure calls for a solid sample to be placed in a porous container and allowing condensed solvent to extract continuously. The apparatus will provide automatic recycle and regeneration of given solvent which is used to extract the constituent of biomass. Biomass seeds is grind and meshed up and kept in a thimble. The Solvent vapor generated by gently heating the reservoir, condenses and is allowed to drip back onto the thimble. The liquid condensate that drips onto the sample performs the extraction which then passes through the container and back into the reservoir. The cycle is repeated continuously and can be sustained as long as needed. As it progresses the species of interest is concentrated in the reservoir. For the analysis purpose the extract phase which contains two layers of liquid upper layer is mainly biofuel and lower one is solvent. Separatory funnel is used separate two immiscible liquid while distillation is used to separate solvent and biofuel more effectively and efficiently.

Integrated solvent recycle process can be considered in three parts:

1. Diffusion of the solvent through the pores of the solid
2. The diffused solvent dissolves the solutes (i.e. transfer the solute to the liquid phase).
3. Transfer of the solution from porous solid to the main bulk of the solution



## SCHEMATIC DIAGRAM

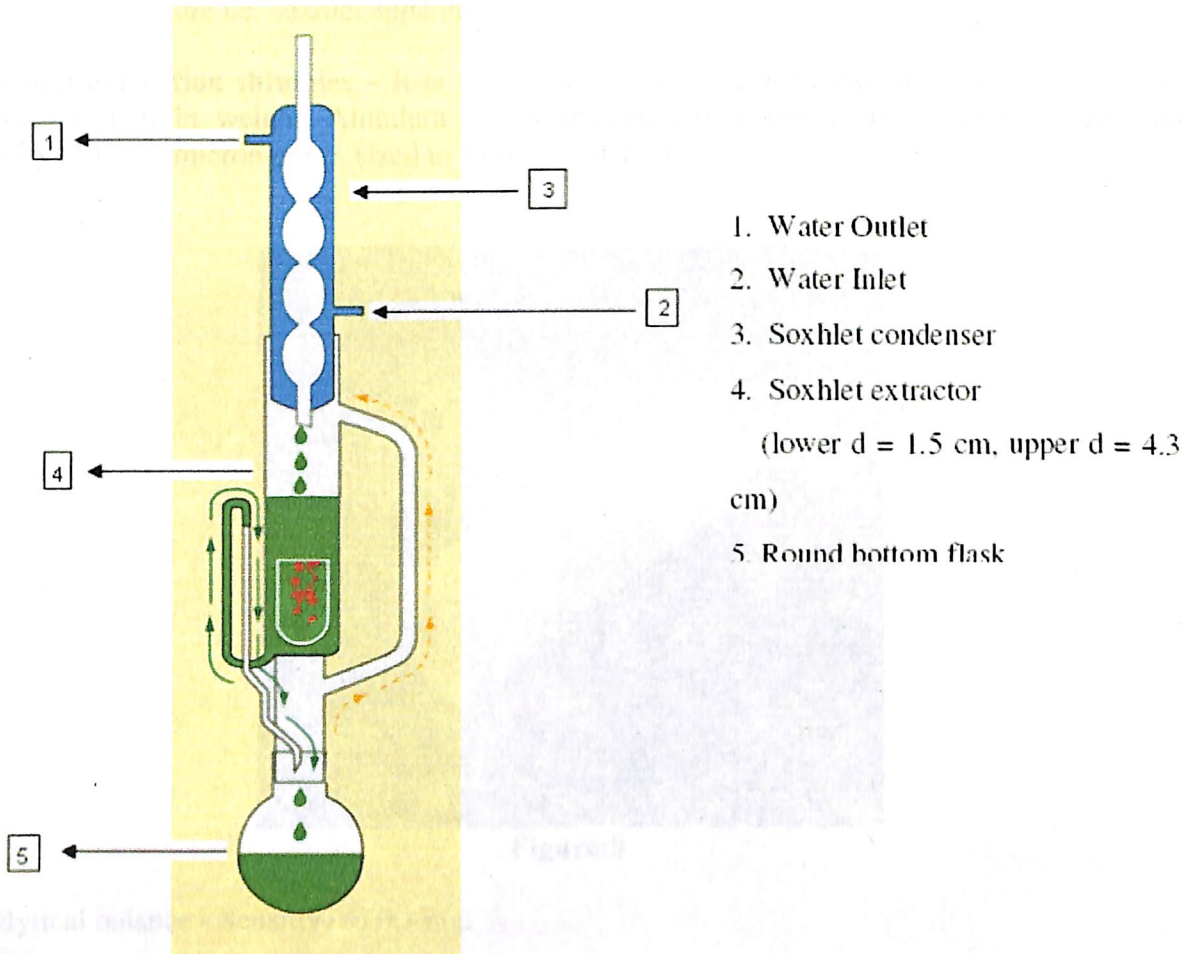


