

PREPARATION OF SUSTAINABLE FUEL FROM DUCKWEED AND STUDY OF ITS FUEL PROPERTIES

Submitted in partial fulfillment of the requirements for the award of
Bachelor of Technology Degree in Chemical Engineering
by

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**DEPARTMENT OF CHEMICAL ENGINEERING
SCHOOL OF BIO AND CHEMICAL ENGINEERING**

SATHYABAMA

INSTITUTE OF SCIENCE AND TECHNOLOGY

(DEEMED TO BE UNIVERSITY)

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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **RAJETH RAGHUNATH (40190019)** and **AL.S. MADHAVARAJ (40190034)** who carried out the project entitled "**PREPARATION OF SUSTAINABLE FUEL FROM DUCKWEED AND STUDY OF ITS FUEL PROPERTIES**" under my supervision from November 2023 to April 2024.

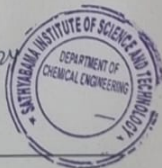
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DECLARATION

We, Rajeth Raghunath and A.L.S. Madhavaraj hereby declare that the Project Report entitled "**PREPARATION OF SUSTAINABLE FUEL FROM DUCKWEED AND STUDY OF ITS FUEL PROPERTIES.**" done by us under the guidance of Dr. Annam Renita.A, M.Tech,Ph.D., is submitted in partial fulfillment of the requirements for the award of Bachelor of Technology degree in Chemical Engineering.

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ABSTRACT

Duckweed (*Lemna minor*) has garnered significant attention in recent years as a versatile resource with potential applications in biofuel production and scientific research. Duckweed is a floating aquatic plant that is abundant in ponds, lakes and other water resources. The physiochemical properties of dried duckweed was analyzed using FTIR, FESEM and XRD techniques. The duckweed oil was extracted by using Soxhlet apparatus with 3 different solvents such as Ethanol, Isopropyl alcohol and Hexane. It was found that the best yield was obtained with the Isopropyl alcohol. The obtained oil was converted to bio diesel by transesterification using homogenous catalyst, sodium hydroxide. The experiments have to be carried out in different ratios in which the higher yield obtained from the ratios is further proceeded with one variable time optimization. The parameters like catalyst weight percentage (wt. %), reaction time and temperature were changed for a particular oil to methanol ratio to obtain the optimized biodiesel. The obtained biodiesel is analyzed using Gas Chromatography-Mass Spectroscopy for analyzing the acids and esters present in it. The properties of duckweed biodiesel were determined according to ASTM D6751 for Free fatty acids, flash point, fire point, pour point, calorific value, density and kinematic viscosity. Engine test was carried with internal combustion engine and the total fuel consumption, specific fuel consumption, brake thermal efficiency, indicated thermal efficiency, torque efficiency, mechanical efficiency, volumetric efficiency and air flow for different loads of biodiesel B15 and B30 blends. Emission analysis for biodiesel was analyzed using fine gas analyzer in which the emission for different gases such as carbon monoxide, oxygen, carbon dioxide, nitrogen oxide and smoke density was analyzed for four different loads. Hence duckweed oil can be successfully converted to bio diesel and can be used in internal combustion engines without any modifications.

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LIST OF ABBREVIATIONS

NaOH	Sodium Hydroxide
KOH	Potassium Hydroxide
FAME	Fatty Acid Methyl Ester
FTIR	Fourier Transform Infrared Radiation
FESEM	Field Emission Scanning Electron Microscope
XRD	X Ray Diffraction
GCMS	Gas Chromatography Mass Spectroscopy
ASTM	American Society of Testing Materials
PMCC	Pensky-Martenz Closed Cup
HCL	Hydrochloric Acid
EDAX	Energy Dispersive X Ray Analysis
SV	Saponification Value
AV	Acid Value
MW	Molecular Weight

CHAPTER - 1

INTRODUCTION

1.1 BACKGROUND

An investigation of the features of duckweed and its potential use for biofuel generation, along with the reasons behind it, usually comprise the background of the project. The following is a broad overview of the project's background.

Growing Need for Sustainable Energy Sources: Researching alternate and sustainable energy sources is becoming more and more important as worries about climate change and the depletion of fossil resources grow. As a viable way to lessen reliance on non-renewable resources and greenhouse gas emissions, biofuels—which come from renewable plant sources—have gained popularity and **Challenges in Biofuel Production:** Traditional biofuel crops, such as corn and sugarcane, have limitations in terms of land and water use, competition with food crops, and the potential for environmental degradation. As a result, there is a need to identify and develop more efficient and environmentally friendly sources of biomass for biofuel production.

The remarkable pace of growth and high starch and fat content of duckweed have made it a viable option for the manufacture of biofuel. A few of the drawbacks of conventional biofuel crops are addressed by these qualities, which make it a desirable option for the manufacture of bioethanol and biobutanol.

Duckweed provides further environmental benefits, including the ability to eliminate excess nutrients from water bodies, such as phosphate and nitrogen. This improves water quality and helps to alleviate problems like algal blooms. **Scientific Research and Model Organism:** Beyond its biofuel potential, duckweed is a valuable model organism for genetic and molecular studies. Its relatively simple genetic makeup allows researchers to investigate various biological processes and develop insights into plant genetics and physiology.

Different species and strains of duckweed are employed in tests based on the objectives of the study. The usual home for these strains is a collection of cultures. **Growth Media:** Duckweed cannot be grown without nutrient solutions or growth

media. Micronutrients, potassium, phosphorus, and nitrogen are among the important nutrients found in these solutions.

Duckweed is cultivated in ponds, tanks, or other regulated aquatic environments. Plastic, concrete, or liners may be needed for these systems' construction. Monitoring equipment for water quality is necessary to maintain ideal growth circumstances. This equipment includes turbidity metres, pH metres, and nutrient testing kits.

1.2 SOXHLET EXTRACTION

There are various methods to extract oil from sources but when it comes to extracting aquatic plants the density differs from the other sources as the raw material has more starch and lipids content. Oil and lipid molecules can be extracted from a variety of botanical sources using the commonly used soxhlet extraction process. The other method is mechanical extraction which is widely used for extracting oil from coconut shells and groundnut but in this case solvent extraction is adopted. By continuously cycling solvents, target chemicals can be effectively extracted from solid matrices using the Soxhlet extraction technique. The extraction solvent is heated to a high temperature, vaporises, condenses, and is then circulated through the sample until the desired extraction is obtained. The success of this process depends on the solubility of the target chemicals in this solvent. Oil from seeds, nuts, plant tissues, and other biological materials can be extracted via Soxhlet extraction, which has an abundance of uses. With its excellent extraction efficiency, ease of use, and capacity for handling big samples, it has many benefits. Ecological restoration efforts by harnessing duckweed's nutrient-absorbing capabilities. Picking an appropriate organic solvent means finding one that is both immiscible with water and effectively dissolves the target compound, in this case, oil. Typical solvents for chemical extractions can be a combination of solvents, hexane, ethanol and isopropyl alcohol.

The solvent flask is positioned underneath the extraction chamber of the Soxhlet extraction device after it has been built. The solvent in the flask vaporises when heated, rising into the condenser as a result. After being cooled by the condenser, the vapour condenses back into liquid and returns to the extraction chamber. It starts

to extract the oil from the solid sample in the thimble as the liquid solvent builds up in the extraction chamber. A continuous cycle is used in the Soxhlet extraction process: when the solvent in chamber the extraction chamber reaches a specific level, it syphons into the solid sample, extracts the oil, and then, because of the temperature difference, drops back into the flask. For a lengthy length of time, this cyclic process keeps the solvent flowing through the solid sample, thereby extracting the oil. Even in situations when the chemical of interest is present in relatively low concentrations in the solid material, the Soxhlet extraction process is intended to guarantee complete extraction of the component. Over time, as the extracted oil dissolves in the circulating solvent, it builds up in the flask. The oil-solvent mixture flask is taken out of the device once a enough amount of oil has been extracted, ending the extraction process. The extracted oil must be separated from the solvent in the following step. This is usually accomplished by evaporation, in which the extracted oil is left behind after the solvent evaporates. The solvent can be effectively removed using methods like basic distillation or rotary evaporation. Oils, lipids, and other organic substances are frequently extracted from solid materials in laboratories using the Soxhlet extraction method. It has a number of benefits, including as excellent extraction efficiency and batch processing capabilities. But it can take a while, and you have to be very careful about safety, especially when handling organic solvents.

1.3 TRANSESTERIFICATION

The transesterification reaction required to produce it can be run on nearly any size, from a small laboratory scale using a few litres of oil to a huge industrial scale that can produce millions of litres of biodiesel annually. Nonetheless, the direct use of vegetable oils and oil mixes is widely regarded as impracticable, despite the potential benefits of commercialization. Some of its most evident disadvantages include its high viscosity, fatty acid composition, and free fatty acid concentration; other disadvantages include gum production from oxidation and polymerization during storage and burning; and carbon deposits and thickening of lubricating oil. due to the finite supply of fossil fuels, an appealing fuel. Biodiesel is synthesised chemically from vegetable and animal fats, which are renewable feedstock, and monoalkylesters of long chain fatty acids. By reacting oil or fat with a monohydric

alcohol in the presence of a catalyst, a process known as transesterification yields the matching monoalkyl esters. With sodium hydroxide acting as an alkaline catalyst, this article presents experimental results on the synthesis of fatty acid methyl esters from sunflower oil. These vegetable oils were used to generate biodiesel, and the factors influencing its yield and properties were investigated. A three-hour reaction period, a 0.5–1.5 weight percent catalyst concentration, and a 1:3–1:9 oil-to-methanol molar ratio were the variables that were examined. The best yield % was found using a based on the findings obtained.

1.3.1 CATALYST

Catalysts are essential to the manufacturing of biodiesel because they aid in the transesterification reaction, which changes triglycerides—which are present in oils and fats—into fatty acid methyl esters (FAMES), which are the main ingredients of biodiesel. Typically, triglycerides and an alcohol (like methanol or ethanol) are reacted in the presence of a catalyst during the transesterification reaction.

Catalysts can be categorized into various types based on their nature, mechanism, and application. Here are some common types of catalysts:

a. Homogeneous Catalysts:

- Sodium Hydroxide (NaOH): A strong base commonly employed in biodiesel production.
- Potassium Hydroxide (KOH): Another strong base often used as a catalyst.

b. Heterogeneous Catalysts:

- Solid Base Catalysts: Examples include calcium oxide (CaO) and barium oxide (MgO), which are solid catalysts.
- Enzymes: Lipases are biological catalysts that can be used for enzymatic transesterification in biodiesel production.

c. Acid Catalysts (for acid-catalyzed transesterification):

- Sulfuric Acid (H₂SO₄): Utilized in the acid-catalyzed method, particularly for low-quality feedstocks.

d. Enzymatic Catalysts:

Lipases: These enzymes can catalyze transesterification reactions in a specific and environmentally friendly manner.