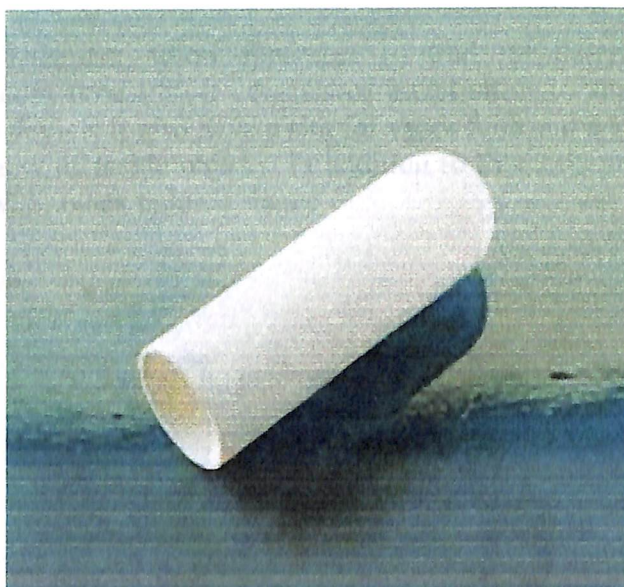
Figure:7



### **Description of Apparatus Used :-**

**Extraction apparatus** - Thimble of suitable size for containing the sample (30 grams) and a 250 mL collection flask is required for the conventional extraction procedure. An automated extraction apparatus with circulating water bath and associated glassware is required for the automated procedure i.e. soxhlet apparatus.

**Alundum extraction thimbles** – It is for all kinds of extractions, except strong alkalis. They remain constant in weight. Alundum will withstand temperatures up to 1450° C. Medium porosity (10 – 15 micron pore), sized to fit the extractor tube

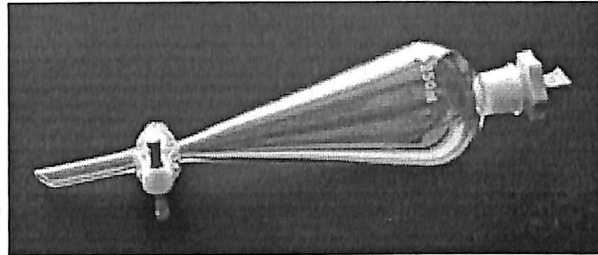


**Figure:8**

**Analytical balance** - Sensitive to 0.1 mg.

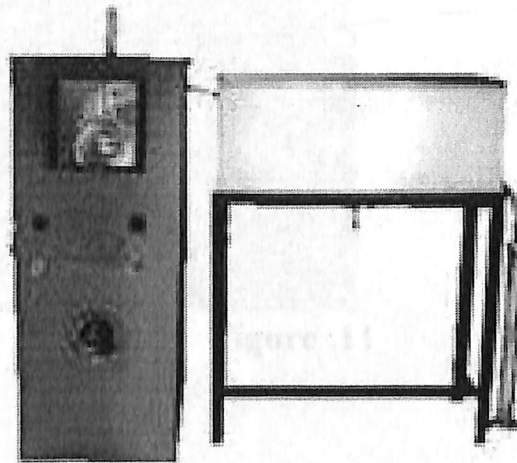
**Vacuum oven or drying oven** - Vacuum oven should be controllable to a temperature of 40 degree celcius and vacuum of between 75 to 100 torr. If drying oven is used in place of the vacuum oven, the drying oven must be able to maintain 45+ 2EC.

**Separatory funnel** - A separatory funnel, also known as separating funnel, is a piece of laboratory glassware used in liquid-liquid extractions to separate the components of a mixture between two immiscible solvent phases of different densities.



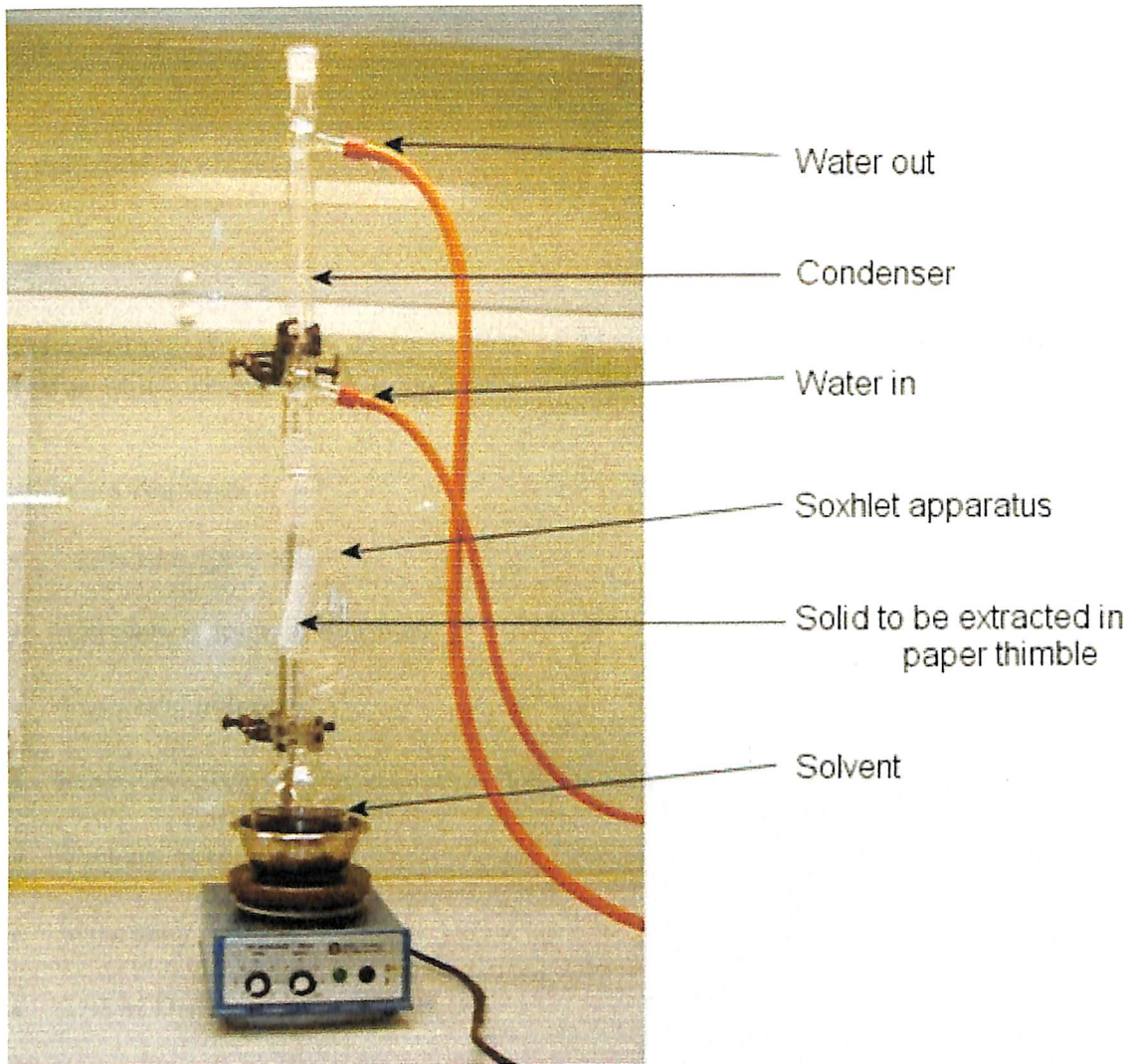
**Figure:9**

**ASTM Distillation** - This distillation apparatus is used for determination of distillation characteristics of petroleum products. It consists of metal sheet to support conical flask with geared height adjustable device. It also have a slot for vapor tube and a glass window is provided in front side for clear view of inside objects. In addition to this, this product also have cooling bath or condenser with M.S. black painted stand.



**Figure:10**

## Experimental Set –Up



**Figure :11**

## **Experiment : 1**

### **Object**

The objective of this experiment is to know the percentage of oil recovery from jatropha seeds by using methanol as a solvent .

### **SCOPE**

This test method covers the determination of Methanol soluble extractives, expressed as the percentage of the oven-dried jatropha seeds.

### **Equipments required**

- Extraction apparatus
- Alundum extraction thimbles
- Analytical balance
- Rotary evaporator with vacuum and water bath
- Vacuum oven or drying.
- Separatory funnel
- ASTM Distillation Apparatus

### **Reagents and Materials**

- Methanol
- Jatropha seeds
- Boiling chips.
- Glass wool.