The choice of catalyst depends on factors such as feedstock quality, reaction conditions, and desired product purity. Homogeneous catalysts are more commonly used in traditional biodiesel production, while heterogeneous catalysts and enzymes may offer advantages in specific applications or for higher-quality biodiesel production.

1.3.2 METHANOL

Triglycerides are the main building blocks of vegetable oil, animal fats, and microalgae. Methanol reacts strongly with these compounds to speed up the transesterification process, which produces biodiesel. A greater degree of freedom in catalyst selection and optimization processes is afforded by the vast spectrum of catalysts with which methanol can react, primarily in transesterification reactions. Methanol helps to improve the purity and quality of the final product by reducing unwanted byproducts or side reactions during the biodiesel synthesis process. Environmental advantages come from the usage of methanol to make biodiesel, which lowers greenhouse gas emissions as compared to petroleum fuel.

1.4 BIOFUEL

As the need for energy rises and non-renewable resources run out, it is imperative that sustainable energy sources be developed. There is a lot of interest in biodiesel as a fossil fuel substitute since it is environmentally benign and sustainable. However, the costly and inefficient previous biodiesel production process has limited its widespread adoption.

Reduction in Greenhouse Gas Emissions: In general, diesel derived from petroleum emits more greenhouse gases than biodiesel. By lowering carbon dioxide and other harmful pollutants that contribute to global warming, it can help moderate climate change. When considering lifecycle greenhouse gas emissions, biodiesel derived from leftover vegetable or animal fats can be substantially less than petroleum fuel.

Diversification of Fuel Sources: There are hazards associated with supply disruptions, geopolitical instability, and price volatility when driving or using energy primarily from petroleum-based fuels. By increasing energy security and lowering reliance on finite fossil fuels, biodiesel provides a means of fuel diversification.

Various renewable resources can be used to make biodiesel, such as animal fats, algae, and even recycled cooking oil. Vegetable oils include soybean, canola, and palm oil. Reducing the demand on non-renewable fossil fuel supplies, these resources can be grown or obtained sustainably and are replenishable.

Encouraging Rural Development and Agriculture: By giving farmers the chance to cultivate certain energy crops or turn agricultural waste into fuel, biodiesel production can boost rural economies. In agricultural regions in particular, this can boost local economies and generate jobs. **Better Air Quality:** Burning biodiesel results in lower emissions of sulphur oxides, particulate matter, and aromatic hydrocarbons than burning conventional diesel fuel.

Petroleum diesel can be easily replaced with biodiesel, which is a suitable drop-in alternative for diesel engines and infrastructure that are already in place. Mixing different amounts of biodiesel with regular diesel (B5, B20, or greater blends) is a standard procedure that facilitates adoption gradually without requiring major adjustments to fleets of vehicles or infrastructure.

1.4.1 ADVANTAGES OF BIOFUEL

The production of biofuels has the potential to boost rural economies by generating employment and giving farmers, landowners, and biofuel producers more revenue options. Sustainable land management techniques, rural development, and money generation for agricultural communities can all be achieved through the cultivation of biomass feedstocks and bioenergy crops.

Compatibility with Current Infrastructure: In many cases, existing engines, cars, and transportation infrastructure may be used to blend biofuels like ethanol and biodiesel with conventional petroleum fuels without requiring significant improvements or adjustments. Due to their compatibility, biofuels may be widely used without requiring large infrastructure investments, allowing for a more gradual shift to renewable fuels.

Advances in biotechnology, agronomy, bioengineering, and process optimisation are fuel by the development and marketing of biofuels. Research and development investments in biofuels help to increase feedstock quality, biomass conversion

technologies, and sustainable bioenergy production methods, which benefits the biofuels business as well as adjacent industries.

The production of biofuels on a local or regional scale can lead to energy independence and a decrease in dependency on remote energy sources. Communities may develop value-added goods, eliminate energy losses connected to fuel distribution and supply chains in transportation, and maximise their own renewable energy potential by leveraging locally accessible biomass resources.

1.4.2 Disadvantages of Biofuel

Competition for resources such as land and water between energy and food crops is one of the main issues with biodiesel production. Land use disputes, deforestation, environmental degradation, and higher food costs might result from large-scale biofuel production that depends on food crops like maize or soybeans.

The significance of developing non-food biomass sources and sustainable feedstock sourcing for biofuel production is emphasised in this article. The proliferation of biofuel feedstock production has the potential to cause alterations in land use, such as clearing forests, destroying natural habitats, and converting natural ecosystems into agricultural land. Soil erosion, biodiversity loss, and disruption of ecosystem services are just a few of the detrimental effects these changes may have on the environment. Plans for land use and sustainable land management are crucial.

A number of variables, including climate, soil quality, agricultural methods, and crop yields, can affect the availability and productivity of biomass feedstocks for the production of biodiesel. In order to meet major energy demands, large land areas must be dedicated to the growth of some bioenergy crops, which may have lower energy outputs than fossil fuels.

Developing high-yielding feedstock varieties, streamlining farming techniques, and boosting agricultural output are crucial approaches to overcome this restriction. The mixture is allowed to separate once the reaction is finished, with the higher-density glycerol at the bottom and the lower-density biodiesel at the top. Improved performance and a high cetane number can be obtained along with cost-effective

and time-efficient biodiesel production through the use of the transesterification method. While biodiesel produces fewer emissions than diesel, it still has an impact on the environment because of its production process, especially when it comes from non-renewable sources. Environmental waste management problems can occasionally arise from glycerol overproduction.

1.5 PROPERTIES OF BIOFUEL

Chemical Composition: The main component of biodiesel is fatty acid methyl esters (FAMES), which are produced from the triglycerides found in animal or vegetable fats. Depending on the feedstock utilised to produce it, biodiesel can have a different precise composition.

Viscosity: In diesel engines, fuel flow and atomization can be impacted by the viscosity of biodiesel, which is generally higher than that of petroleum diesel. However, variables like temperature and feedstock mix affect how viscous of the biodiesel.

Cetane Number: This is a metric used to assess how well diesel fuel ignites. When compared to petroleum diesel, biodiesel typically has a higher cetane number.

Oxidative Stability: When it comes to oxidation and degradation during handling and storage, biodiesel is more vulnerable than petroleum diesel due to its poorer oxidative stability. To extend the shelf life of biodiesel and stop gums and deposits from forming, antioxidants and stabilisers can be added.

Lubricity: Due to its lower concentration of sulphur compounds, which function as lubricants, biodiesel offers superior lubricating qualities than ultra-low sulphur diesel (ULSD). Because biodiesel is more lubricating, it can shield the fuel system.

The temperature at which a fuel releases enough vapours to continue burning once it is ignited is known as its "fire point." Usually, it is greater than the flash point—the temperature at which, in the presence of an ignition source, fuel vapour can briefly ignite. Because it represents the temperature at which a fuel can burn continuously when it is lit, as opposed to only flashing briefly, the fire point is a crucial safety criterion.

Fire point: When compared to petroleum diesel, biodiesel often has a greater fire point. This is so because the chemicals found in biodiesel tend to have higher

molecular weights and need more energy to vaporise and continue burning. As a result, when it comes to fire safety, biodiesel is preferred over petroleum-based diesel.

Calorific value: Biodiesel's precise composition of fatty acid methyl esters (FAMES) and other additives that may impact its energy content, as well as the feedstock used in its manufacturing, can all affect the fuel's calorific value.

The calorific value of biodiesel is comparatively high to petroleum diesel, with an average calorific value of about 37 to 42 megajoules per kilogramme (MJ/kg). As biodiesel obtained from various feedstocks or manufacturing procedures has slightly different energy levels, the precise calorific value of biodiesel might vary depending on its chemical makeup.

Density: The feedstock utilized, the method of production, and the presence of any additions or contaminants can all affect the density of biodiesel. However, at standard temperature and pressure (STP), which is defined as 25 degrees Celsius (77 degrees Fahrenheit) and 1 atmosphere of pressure, the density of biodiesel generally ranges between 0.86 and 0.90 grams per millilitre (g/mL). The densities of biodiesel blends, like B20, which is composed of 80% petroleum diesel and 20% biodiesel, will range between that of pure biodiesel and petroleum diesel; the precise figure will depend on the mix ratio.

Pour point: The lowest temperature that allows biodiesel to continue flowing or pumping without hardening or becoming too viscous is known as the pour point. Since it affects the functionality of vehicles and equipment that run on biodiesel, it is a crucial attribute, particularly in colder climates or during the winter. Various factors, including the feedstock used in the production process, any added materials or contaminants, might affect the pour point of biodiesel. The pour point range of biodiesel is typically between -9°C and -15°C (16°F and 5°F). While pure biodiesel has a somewhat lower pour point, some blends, like B20 (20% biodiesel, 80% petroleum diesel), may have a slightly greater pour point.

Flash point: When an ignition source is present and certain test circumstances are met, the lowest temperature at which the fuel's vapor can momentarily ignite is known as the biodiesel flash point. For the handling, moving, and storage of biodiesel, it is a crucial safety factor. An estimated range of 120°C to 160°C (248°F

to 320°F) is the flash point range for biodiesel. Conventionally, testing protocols like ASTM D93 or ISO 2719 are used to determine the flash point. These experiments include gradually heating a small sample of biodiesel and frequently passing a small flame over the sample's surface. The flash point of a sample is the temperature at which the vapor above it briefly ignites.

1.6 APPLICATIONS OF BIODIESEL

Biodiesel is a renewable and eco-friendly fuel substitute that can be used in a variety of industries. Typical uses for biodiesel include:

Transportation Fuel: With minimal to no modification needed, biodiesel can be used directly in diesel engines to replace petroleum diesel. It can be utilised in a variety of vehicles, including cars, lorries, buses, trains, boats, and agricultural equipment, and blended with petroleum diesel in different ratios (such as B5, B20, or greater blends).

Heating Fuel: In commercial, industrial, and domestic heating systems, including space heaters, boilers, and furnaces, biodiesel can be used in place of heating oil.

Production of electricity: Biodiesel can be used as fuel for combined heat and power (CHP) systems and stationary diesel generators, which produce both heat and power. Microgrids, distributed energy systems, and renewable energy installations with intermittent or limited access to grid electricity are among the many settings where biodiesel-fueled generators find frequent application.

Applications in the Marine and Aviation Industries: Biodiesel can be utilised as fuel for maritime vehicles, including ships, boats, and ferries, as well as in aviation applications, like aviation biofuels or as a blend element in jet fuel. Even though the marine and aviation industries utilise biodiesel in very small amounts when compared to other forms of transportation, continued research and development efforts are being made to increase its application and compatibility with marine and aviation engines. **Industrial and Commercial Applications:** Biodiesel can be employed as a feedstock or precursor in a number of industrial processes, including those that create surfactants, lubricants, bioplastics, and other materials and chemicals derived from plants.