## Procedure

1. Weigh 35 grams of jatropha seeds.
2. Crush and dry them in an oven for 25 minutes at 95°C and determine the moisture content. Place a plug of glass wool on top of the sample to prevent sample loss during the extraction.
3. Wrap the sample in alundum extraction thimbles, do not overfill and leave at least a 10 mm gap between the top of the filter paper and the sample.
4. Place boiling chips into a dry, clean round bottom flask. Weigh the container, with chips, and record as the tare weight of the container.
5. Pour 150 ml Methanol into the dry, clean round bottom flask. Insert Alundum extraction thimbles & sample into the extractor, assemble the Soxhlet apparatus and start extraction.
6. Heat at reflux for 8 hours. Periodically check the reflux rate and adjust the heating rate to give four to five solvent exchanges per hour in the thimble.
7. When the extraction stops, remove sample carefully transfer it into an evaporating dish. Weigh the sample. Dry it in an oven for 10 minutes at 75°C. Weigh the sample the next day again.
8. Weigh the round bottom flask with Methanol
9. Transfer the sample to a separatory funnel two separate two immiscible liquid.
10. After all of the visible solvent is removed by the separatory funnel use ASTM distillation for the maximum recovery.
11. Weigh the recovered Methanol.
12. Let the round bottom flask with recovered oil dry for 48 hrs. Weigh the container after the process is completed

Note: Samples for total solids determination must be weighed out at the same time as the samples for the extractives determination. If this determination is done at a later time, an error in the calculation of the amount of extractives will be introduced, since the moisture content of a biomass sample can change rapidly when exposed to air.

## Result

Replications	Sample weight	Dry sample content	Moisture content	% of moisture content	Volume Extracted	Solvent Volume	Lipid Mass	Percent Lipids
1	35 g	32.2 g	2.8 g	8 %	145 ml	150 ml	10 g	31 %
2	40 g	36.87 g	3.13 g	7.825 %	146 ml	150ml	11.8 g	32 %

**Table:2**

## HSE Considerations and Hazards

- No smoking or open flame in handling areas.
- Refer to MSDS of Methanol before using Methanol.

## **Experiment : 2**

### **Object**

The objective of this experiment is to know the percentage of oil recovery from jatropha seeds by using Carbon tetrachloride as a solvent.

### **SCOPE**

This test method covers the determination of Carbon tetrachloride soluble extractives, expressed as the percentage of the oven-dried jatropha seeds.

### **Equipments required**

- Extraction apparatus
- Alundum extraction thimbles
- Analytical balance
- Rotary evaporator with vacuum and water bath
- Vacuum oven or drying.
- Separatory funnel
- ASTM Distillation Apparatus

### **Reagents and Materials**

- Carbon tetrachloride
- Jatropha seeds
- Boiling chips.
- Glass wool.



## Procedure

1. Weigh 35 grams of jatropha seeds.
2. Crush and dry them in an oven for 25 minutes at 95°C and determine the moisture content. Place a plug of glass wool on top of the sample to prevent sample loss during the extraction.
3. Wrap the sample in alundum extraction thimbles, do not overfill and leave at least a 10 mm gap between the top of the filter paper and the sample.
4. Place boiling chips into a dry, clean round bottom flask. Weigh the container, with chips, and record as the tare weight of the container.
5. Pour 150 ml Carbon tetrachloride into the dry, clean round bottom flask. Insert Alundum extraction thimbles & sample into the extractor, assemble the Soxhlet apparatus and start extraction and continue the process till twenty siphons.
6. Heat at reflux for 8 hours. Periodically check the reflux rate and adjust the heating rate to give four to five solvent exchanges per hour in the thimble.
7. When the extraction stops, remove sample carefully transfer it into an evaporating dish. Weigh the sample. Dry it in an oven for 10 minutes at 75°C. Weigh the sample the next day again.
8. Weigh the round bottom flask with Carbon tetrachloride .
9. Transfer the sample to a separatory funnel two separate two immiscible liquid.
10. After all of the visible solvent is removed by the separatory funnel use ASTM distillation for the maximum recovery.
11. Weigh the recovered Carbon tetrachloride.
12. Let the round bottom flask with recovered oil dry for 48 hrs. Weigh the container after the process is completed.



**Result:-**

Rep licat ions	Sample weight	Dry sample content	Moisture content	% of moisture content	Volume Extracte d	Solvent Volume	Lipid Mass	Percent Lipids
1	35 g	32.2 g	2.8 g	8 %	145 ml	150 ml	13.20g	41 %
2	40 g	36.87 g	3.13 g	7.825 %	146 ml	150ml	14.35g	39 %

**Table:3**

**HSE Considerations and Hazards**

- Avoid heat, flames, sparks and other sources of ignition. Containers may rupture or explode if exposed to heat
- Refer to MSDS of  $\text{CCl}_4$  before using  $\text{CCL}_4$ .

## **Experiment : 3**

### **Objective:**

The objective of this experiment is to know the percentage of oil recovery from castor seeds by using methanol as a solvent .

### **SCOPE**

This test method covers the determination of Methanol soluble extractives, expressed as the percentage of the oven-dried castor seeds.

### **Equipments required**

- Extraction apparatus
- Alundum extraction thimbles
- Analytical balance
- Rotary evaporator with vacuum and water bath
- Vacuum oven or drying.
- Separatory funnel
- ASTM Distillation Apparatus

### **Reagents and Materials**

- Methanol
- Castor seeds
- Boiling chips.
- Glass wool.

## Procedure

1. Weigh 35 grams of castor seeds.
2. Crush and dry them in an oven for 25 minutes at 110°C and determine the moisture content. Place a plug of glass wool on top of the sample to prevent sample loss during the extraction.
3. Wrap the sample in alundum extraction thimbles, do not overfill and leave at least a 10 mm gap between the top of the filter paper and the sample.
4. Place boiling chips into a dry, clean round bottom flask. Weigh the container, with chips, and record as the tare weight of the container.
5. Pour 150 ml Methanol into the dry, clean round bottom flask. Insert Alundum extraction thimbles & sample into the extractor, assemble the Soxhlet apparatus and start extraction.
6. Heat at reflux for 12 hours. Periodically check the reflux rate and adjust the heating rate to give three to four solvent exchanges per hour in the thimble.
7. When the extraction stops, remove sample carefully transfer it into an evaporating dish. Weigh the sample. Dry it in an oven for 10 minutes at 85°C. Weigh the sample the next day again.
8. Weigh the round bottom flask with Methanol
9. Transfer the sample to a separatory funnel two separate two immiscible liquid.
10. After all of the visible solvent is removed by the separatory funnel use ASTM distillation for the maximum recovery.
11. Weigh the recovered Methanol.
12. Let the round bottom flask with recovered oil dry for 48 hrs. Weigh the container after the process is completed

## Result

Replications	Sample weight	Dry sample content	Moisture content	% of moisture content	Volume Extracted	Solvent Volume	Lipid Mass	Percent Lipids
1	35 g	33.25 g	1.75 g	5.2%	145 ml	150 ml	12 g	36%
2	40 g	38.2 g	1.8 g	4.7 %	146 ml	150ml	13.2 g	34.5 %

**Table:4**

## HSE Considerations and Hazards

- No smoking or open flame in handling areas.
- Refer to MSDS of Methanol before using Methanol

## **Experiment : 4**

### **Objective**

The objective of this experiment is to know the percentage of oil recovery from castor seeds by using Carbon tetrachloride as a solvent .

### **SCOPE**

This test method covers the determination of Carbon tetrachloride soluble extractives, expressed as the percentage of the oven-dried castor seeds.

### **Equipments required**

- Extraction apparatus
- Alundum extraction thimbles
- Analytical balance
- Rotary evaporator with vacuum and water bath
- Vacuum oven or drying.
- Separatory funnel
- ASTM Distillation Apparatus

### **Reagents and Materials**

- Carbon tetrachloride
- Castor seeds
- Boiling chips.
- Glass wool.

## Procedure

1. Weigh 35 grams of castor seeds.
2. Crush and dry them in an oven for 25 minutes at 110°C and determine the moisture content. Place a plug of glass wool on top of the sample to prevent sample loss during the extraction.
3. Wrap the sample in alundum extraction thimbles, do not overfill and leave at least a 10 mm gap between the top of the filter paper and the sample.
4. Place boiling chips into a dry, clean round bottom flask. Weigh the container, with chips, and record as the tare weight of the container.
5. Pour 150 ml Carbon tetrachloride into the dry, clean round bottom flask. Insert Alundum extraction thimbles & sample into the extractor, assemble the Soxhlet apparatus and start extraction .
6. Heat at reflux for 12 hours. Periodically check the reflux rate and adjust the heating rate to give three to four solvent exchanges per hour in the thimble.
7. When the extraction stops, remove sample carefully transfer it into an evaporating dish. Weigh the sample. Dry it in an oven for 10 minutes at 85°C. Weigh the sample the next day again.
8. Weigh the round bottom flask with Carbon tetrachloride
9. Transfer the sample to a separatory funnel two separate two immiscible liquid.
10. After all of the visible solvent is removed by the separatory funnel use ASTM distillation for the maximum recovery.
11. Weigh the recovered Carbon tetrachloride.
12. Let the round bottom flask with recovered oil dry for 48 hrs. Weigh the container after the process is completed

## Result

Rep licat ions	Sample weight	Dry sample content	Moisture content	% of moisture content	Volume Extracte d	Solvent Volume	Lipid Mass	Percent Lipids
1	35 g	33.25 g	1.75 g	5.2%	145 ml	150 ml	13.95 g	42%
2	40 g	38.2 g	1.8 g	7.825 %	146 ml	150ml	16.8 g	44 %

**Table:5**

## HSE Considerations and Hazards

- Avoid heat, flames, sparks and other sources of ignition. Containers may rupture or explode if exposed to heat
- Refer to MSDS of Carbon tetrachloride before using Carbon tetrachloride