Biodiesel can be a feedstock or a solvent in a variety of industrial processes. It can be used in industries like manufacturing, chemical production, and others where it is preferable to have a renewable and biodegradable product instead of one derived from petroleum. All things considered, biodiesel finds use in a variety of industries and provides a sustainable and green substitute for fuels derived from petroleum. To fully realise biodiesel's promise as a contributor to a more sustainable energy future, research and development in both production and use must continue.

CHAPTER - 2

LITERATURE REVIEW

- **GH Niawanti, N N Octavia et al. (2018)** Using a Soxhlet device and 500 mL of 96% ethanol as a solvent, the study extracted the leaves of *Averrhoa bilimbi*. Because bioactive chemicals are soluble in ethanol and are both non-toxic and biodegradable, ethanol was employed as a solvent. After being gathered, the leaves were cut and dried at 60 °C until their weight stabilised. After 24 hours of drying, the leaf's moisture content was 48.67%. Examining the impact of extraction time on leaf ethanol extract yield was the aim of the study. The extraction process was run of total flavonoids were produced, which was the highest yield. Because of the ideal extraction time of 360 minutes, the yield was lowered.
- **Haifeng Su et al. (2014)** The family of aquatic plants known as duckweed (*Lemnaceae*) has the potential to serve as the feedstock for the next generation of alternative energy sources, however not much information about turning duckweed into higher alcohols has been published. We looked into the fermentation procedure used to extract higher alcohols from duckweed. The production of higher alcohols with high energy yields through bioconversion by *Clostridium acetobutylicum*, mutant yeast strains, and bioengineered. Duckweed acid hydrolysate was used to create butanol and total solvent at concentrations of 12.03 and 20.03 g/L through fermentation by *C. acetobutylicum*.
- **Kamrun Nahar et al. (2019)** As a potential substitute for natural gas and crude oil, which are finite resources on which Bangladesh is heavily dependent, duckweed has great promise as a feedstock for ethanol and biogas production. The energy needed for long-term economic expansion cannot be met by the available reserves. The burning of biofuels produces significantly less hydrocarbons, and particulate matter than that of fossil fuels, making them harmless and biodegradable substitutes. Over millions of

hectares of lakes, rivers, and ponds make up Bangladesh's prospective sites for phytoremediation.

- **Gui Li Yang et al. (2022)** Based on biomass, biofuels are seen as the most promising alternative due to the increasing shortage of conventional energy sources and the corresponding serious environmental challenges. The manufacturing processes and synthesis mechanisms of this essential raw material for biomass energy are the subject of growing investigation. Owing to its many benefits, such as quick biomass buildup, high starch content, high biomass conversion efficiency, and sewage remediation, duckweed has been utilised as a premium new biomass feedstock in recent years. Duckweed is a new energy plant. This paper offers a comprehensive assessment of its growth traits, starch metabolic routes, and strategies for enhancing starch accumulation.
- **Nazim Muradov et al. (2014)** One of the most extensively explored topics in the world is phytoremediation, which offers an appealing option for the bioremediation of water pollution. It is based on the capacity of aquatic plants to recycle nutrients. An affordable and environmentally beneficial solution to the problem of water pollution and the creation of value-added products is the following application of the biomass from the remediation for the production of fuels and petrochemicals. Results This study investigates the viability of using duckweed and azolla aquatic plants in tandem to treat wastewater and produce petrochemicals and renewable fuels.
- **Md Arman Arefin et al. (2020)** Aquatic plants have been deemed concerning. Still, one new energy source is the creation of biofuel from aquatic plants. So, along with their physicochemical characteristics, this research provides a thorough overview of the production of biofuel from floating aquatic plants. This research aims to assess some of the key aquatic plants' capacity to produce biofuel. Also, some viable techniques for producing biofuel from aquatic plants are presented in this research for use in future energy production. This study examines five main categories of floating aquatic plants: azolla, water hyacinth, water fern, water lettuce, and

duckweed. It also examines the process of producing biofuel from these plants using transesterification, pyrolysis, hydrolysis, and torrefaction. Also, this essay assesses ideal.

- **Wei-Hein Chen et al. (2016)** The investigation focused on the effects of extraction solvents on the yields of product fractions and the makeup of the bio-oils that resulted from hydrothermally processing duckweed at 350°C for 30 minutes. A total of ten solvents were used, comprising nonpolar solvents like petroleum ether and cyclohexane as well as polar solvents like isopropanol, ethyl acetate, dichloromethane, diethyl ether, dichloroethane, benzene, and carbon disulfide. Bio-oil yields were often greater in the extraction solvents with high relative polarity values.
- **Asaad H. Sayer et al. (2023)** One potential substitute that could help provide energy security is biodiesel made from renewable resources. By using direct transesterification, this study attempted to create biodiesel from duckweed plants and *Oedogonium* algae. The considered duckweed plant was given 5 g of dry biomass, and to extract fatty acid methyl esters (FAME), the algae was submerged in 50 mL of a 10:1:1 v/v/v solvent mixture of methanol, hydrochloric acid, and chloroform. Utilising FT-IR and GC-Mass spectrometry, the isolated components were collected for characterization.
- **Natalia Politaeva et al. (2018)** In this work, the same polar solvent (ethanol) is used in combination with different non-polar solvents (methanol-ethanol, chloroform-ethanol, hexane-ethanol, diethyl ether-ethanol, and petroleum ether-ethanol) at varying volume ratios to investigate the effects of different solvent extraction systems on the recovery of lipids extracted from the dry biomass of *Lemna minor*. Finding a Soxhlet extraction technique that uses a variety of solvents to improve lipid recovery while reducing the extraction time is the aim.
- **Amalate Ann Obuebite et al. (2022)** By using pyrolysis, duckweed was converted into bio-oil and its properties were determined. With the use of pyrolysis, the study's objective is to create and evaluate bio-oil from

duckweed. A thermocouple attached to the reactor monitored the temperature, and a stopwatch tracked the amount of time until the final drop of product was observed from the system. This study used the pyrolysis method to produce bio-oil from duckweed. A fix-bed reactor was connected to condensers, and heat was applied from a gas hob.

- **Yulia Smyatskaya, Natalia Politaeva et al. (2018)** The literature contains earlier research on the makeup of higher aquatic plants' lipid content. In the current work, ethanol is used as the polar solvent, and different non-polar solvents, such as methanol, chloroform, hexane, diethyl ether, and petroleum ether, are used at different volume ratios to study the effects of different solvent extraction systems on the recovery of lipids extracted from the dry biomass of *Lemna minor*. Using several solvents, the objective is to choose a Soxhlet extraction technique that maximises lipid recovery while reducing the extraction time.
- **Akinwale, A. Olusegun, Ojo-Awo, A. Phillip et al. (2014)** To produce biofuel, land crops must be grown indefinitely. This might destroy forests and raise greenhouse gas emissions. To assess possible applications in industry and household as a replacement for traditional oils and non-renewable fossil fuels, oil extraction was done on microalgae and two aquatic plants. An earthen fish pond's surface was used to sift the aquatic plants Elodea and Lemna using a hand net, while the walls and water's surface of a concrete fish tank were scraped for microalgae samples (MCA). The samples' oils were extracted using the Soxhlet extraction process.
- **F. Ferella a, G. Mazziotti Di Celso et al. (2010)** In order to produce biodiesel, the transesterification process of rapeseed oil was studied in this work using response surface methodology (RSM). Using a full factorial design, the three primary factors that influence the conversion of triglycerides into fatty acid methyl esters (FAME) were investigated at two levels. Temperature, reaction duration, and catalyst concentration (KOH) were these variables. The scope of each factor's investigation was chosen with consideration for Fox Petroli

S.p.A.'s procedures. The elements that largely influence the first of the two transesterification stages were found to be significant based on their interactions, which were ascertained using analysis of variance (ANOVA). The optimal transesterification reaction operating conditions for Fox's factory were demonstrated by this analysis: Temperature 50 °C, KOH content 0.6% w/w, and reaction.

- **T Kuznetsova¹, N Politaeva¹ et al. (2019)** The use of duckweed for the national economy is examined in this research, including its use in phytoremediation, medicine, bioethanol and biogas generation, and livestock feed. The goal of the St. Petersburg climate was achieved through the cultivation of duckweed, *Lemna minor*. In an artificial reservoir and a natural reservoir.
- **Małgorzata Krzywonos et al. (2023)** One growing trend among scientists is the use of plants from the Lemnaceae family. The study aimed to describe patterns in scientific literature concerning the application of aquatic plants belonging to the Lemnaceae family for energy generation, particularly for the creation of biogas, bioethanol, and other biofuels. The idea of a circular economy is ideally suited for these plants. To assess the utilisation of plants in the Lemnaceae family for biofuel generation, this study conducted a thorough content analysis and bibliometric analysis.
- **Oraleou Sangué Djandja, Linxin Yin et al. (2021)** Duckweed processing has been listed as one of the possible avenues for producing biofuels. Its capacity for high protein or starch content, depending on its species and growth conditions, and its straightforward hand harvesting process are credited with this attribute. Duckweed can be reliably utilised while also providing a reliable answer to energy and environmental issues through the generation of biofuels from the plant. The pyrolysis and hydrothermal liquefaction procedures used to produce bio-oil from duckweed are the main topics of this critical evaluation.

- **Elfina Azwar, Hajar Rastegari et al. (2022)** Aquatic weeds in treatment ponds develop quickly, which presents an unfavourable obstacle to shellfish aquaculture and necessitates continuous weed disposal by the farmers. This article examines the potential and uses of a variety of aquatic weeds in the context of current thermochemical processes (torrefaction, hydrothermal carbonization/liquefaction, pyrolysis, gasification) for the production of biofuels. A discussion was held regarding the impact of crucial operational factors on maximising the efficiency of aquatic weed conversion. This included an analysis of the benefits, limitations, and techno-economic features of thermochemical technologies, as well as their feasibility for widespread implementation.

CHAPTER 3

AIM AND SCOPE

3.1 AIM

- To determine the best solvent for the extraction of duckweed oil using various solvents.
- To convert the duckweed oil to duckweed biodiesel using transesterification process.
- To determine the physiochemical properties of duckweed (*lemna minor*) using FTIR, FESEM and XRD analysis.
- To identify and quantify the presence of esters in biodiesel from GCMS analysis.
- To determine fuel properties according to ASTM standards.
- To conduct engine test for the prepared biodiesel.
- To conduct emission analysis for the prepared biodiesel.

3.2 SCOPE

- Developing effective processes to transform duckweed biomass which is considered as waste i.e., turned into biofuels is the project's main goal. Developing extraction techniques to turn lipids into fuel that can be used is part of this.
- The project's scope includes future work of scaling up the process for commercial purpose.
- Biofuels can replace the fossil fuels in near future and it is to be noted that from where the source of the biofuel comes from, it is appreciable if it is obtained from natural resource like in this project it is obtained from the aquatic plants that is Duckweed which is a promising candidate for the production of biofuel.

CHAPTER – 4

MATERIALS AND METHODS

4.1 MATERIALS AND CHEMICALS

The materials used for the producing biodiesel are duckweed obtained from water resources. Sodium hydroxide used as the catalyst and methanol are used for the transesterification process. The primary process used to convert oil into biodiesel (fatty acid methyl esters, or FAMES) is transesterification, which is where methanol plays a critical role in the manufacturing of biodiesel. In the process of making biodiesel. Triglycerides found in oils are converted into fatty acid methyl esters, which are the primary ingredients of biodiesel, through a process known as the transesterification reaction. Catalysts like sodium hydroxide (NaOH) must be used with an alcohol, usually methanol for the production of biodiesel.

4.2 EXTRACTION OF OIL FROM DUCKWEED

Duckweed is a promising feedstock for producing bio fuel. It is obtained from sources like ponds, lakes. The obtained duckweed is in wet condition and its weight is around 1000 grams.



FIG 4.1: Duckweed



FIG 4.2: Drying of duckweed

The obtained duckweed is spreaded over a thin cloth and got it dried in presence of sunlight. The resulting dried product is measured and the dry weight is around 32 g.

1000 grams (Wet duckweed) ➡ 32 grams (Dried duckweed)

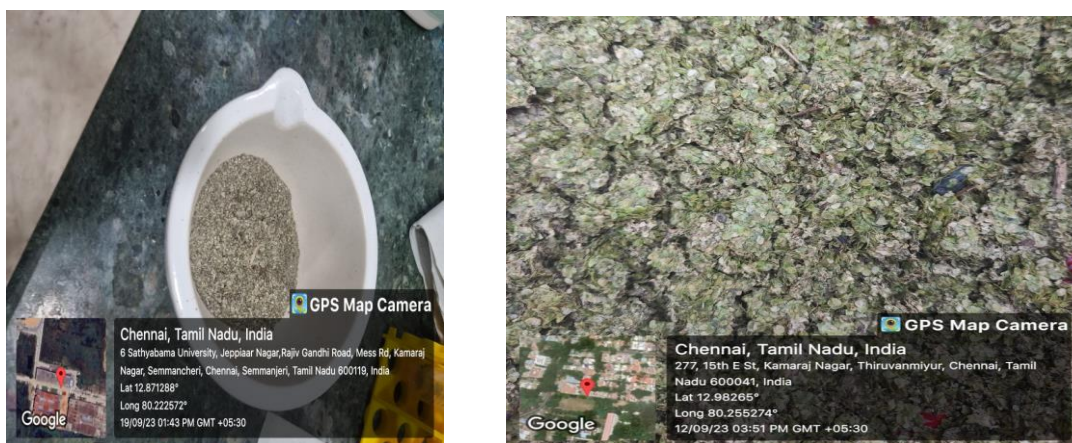


FIG 4.3: Crushing of duckweed

The dried duckweed is separated into 3 samples each sample contains of 10 grams and it is packed in a filter paper by crushing it into fine powder using mortar and pestle for solvent extraction. The solvent used for extraction are ethanol, isopropyl alcohol and hexane. The boiling point of the solvents are 78.7°C, 82.5°C, 68.7°C respectively. The extraction is done by using an equipment called Soxhlet apparatus. This equipment is used to extract oil from the duckweed.

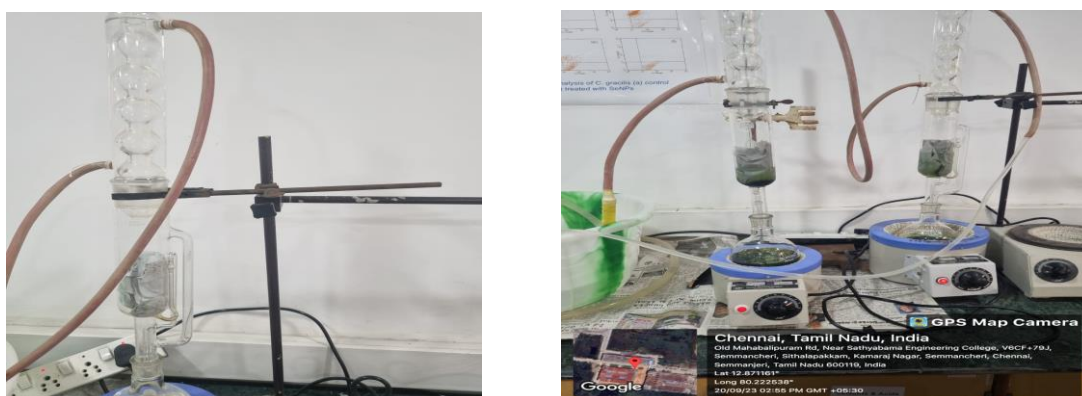


FIG 4.4: Soxhlet apparatus

The apparatus in and outflow connected with the water tub as condensation and vaporization process happens simultaneously. The solvent is poured in a round bottom flask and the solvent taken for extraction weighs of 300ml. The extraction is done for around 4 to 5 hours. Icepack has been added to the water tub periodically.

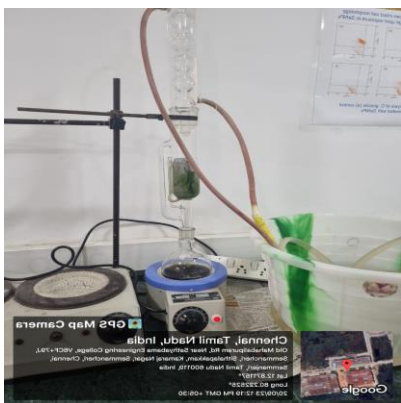


FIG 4.5: Solvent extraction



FIG 4.6: Rotary evaporator

The extracted oil is then proceeded to separate out the oil from the solvent this is done by using vacuum evaporator. The temperature in the vacuum evaporator has been set so that oil from the solvent can be separated. It is done around for 20 minutes. Now we have the duckweed oil separated from solvent.

The oil weighs for 3 different solvents such as ethanol(10.009ml), isopropyl alcohol (28.25 ml) and hexane (7.441ml). So among the three solvents used for extraction, the extraction done using ethanol solvent gave more oil than the other 2 solvents.

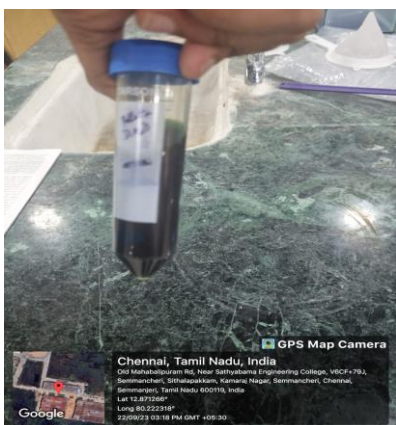


FIG 4.7: Oil extracted from different solvents

The yield of the oil is calculated on the basis of amount of oil obtained from the extraction. Among the three solvents used for extraction, in terms of quantity the isopropyl alcohol gave more yield. The other properties of the oil such as specific gravity and density of the oil is studied to finalise the oil for further production of biodiesel. Specific gravity of the oils are calculated using Picnometer i.e, specific gravity bottle in which the empty bottle weight is measured (w_1) and the bottle is

filled with is water and weight is measured and mark it as (w2) and the bottle is filled with the oil samples and the weight is measured (w3),(w4),(w5)respectively

Specific gravity = Density of oil / Density of water

Specific gravity = (w2 -w1)/ (w3-w1)

The specific gravity of the oil obtained from ethanol,isopropyl alcohol,hexane solvents are (0.86),(0.74),(0.97) respectively.Specific gravity is a relative quantity therefore the oil with a higher specific gravity results in producing poor diesel.The density of the biodiesel is 1.006g/cc which meets the ASTM standards for producing quality biodiesel.

4.3 PROPERTIES OF DUCKWEED OIL.

4.3.1 DETERMINATION OF ACID VALUE OF OIL

The quantity of free fatty acids contained in an oil is indicated by its acid value. In the food and pharmaceutical industries in particular, it is a crucial characteristic for the examination of oils and fats.

The acid value is commonly represented as the milligrams of sodium hydroxide (NaOH) needed to neutralize one gram of oil's worth of free fatty acids. Higher acid values signify a higher level of rancidity or deterioration, making it a gauge of the oil's quality and freshness.

A standardized NaOH solution can be used for titration, one of the analytical techniques that can be used to determine the acid value. It is a crucial criterion for determining if oils are suitable for a range of uses, such as cooking.

Chemicals required: Ethanol,NaOH,Indicator (phenolphthalein)

Materials required: Oil sample,burette with stand,hot plate,weighing balance.

- Prepare 0.1n NaOH solution.
- Add few drops phenolphthalein indicator to 50 ml Ethanol.
- Titrate this against NaOH to neutralize.
- Neutralized ethanol is added with 10g of oil and it is heated using hot plate.
- After 20 mins add few drops of indicator and titrate against 0.1 NaOH

- Note initial and final burette readings.

Acid value = $40 \times 0.1 \times (F.V - I.V) / \text{Weight of the sample}$

Acid value = $40 \times 0.1 \times 1.6 / 10$

Acid value of the oil = 0.64

4.3.2 DETERMINATION OF FFA OF OIL

The term "free fatty acids" (FFAs) describes the unesterified fatty acids that are found in oil. Since they are not connected to any other molecule, like glycerol, they are referred to as "free." FFAs naturally occur in oil as a result of oxidation, hydrolysis, or other degrading processes.

Particularly in the food sector, the amount of FFAs in an oil is a significant determinant of its quality. Increased levels of FFAs can cause food products to have bad Flavors, have a shorter shelf life, and have less nutritional content. Consequently, reducing FFAs is crucial to preserving the stability and quality of oils. The acid value determination method, which quantifies the quantity of KOH needed to neutralize the FFAs, is frequently used to test FFAs in oil.

Free fatty acid is calculated by : Acid value of oil/2

The obtained FFA of oil is 0.32. It means that the extracted duckweed oil is to be proceeded with single step process i.e, transesterification.



FIG 4.8: Acid value determination

4.3.3 DETERMINATION OF SAPONIFICATION VALUE OF OIL

The amount of alkali required to saponify a specific amount of fat or oil is indicated by the oil's saponification value. The amount of potassium hydroxide (KOH) milligrams needed to saponify one gram of oil is how it is expressed.

The amount of lye (potassium or sodium hydroxide) required to convert a given volume of oil into soap is calculated in the soap industry using the saponification value. Additionally, it offers details regarding the fatty acids' average molecular weight in the oil.

Chemicals required: Absolute ethanol, KOH, HCL, Indicator.