**1.Environmental, economic, and energetic costs and benefits of biodiesel and ethanol biofuels-Contributed by David Tilman, June 2, 2006**

Negative environmental consequences of fossil fuels and concerns about petroleum supplies have spurred the search for renewable transportation biofuels. To be a viable alternative, a biofuel should provide a net energy gain, have environmental benefits, be economically competitive, and be producible in large quantities without reducing food supplies. We use these criteria to evaluate, through life-cycle accounting, ethanol from corn grain and biodiesel from soybeans. Ethanol yields 25% more energy than the energy invested in its production, whereas biodiesel yields 93% more. Compared with ethanol, biodiesel releases just 1.0%, 8.3%, and 13% of the agricultural nitrogen, phosphorus, and pesticide pollutants, respectively, per net energy gain. Relative to the fossil fuels they displace, greenhouse gas emissions are reduced 12% by the production and combustion of ethanol and 41% by biodiesel. Biodiesel also releases less air pollutants per net energy gain than ethanol. These advantages of biodiesel over ethanol come from lower agricultural inputs and more efficient conversion of feedstocks to fuel. Neither biofuel can replace much petroleum without impacting food supplies. Even dedicating all U.S. corn and soybean production to biofuels would meet only 12% of gasoline demand and 6% of diesel demand. Until recent increases in petroleum prices, high production costs made biofuels unprofitable without subsidies. Biodiesel provides sufficient environmental advantages to merit subsidy. Transportation biofuels such as synfuel hydrocarbons or cellulosic ethanol, if produced from low-input biomass grown on agriculturally marginal land or from waste biomass, could provide much greater supplies and environmental benefits than food-based biofuels.



## **2. The Path Forward for Biofuels and Biomaterials –by Arthur J. Ragauskas, Charlotte K. Williams**

Biomass represents an abundant carbon-neutral renewable resource for the production of bioenergy and biomaterials, and its enhanced use would address several societal needs. Advances in genetics, biotechnology, process chemistry, and engineering are leading to a new manufacturing concept for converting renewable biomass to valuable fuels and products, generally referred to as the biorefinery. The integration of agroenergy crops and biorefinery manufacturing technologies offers the potential for the development of sustainable biopower and biomaterials that will lead to a new manufacturing paradigm.

## **3. How Green Are Biofuels? –by Jbrn P. W. Scharlemann and William F. Laurance**

Global warming and escalating petroleum costs are creating an urgent need to find ecologically friendly fuels. Biofuels—such as ethanol from corn (maize) and sugarcane—have been increasingly heralded as a possible savior (1, 2). But others have argued that biofuels will consume vast swaths of farmland and native habitats, drive up food prices, and result in little reduction in greenhouse-gas emissions (3-5). An innovative study by Zah *et al.* (6), commissioned by the Swiss government, could help to resolve this debate by providing a detailed assessment of the environmental costs and benefits of different transport biofuels.

## **4. An extraction method for measuring soil microbial biomass C-by E.D. Vance, P.C. Brookes and D.S. Jenkinson**

The effects of fumigation on organic C extractable by 0.5 M K<sub>2</sub>SO<sub>4</sub> were examined in a contrasting range of soils.  $E_C$  (the difference between organic C extracted by 0.5 M K<sub>2</sub>SO<sub>4</sub> from fumigated and non-fumigated soil) was about 70% of  $F_C$  (the flush of CO<sub>2</sub>-C caused by fumigation during a 10 day incubation), meaned for ten soils. There was a close relationship between microbial biomass C, measured by fumigation-incubation (from the relationship Biomass C =  $F_C/0.45$ ) and  $E_C$  given by the equation: Biomass C =  $(2.64 \pm 0.060) E_C$  that accounted for 99.2% of the variance in the data. This relationship held over a wide range of soil pH (3.9–8.0).

ATP and microbial biomass N concentrations were measured in four of the soils. The  $(ATP)/(E_C)$  ratios were very similar in the four soils, suggesting that both ATP and the organic C rendered decomposable by CHCl<sub>3</sub> came from the soil microbial biomass. The C:N ratio of the biomass in a strongly acid (pH 4.2) soil was greater (9.4) than in the three less-acid soils (mean C:N ratio 5.1).

We propose that the organic C rendered extractable to 0.5 M  $K_2SO_4$  after a 24 h  $CHCl_3$ -fumigation ( $E_C$ ) comes from the cells of the microbial biomass and can be used to estimate soil microbial biomass C in both neutral and acid soils.