- Materials required: Reflux column, Weighing balance, Burette with stand, Oil sample.
- Measure 200 ml ethanol add 1.67 g of KOH and add 1g of granulated aluminium foil and transfer it into round bottom flask and heat it using heating mantle with reflux column on top.
- Using rotary evaporator Ethanolic KOH is separated.
- Prepare a sample of blank and oil.
- Titrate against 0.1N Hcl. Note initial and final readings.
- Blank sample : $(50-0) + (39-20) = 69$ ml
- Oil sample : $(17.5 - 11.5) = 6$
- Saponification value = $28.05 * (V_b - V_s) / W_s$
- $S.V = 28.05 * (69 - 6) / 5.2$
- Saponification value of oil = 283

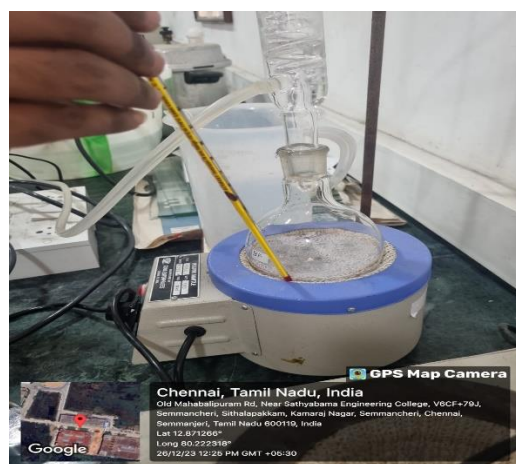


FIG 4.9: Reflux column

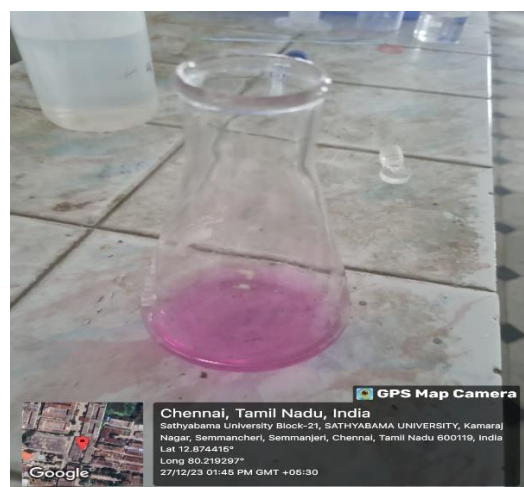


FIG 4.10: Saponification value determination

4.3.4 DETERMINATION OF MOLECULAR WEIGHT OF THE OIL

$$M.W = 56.1 \times 1000 \times 3 / (S.V - A.V)$$

$$M.W = 56.1 \times 1000 \times 3 / (283 - 0.6)$$

Molecular weight of the extracted oil = 596 g/mol.

4.4 PREPARATION OF BIODIESEL

Biodiesel is prepared in different ratios and in different conditions by changing the parameters. The experiment is carried out in a magnetic stirrer set up with a condensation column fixed so that it prevents the solvent from evaporating. Obtaining and getting ready the reactants required for the transesterification reaction is the first stage. The feedstock in this case would usually be vegetable or animal fat, the alcohol would be methanol (or ethanol), and the catalyst would be sodium hydroxide (NaOH).

Reactant mixing involves combining methanol and vegetable (or animal) oil in a reaction vessel with the catalyst present. The vessel is set on a hot plate for a magnetic stirrer and has a magnetic stir bar within. The transesterification reaction is sped up by heating the mixture to a certain temperature, usually between 50°C and 60°C. Heating increases the rate of reaction and reduces the oil's viscosity.

The magnetic stir bar inside the reaction vessel spins quickly due to the magnetic field created by the magnetic stirrer. In addition to ensuring complete mixing of the reactants, this stirring movement also facilitates effective contact between the alcohol, catalyst, and oil.

The transesterification reaction produces fatty acid methyl esters (FAMES) and glycerol when the oil's triglycerides combine with the alcohol in the presence of the catalyst. Triglycerides are better converted to biodiesel thanks to the swirling action, which also helps to improve reaction kinetics.

Reaction Monitoring: Temperature, pH, viscosity, and other variables are usually measured to track the transesterification reaction's development over time. The mixture is permitted to settle after the reaction is finished in order to separate the biodiesel (FAME) layer.

4.4.1 Experimental calculation for biodiesel

Molecular weight of the oil = 596 g/mol

Molecular weight of the methanol = 32.04 g/mol

Density of the oil = 1.006 g/cc

Density of the methanol = 0.791 g/cc

For 1:3 ratio

1 mol of oil : 3 mol of methanol

$$1 \times 596.04 = 3 \times 32.04$$

$$596.04 = 96.12$$

$$596 / 1.006 = 96.12 / 0.791$$

$$592.48 = 121.51$$

40 ml of oil = x ml of methanol

X = 8.2 ml methanol required for transesterification.

Mass of methanol = Density of methanol * Volume of methanol

$$\text{Mass} = 0.791 \times 8.2 = 6.486$$

Mass of the oil = 40.24

Total mass = 46.24

Catalyst at 3 Wt % $X / X + 46.24 = 3 / 100$

X = 1.43 grams required for transesterification.

For 1:6 ratio

$$596.04 / 1.006 = 6 \times 32.04 / 0.791$$

$$592.48 = 243.034$$

$$40 = X \text{ ml of methanol}$$

$$X = 16.40 \text{ ml}$$

$$\text{Total mass} = 53.21$$

$$\text{Catalyst at 3 Wt \%} = 1.65 \text{ grams.}$$

For 1:9 ratio

$$592.48 = 9 * 32.04 / 0.791$$

$$X = 24.611 \text{ ml of methanol}$$

$$\text{Total mass} = 59.7$$

$$\text{Catalyst at 3 Wt \%} = 1.84 \text{ grams}$$

For 1:12 ratio

$$592.48 = 12 * 32.04 / 0.791$$

$$X = 32.81 \text{ ml}$$

$$\text{Total mass} = 66.19$$

$$\text{Catalyst} = 2.24 \text{ g}$$

For 1:15 ratio

$$592.48 = 15 * 32.04 / 0.791$$

$$X = 40.9 \text{ ml}$$

$$\text{Total mass} = 72.54$$

$$\text{Catalyst} = 2.44 \text{ g}$$



FIG 4.11: Transesertification setup



FIG 4.12: Biodiesel

4.4.2 OPTIMIZATION OF BIODIESEL

In order to increase productivity, yield, and product quality while lowering expenses and having a minimal negative impact on the environment, biodiesel production must be optimized in a number of ways. Make sure that the alcohol-to-oil ratio, reaction temperature, reaction time, catalyst type, and concentration are all

optimized for the transesterification reaction. The yield, purity, and conversion efficiency of biodiesel are influenced by these factors.

From the below table, particular ratio 1:12 is tried with change in parameters like time, temperature and in catalyst weight percentage to obtain the optimized biodiesel. The one with the highest yield among the ratios is tried with different experiments. The final output is proceeded to characterization to know more in detail

TABLE 4.1 OPTIMIZATION OF BIODIESEL

S. no	Ratio	Oil (ml)	Methanol (ml)	Mass of methanol	Mass of Oil	Catalyst Wt. %	Catalyst in (g)	Time in min	Temp °C	Yield %
1	1:3	40	8.2	4.0341	40.2	3	1.26	45	60	42
2	1:6	40	16.4	8.068	40.2	3	1.38	45	60	49.6
3	1:9	40	24.611	12.11	40.2	3	1.51	45	60	63
4	1:12	40	32.81	16.11	40.2	3	1.63	45	60	74
5	1:12	40	32.81	16.11	40.2	1	1.45	45	60	72
6	1:12	40	32.81	16.11	40.2	1	1.45	30	45	76
7	1:12	40	32.81	16.11	40.2	2	1.58	40	45	78
8	1:12	40	32.81	16.11	40.2	2	1.58	30	45	74
9	1:12	40	32.81	16.11	40.2	3	1.58	60	60	70
10	1:15	40	40.9	20.15	40.2	3	1.76	45	60	54.2
11	1:18	40	49.22	24.20	40.2	3	1.88	45	60	61

CHAPTER 5

RESULTS AND DISCUSSIONS

5.1 PHYSIOCHEMICAL PROPERTIES OF DUCKWEED

5.1.1 FTIR

Fourier transform – infrared radiation spectroscopy The Fourier transform infrared spectroscopy (FTIR) is a valuable tool for identifying organic compounds, polymers, and functional groups in a variety of samples. It does this by measuring the interaction of materials with infrared light.

Once the functional groups have been identified, several characteristics of the sample, including its chemical composition, structure, purity, and even some physical attributes, can be inferred. An infrared absorbance (or transmittance) vs wavenumber (or wavelength) plot results in the resulting spectrum. It is possible to identify the functional groups present in the sample by comparing the peaks and troughs in the spectrum to recognized spectral patterns found in databases or literature.

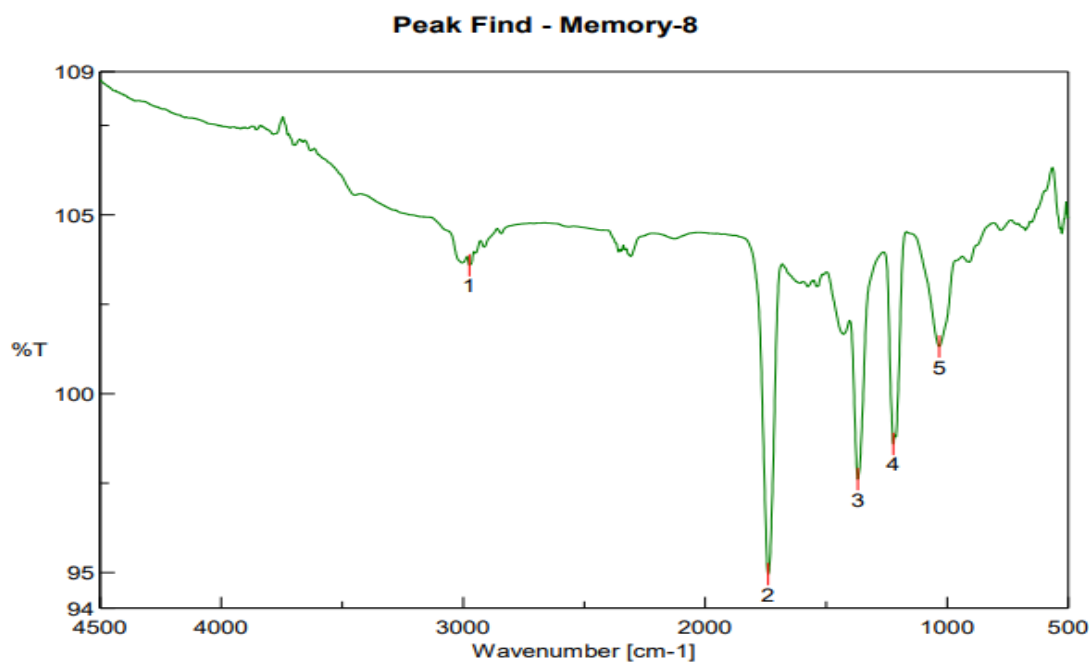


FIG 5.1: FTIR for duckweed

From the above graph peak level at different position indicates the functional groups present in it. 2973.7 shows the presence of C-H stretch with variable intensity. 1739.48 shows the presence of C=O Ketone with strong intensity. 1369.21 shows the presence of C-O stretch with strong intensity. 1222.65 shows the presence of Alkyl ketone stretch with strong intensity. 1032.69 shows the presence of C-F stretch with strong intensity.

5.1.2 FESEM

Field Emission Scanning Electron Microscopy, or FESEM for short, is a high-resolution imaging method that probes the nanoscale structure and surface morphology of materials. The morphology of biodiesel, specifically the size, shape, and distribution of the particles or aggregates present, can be understood through the application of FESEM analysis.

Using an electron beam, the FESEM device examines the sample's surface. Fine surface details can be seen in images produced with a very high resolution and magnification by detecting secondary electrons emitted from the surface.

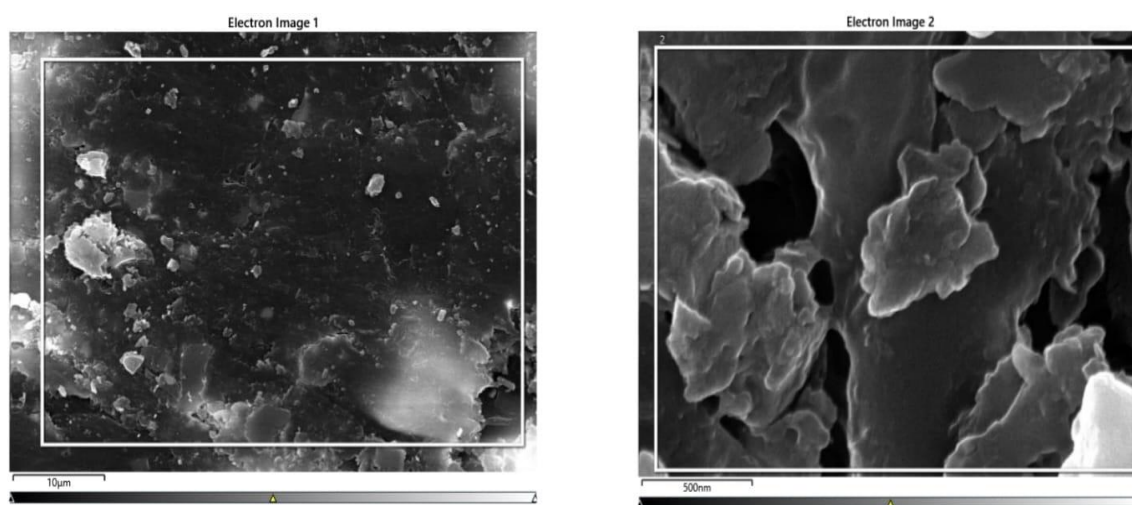


FIG 5.2: SEM images of duckweed

Given that duckweed is a plant, it is likely that the powder's particles are asymmetrical and uneven. The shapes and distribution of particle sizes are seen in the FESEM image. The morphology of the cell wall is visible in the FESEM image as the resolution is good enough.

5.1.3 EDAX

EDAX is an analytical method that measures the energy of X-rays released when electrons interact with a sample. It is frequently used in conjunction with scanning electron microscopy (SEM) to ascertain the elemental composition of a sample. It is utilized for elemental analysis in a variety of applications, such as geology and materials characterization.

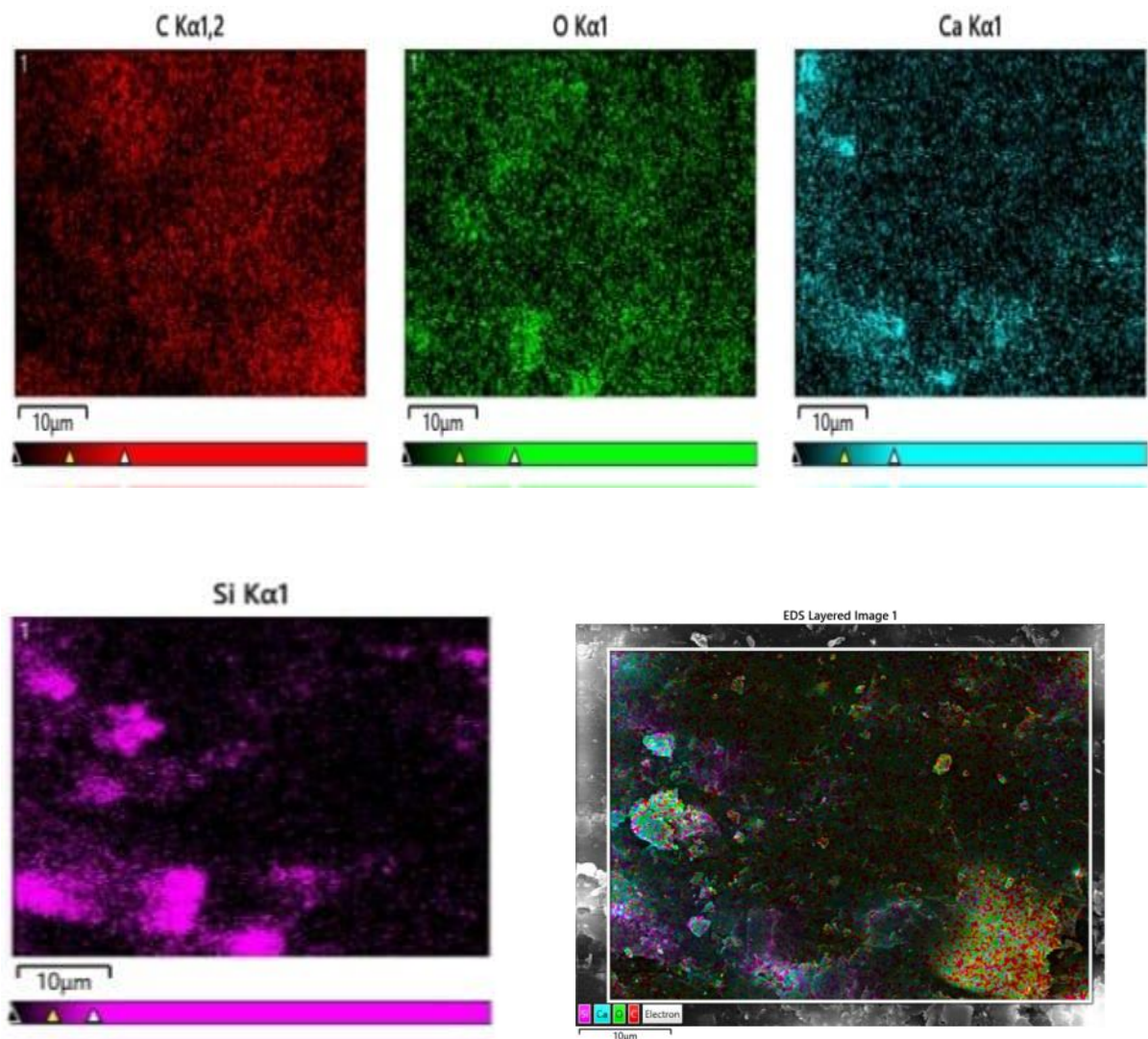


FIG 5.3: EDAX for duckweed

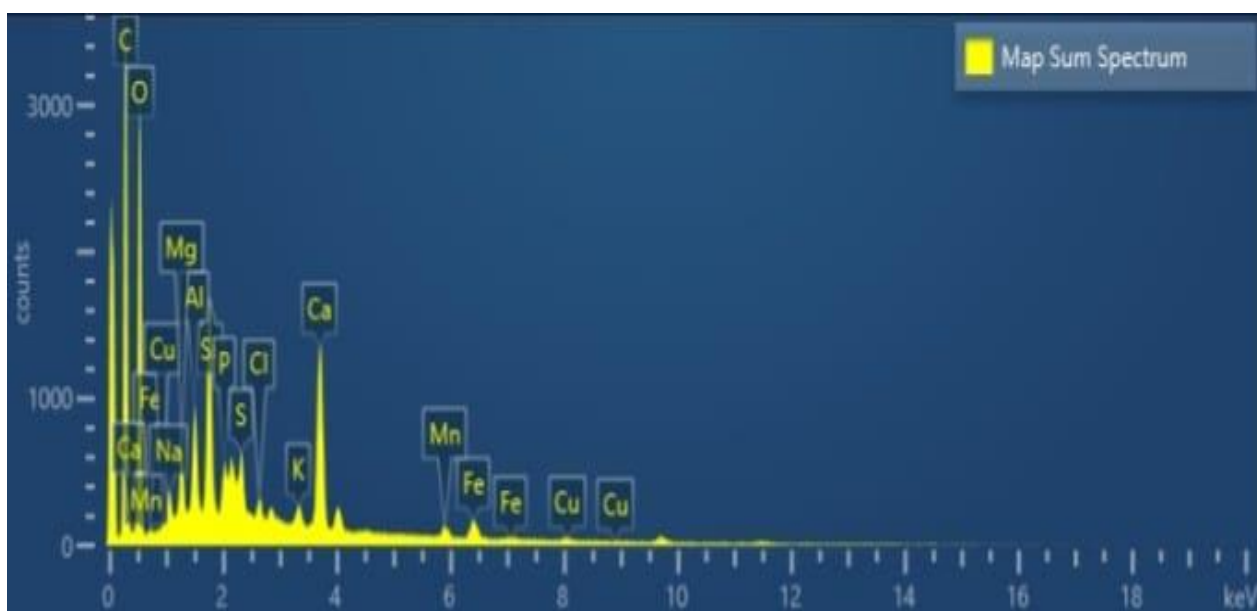


FIG 5.4: EDAX mapping for duckweed

TABLE 5.1 ELEMENTAL COMPOSITION IN DUCKWEED

Map Sum Spectrum Element	Line Type	Weight %	Weight % Sigma	Atomic %
C	K series	53.31	0.58	64.01
O	K series	33.72	0.47	30.40
Na	K series	0.64	0.05	0.40
Mg	K series	1.01	0.04	0.60
Al	K series	1.30	0.04	0.70
Si	K series	2.49	0.05	1.28
P	K series	0.36	0.04	0.17
S	K series	0.61	0.04	0.27
Cl	K series	0.36	0.03	0.15
K	K series	0.36	0.03	0.13
Ca	K series	3.88	0.07	1.40
Mn	K series	0.55	0.05	0.14
Fe	K series	1.07	0.06	0.28
Cu	K series	0.32	0.08	0.07
Total		100.00		100.00

The above table shows the list of chemical elements present in duckweed. This result is obtained as the result of EDAX.

5.1.4 XRD

An effective method in materials science for figuring out a crystalline material's atomic and molecular structure is X-ray diffraction (XRD) examination. Indexing the diffraction peaks to ascertain the lattice parameters and crystal symmetry, fine-tuning the parameters to match the experimental data.