#### **5. Solid-liquid extraction from plants — experimental kinetics and modeling-by E. Simeonov, I. Tsibranska and A. Minchev**

The kinetics and structural changes in the solid were studied for solid-liquid extraction from plants. Two extraction systems were investigated: tobacco leaves (*Nicotiana tabacum* L.)-water, and oak bark (*Quercus frainetto* Ten)-water. The mathematical model of the process, accounting for variable effective diffusivity and porosity, was stated and solved numerically. The solution was checked with experimental kinetics and porosity data to determine the effective diffusion coefficient. A very good correspondence was found between this value and the results obtained with the regular regime and the standard function methods.

#### **6. Biodiesel from Seeds of Jatropha Found in Assam, India by Pranab K. Barua Department of Energy Tezpur University Assam, India**

Biodiesel produced from Jatropha seeds found in Assam, India is comparable with the geodiesel marketed by the Numaligarh Refinery, Assam. Experiments have shown the superiority of Jatropha biodiesel over conventional diesel as far as sulphur content is concerned. Our biodiesel B-100 is of slightly higher density, of slightly lower cetane number, and of slightly lower final boiling point. In addition, it is of low pour point and significantly high flash point. The carbon residue is slightly higher than that of geodiesel. These points lead us to conclude that biodiesel produced from Jatropha seeds found in Assam is of good quality.

#### **7. Phase Separation and Liquid Crystal Self-Assembly in Surfactant-Inorganic-Solvent Systems-by Flor R. Siperstein and Keith E. Gubbins**

The behavior of surfactant-inorganic oxide-solvent systems is studied using lattice Monte Carlo simulations. Under no inorganic condensation conditions, these systems phase separate into a liquid crystal phase that contains mainly surfactant and inorganic oxide, in equilibrium with a solvent-rich phase. In the systems studied, the solvent and the inorganic oxide have favorable interactions with the surfactant head, but the inorganic oxide-surfactant interactions are stronger than the solvent-surfactant interactions, which leads to a phase separation, regardless of the oxide-solvent miscibility. The formation of ordered liquid crystal phases is observed in the phase containing a high surfactant concentration, and the structure of this phase depends on the system composition and strength of the interactions. The formation of hexagonal and lamellar structures at different conditions is in qualitative agreement with experimental evidence on the formation of surfactant-silica liquid crystals and the synthesis of MCM-41 type materials. The effects of temperature and surfactant architecture are also investigated. We show that the increase in surfactant solubility in the solvent-rich phase with temperature can result in a lamellar to hexagonal transformation and that surfactants with small head/tail ratios favor the formation of lamellar phases.

**8. Biodiesel from castor oil: a promising fuel for cold weather by Carmen Leonor Barajas Forero, Department of Hydraulic, Fluids and Thermal Sciences Francisco de Paula Santander University**

*Ricinus communis* is a species that belongs to the Euphorbiaceae family and it is commonly known as castor oil plant, and Palma Christi. This plant originates in Africa but it is found in both wild and cultivated states in all the tropical and subtropical countries of the world. In wild conditions this plant is well-adapted to arid conditions and is able to stand long periods of drought. Results of transesterification experiments of castor oil with methanol are presented. The castor biodiesel has very interesting properties (very low cloud and pour points) that show that this fuel is very suitable for using in extreme winter temperatures.

**9. Biodiesel production from jatropha oil and its characterization by S. Antony Raja, D.S. Robinson smart, and C. Lindon Robert Lee, School of Mechanical Sciences, Karunya University, Coimbatore, INDIA**

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 *Jatropha* oil by transesterification process represents one of the most promising options for the use of conventional fossil fuels. The *Jatropha* oil is converted into *jatropha* oil methyl ester known as biodiesel 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 *Jatropha* oil and *Jatropha* methyl ester. The same characteristics study was also carried out for the diesel fuel for obtaining the base line data for analysis. The values obtained from the *Jatropha* methyl ester is closely matched with the values of conventional diesel and can be used in the existing diesel engine without any modification.

**10. JATROPHA CURCAS SEED OIL AS A VIABLE SOURCE FOR BIODIESEL by UMER RASHID**

The purpose of the present study was to explore the utility of *Jatropha* (*Jatropha curcas*) seed oil for biodiesel production. The preliminarily evaluated *Jatropha* oil was transmethylated under optimized set of reaction conditions: methanol/oil molar ratio (6:1), sodium methoxide catalyst concentration (1.00%), temperature (65°C) and mixing intensity (600 rpm) providing 94.00% yield of *Jatropha* oil methyl esters (JOMEs)/biodiesel. The gas chromatographic (GC) analysis showed that JOMEs mainly comprised of six fatty acids: linoleic (49.75%), stearic (16.80%), oleic (13.00%), palmitic (12.15%), arachidic (5.01%) and gadoleic (2.00%) acids. <sup>1</sup>H-NMR spectrum of JOMEs was also recorded. The thermal stability of the JOMEs produced was assessed by thermo gravimetric analysis (TGA). The fuel properties of the biodiesel produced were found to be within the standards specifications of ASTM D 6751 and EN 14214.