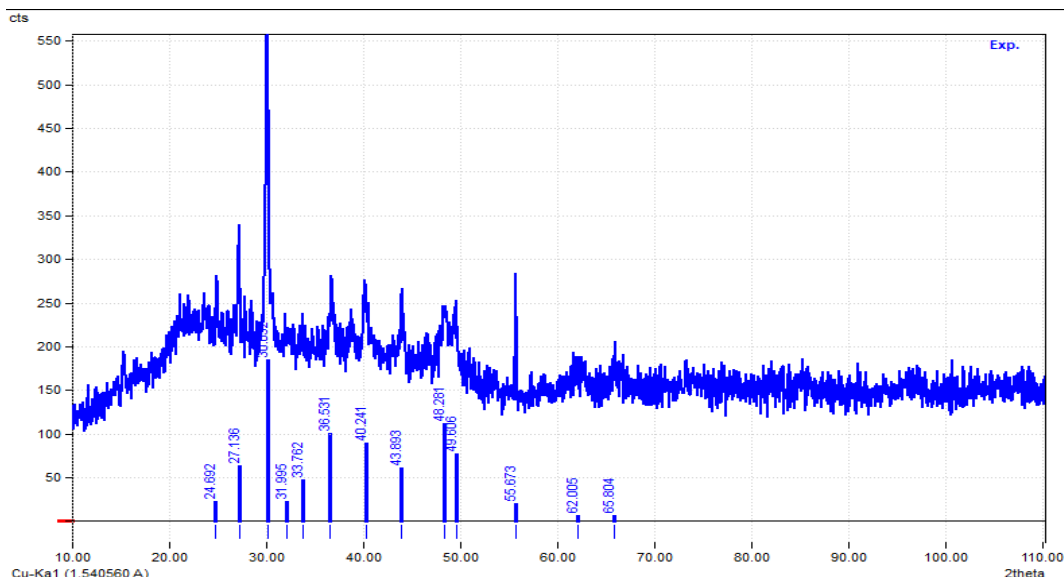


FIG 5.5: XRD for duckweed

Peak List						
No.	2theta [°]	d [Å]	I/I0 (peak height)	Counts (peak area)	FWHM	
1	24.69	3.6026	184.33	21.93	0.2356	
2	27.14	3.2834	372.78	62.73	0.3333	
3	30.05	2.9711	1000.00	183.64	0.3637	
4	32.00	2.7949	58.37	21.90	0.7431	
5	33.76	2.6526	80.19	45.86	1.1328	
6	36.53	2.4577	173.13	100.02	1.1443	
7	40.24	2.2392	215.83	88.07	0.8082	
8	43.89	2.0610	155.59	60.33	0.7680	
9	48.28	1.8834	174.14	111.08	1.2531	
10	49.61	1.8362	107.92	75.75	1.2530	
11	55.67	1.6496	318.36	18.93	0.1178	
12	62.00	1.4955	97.81	5.82	0.1178	
13	65.80	1.4180	99.33	5.91	0.1178	

Integrated Profile Areas		
Based on calculated profile		
Profile area	Counts	Amount
Overall diffraction profile	585319	100.00%
Background radiation	499298	85.30%
Diffraction peaks	86021	14.70%
Peak area belonging to selected phases	0	0.00%
Unidentified peak area	86021	14.70%

Peak Residuals		
Peak data	Counts	Amount
Overall peak intensity	802	100.00%
Peak intensity belonging to selected phases	445	55.43%
Unidentified peak intensity	357	44.57%

FIG 5.6: Peak report of XRD

XRD spectrum indicated in the above graph shows well defined and intense peak at 30.02 indicates the emergence of crystalline.

5.2 CHARACTERIZATION OF BIODIESEL

5.2.1 FTIR.

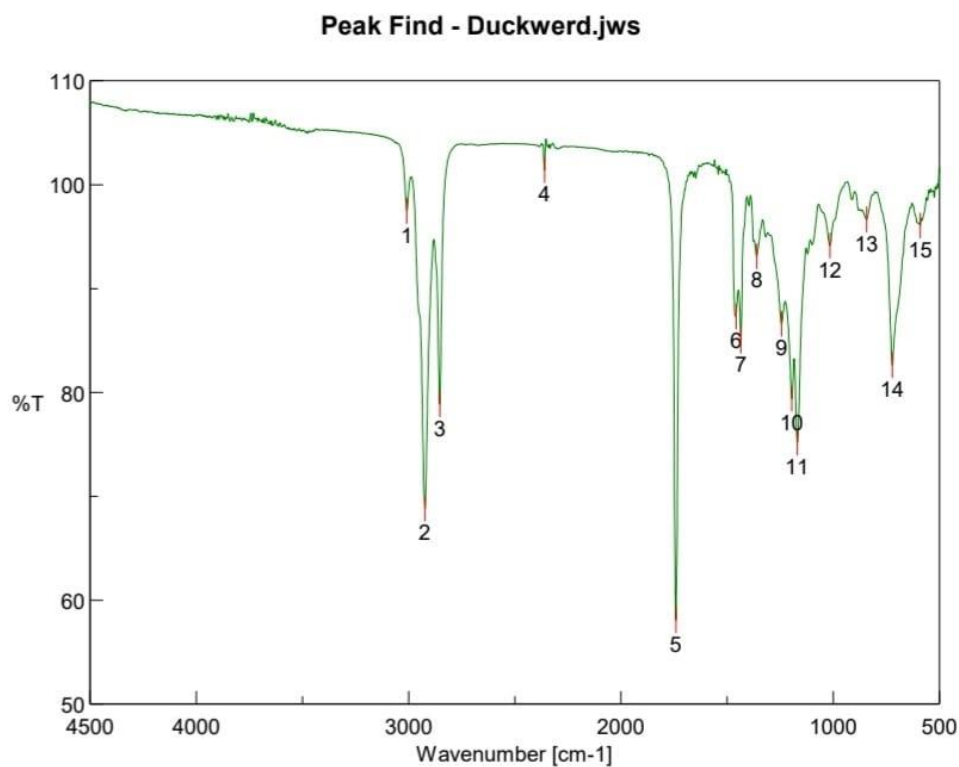


FIG 5.7: FTIR for biodiesel

[Result of Peak Picking]

No.	Position	Intensity	No.	Position	Intensity
1	3008.41	97.4442	2	2923.56	68.8111
3	2853.17	78.8325	4	2360.44	101.385
5	1741.41	58.0784	6	1457.92	87.2999
7	1435.74	84.9783	8	1361.5	93.1005
9	1243.86	86.605	10	1195.65	79.3903
11	1169.62	75.1517	12	1016.3	94.1431
13	843.704	96.6588	14	722.211	82.6245
15	591.075	96.0583			

FIG 5.8: Peak report of FTIR

From the above graph peak level at different position indicates the functional groups present in it. 3008.41 shows the presence of C-H Stretching group with medium intensity. 2923.56 shows the presence of O-H Stretching with strong broad. 1741.41 shows the presence of C=O stretching (Esters) with strong intensity. 1169.62 shows the presence of C-O stretching (Esters) with strong intensity. 591.5 shows the presence of S=O sulfonate stretching with strong intensity.

5.2.2 PROPERTIES OF BIODIESEL

TABLE 5.2 PROPERTIES OF BIODIESEL

S. No	Parameters	Results
1	Density (g/cc)	0.86
2	Kinematic Viscosity @ 40 (°C)	3.8
3	Flash point by PMCC method (°C)	154.2
4	Fire point by PMCC method (°C)	172
5	Pour point by ASTM method (°C)	-4.4
6	Calorific value (MJ/Kg)	35.5

The above table shows the properties of optimized biodiesel. The values of parameters meet the standards according to ASTM D6751-1

5.2.3 GCMS

Gas Chromatography-Mass Spectrometry (GC-MS) analysis is a widely employed method for the evaluation and verification of biodiesel's quality. Typically, specialist software is used to process the results from GC-MS analysis. By comparing the mass spectra of the sample components with those of established standards or databases, this program assists in the identification of the various chemicals included in the biodiesel sample. Additionally, it measures how much of each ingredient is present in the sample. By giving manufacturers precise information about the chemical makeup of biodiesel, GC-MS analysis enables them to monitor product quality and optimize production procedures.

a. GCMS ANALYSIS OF DUCKWEED OIL

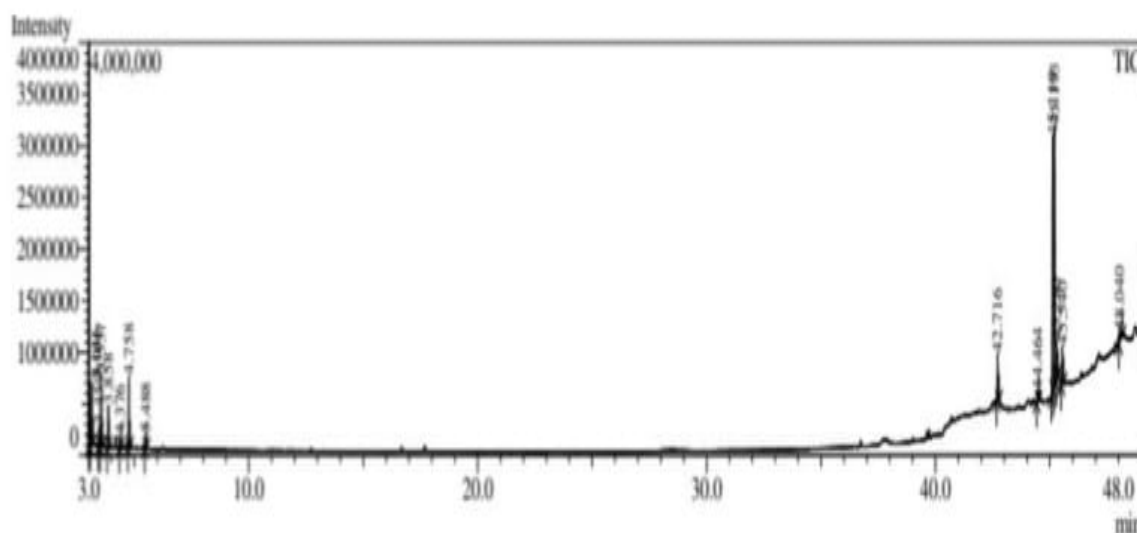


FIG 5.9: GCMS for duckweed oil

Duckweed oil can be identified and its constituent parts quantified using a widely used technique called gas chromatography-mass spectrometry (GC-MS) analysis. Duckweed oil is made from the microscopic aquatic plants known as duckweed. Its composition, which consists of fatty acids, triglycerides, and other substances, may be determined via GC-MS analysis. Sample Preparation: Before being subjected to GC-MS analysis, duckweed oil must be appropriately prepared. This usually entails employing a suitable solvent, like hexane or ethanol, to extract the oil from the biomass of duckweed. After extraction, the oil is concentrated and cleaned to get rid of contaminants.