## Chapter-5:

## RESULTS AND DISCUSSION

The biomass lipid extraction uses the Integrated Solvent Recycle method with carbon tetrachloride and methanol as the solvent. The result of each of the replication is shown under the respective experiments.

The first experiment uses methanol as a solvent and jatropha seeds as the feed which gives a low yield of extracted lipids i.e. 31.5 %.

The second experiment uses carbon tetrachloride as a solvent and jatropha seeds as the feed which gives a high yield of extracted lipids i.e. 40 %.

The third experiment uses methanol as a solvent and Castor seeds as the feed which gives a low yield comparative to the other solvent but a high yield comparative to jatropha seed with the same solvent of extracted lipids i.e. 35 %.

The fourth experiment uses carbon tetrachloride as a solvent and Castor seeds as the feed which gives a high yield comparative to other solvent but a high yield comparative to jatropha seed with the same solvent of extracted lipids i.e. 43%.

From the result of different experiments it is known that castor seed contain less moisture content than Jatropha seeds.

From the result it is clear that non polar solvents are more effective than polar solvents.

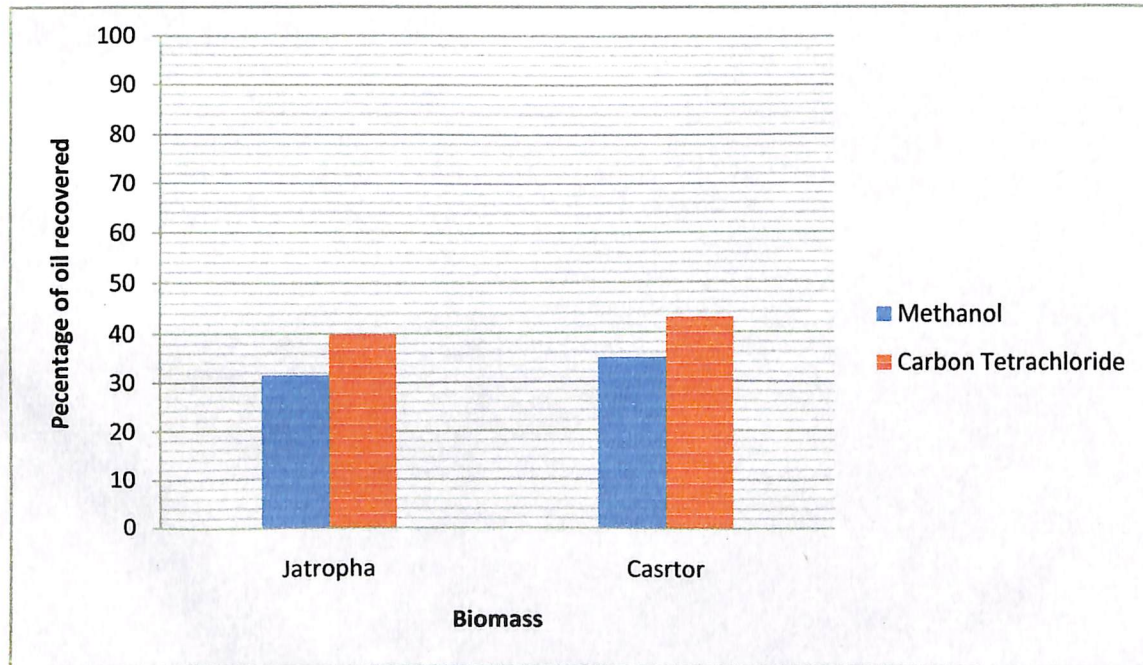


Figure.12

## **CHAPTER 6:**

## **CONCLUSIONS AND RECOMMENDATIONS**

The comparison of the laboratory grade solvent, methanol and carbon tetrachloride, used in lipid extraction from biomass (i.e. jatropha seeds and castor seeds) to the commercial solvents, ethanol and hexane shows that a high percentage of lipids can be extracted using the laboratory solvents, methanol and carbon tetrachloride. The average percent of lipids extracted using carbon tetrachloride and methanol was 37.37%, while the average percent of lipids extracted using commercial grade hexane and ethanol was 34% which is less compared to present yield of the Soxhlet extraction using the given solvent. Castor seeds contain less moisture than Jatropha seeds.

It is recommended to carry out gas chromatography for the recovered lipids to know the carbon compound which can be used as biodiesel. Thus, from our experiment it is concluded that Castor seeds show more lipid content than jatropha seeds.

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