TABLE 5.3 PEAK REPORT OF GCMS FOR DUCKWEED OIL

Peak#	R.Time	Area	Area%	Name
1	3.306	1108362	4.34	Ethylbenzene
2	3.141	1269472	4.98	BENZENE, 1,2-DIMETHYL-
3	3.459	362705	1.42	BENZENE, 1,2-DIMETHYL-
4	3.537	1475516	5.78	2-PENTANETHIOL, 2-METHYL-
5	3.858	837886	3.28	2-Aminoethanol, N, O-diacetyl
6	4.376	227273	0.89	Pentane, 3-ethyl-2,4-dimethyl-
7	4.758	1536711	6.02	3-Hexen-2-one
8	5.488	291651	1.14	BENZENE, 1,2,3-TRIMETHYL-
9	42.716	1017351	3.99	Glycidol Stearate
10	44.464	243164	0.95	9,12-Octadecadienoic acid(Z,Z)-, 2,3-dihydrate
11	45.119	6725170	26.36	14-METHYL-8-HEXADECYN-1-OL#
12	45.193	8209109	32.17	9-Octadecenoic acid, 1,2,3-propanetriyl ester
13	45.280	866450	3.40	Cis, cis-7, 10,-Hexadecadienal
14	45.540	890324	3.49	Glycidol Stearate
15	48.040	454572	1.78	9-Octadecenoic acid(Z)-, 2,3-dihydroxypropane
		25515716	100.00	

The above GCMS result for duckweed oil demonstrates the presence of octadecadienoic acid and other components present in it.

b. GCMS ANALYSIS OF DUCKWEED BIODIESEL

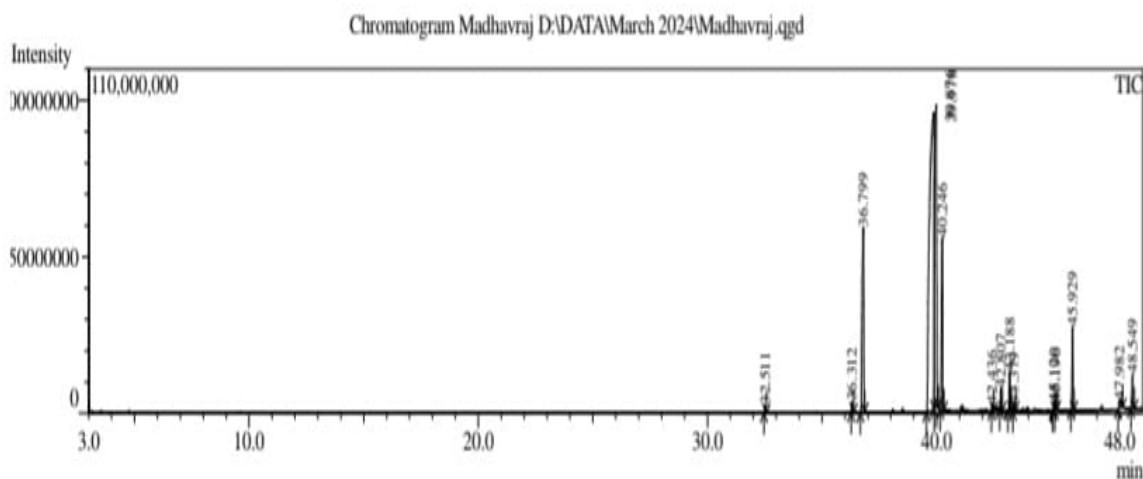


FIG 5.10: GCMS for biodiesel

TABLE 5.4 PEAK REPORT OF GCMS FOR BIODIESEL

Peak#	R.time	Area	Area%	Name
1	32.511	5531425	0.22	TETRADECANOIC ACID, METHYL ESTER
2	36.312	8468037	0.34	9-Hexadecanoic acid, methyl ester, (Z)-
3	36.799	264521271	10.61	HEXADECANOIC ACID, METHYL ESTER
4	39.876	1286550895	51.59	Methyl 10-trans,12-cis-octadecadienoate
5	39.978	560926666	22.49	9-OCTADECENOIC ACID, METHYL ESTER
6	40.246	185679793	7.45	Methyl stearate

7	42.436	4179393	0.17	OXIRANEOCTANOIC ACID,3-OCTYL
8	42.807	16961138	0.68	Cis-11-Eicosenoic acid, methyl ester
9	43.188	30451199	1.22	EICOSANOIC ACID, METHYL ESTER
10	43.379	4863741	0.20	OXIRANEOCTANOIC ACID, 3-OCTYL-,
11	45.120	6042643	0.24	BICYCLO[10.1.0]TRIDEC-1-ENE
12	45.196	5892554	0.24	9-Octadecenoicacid, 1,2,3-propanetriyl ester
13	45.929	68391835	2.74	Methyl 20-methyl-heneicosanoate
14	47.982	14622919	0.59	9,12-OCTADECADIENOIC ACID (Z,Z)-
15	48.549	30878970	1.24	TETRACOSANOIC ACID, METHYL ESTER
		2493962479	100	

The above table and peaks present in the graph demonstrates the presence of methyl ester which is the main component of biodiesel and confirms whether the oil is converted into biodiesel and this is attained by transesterification process. This confirms the presence of methyl esters and acids present in the duckweed biodiesel. As duckweed oil is converted to biodiesel, the triglycerides it contains undergo transesterification, which yields these methyl esters of fatty acids.

5.2.4 INTERNAL COMBUSTION ENGINE STUDY

Internal combustion engines (ICEs) running on biodiesel fuel would be the subject of a study that would examine a number of topics including performance, emissions, and engine optimisation. The IC engine study for various blends [B15, B30] are studied.

Brake thermal efficiency (BTE) is a crucial metric for assessing an internal combustion engine's efficiency. It calculates the usable work production in relation

to the engine's total energy input. When evaluating the performance of engines using various fuels, BTE is a crucial parameter to consider.

Calculate the amount of heat created when the biodiesel in the engine burns. This entails taking the biodiesel fuel's energy content and combustion efficiency into account. Incomplete combustion and heat loss to the environment are losses that are taken into account by the combustion efficiency. Utilizing the torque and rotational speed, compute the engine's work output. With the engine displacement volume and the torque-speed connection, one may compute the amount of work performed in a single engine cycle. When operating under similar conditions, compare the brake thermal efficiency of conventional diesel fuel and biodiesel fuel. The relative effectiveness of utilizing biodiesel as a fuel in internal combustion engines is revealed by means of this comparison. An evaluation of the mechanical efficiency of biodiesel usually entails comparing the performance of machinery or engines running on biodiesel fuel to those running on regular diesel fuel. The purpose of this investigation is to determine how biodiesel affects the system's mechanical efficiency.

Examining the torque production of an engine or piece of equipment when running on biodiesel fuel as opposed to regular diesel fuel is known as torque analysis for biodiesel. Because it indicates the rotating force produced by the engine's drivetrain, torque is an important metric for evaluating an engine's mechanical performance. To see the engine's torque output over its operating range, plot torque curves for both conventional diesel fuel and biodiesel.

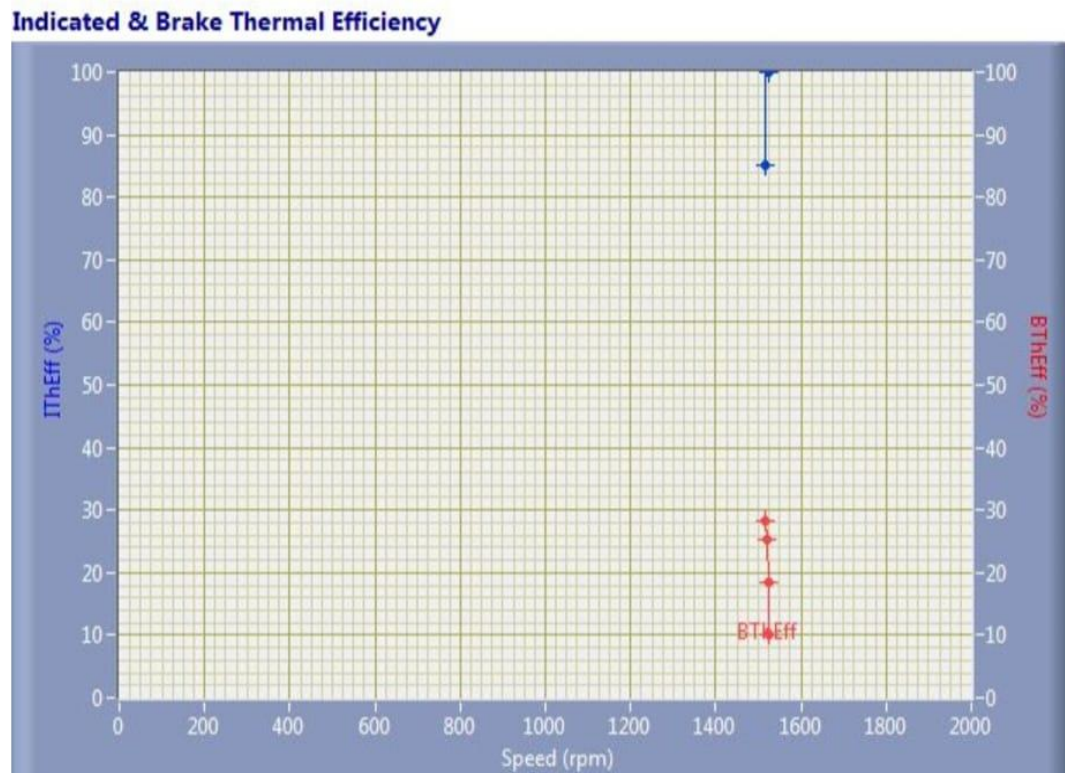


FIG 5.11: Brake thermal efficiency for b15

TABLE 5.5 BRAKE THERMAL EFFICIENCY DATA FOR B15

Speed (rpm)	Load (kg)	IThEff (%)	BThEff (%)
1524.00	0.59	0.00	0.00
1524.00	0.59	149.42	5.87
1526.00	3.12	85.36	15.55
1524.00	6.13	0.00	0.00
1520.00	6.12	113.69	23.39
1518.00	9.07	91.14	28.14

Indicated and brake thermal efficiency for B15 blends is studies for different loads ranging from 0.59 Kg to 9.07 Kg. In which the percentage efficiency of IThEff decreases with increase in load whereas in BThEff the percentage efficiency increases with increase in load. At 1500 rpm, BThEff is around 30%. At 1500 rpm

IThEff is around 85%. As speed increases BThEff and IThEff increases. The brake thermal efficiency increases as the speed (rpm) increases from 200 rpm to 1000 rpm. The brake thermal efficiency then starts to decrease as the speed (rpm) increases from 1000 rpm to 1800 rpm. The highest brake thermal efficiency observed in the graph is around 28.20% at 1518 rpm.

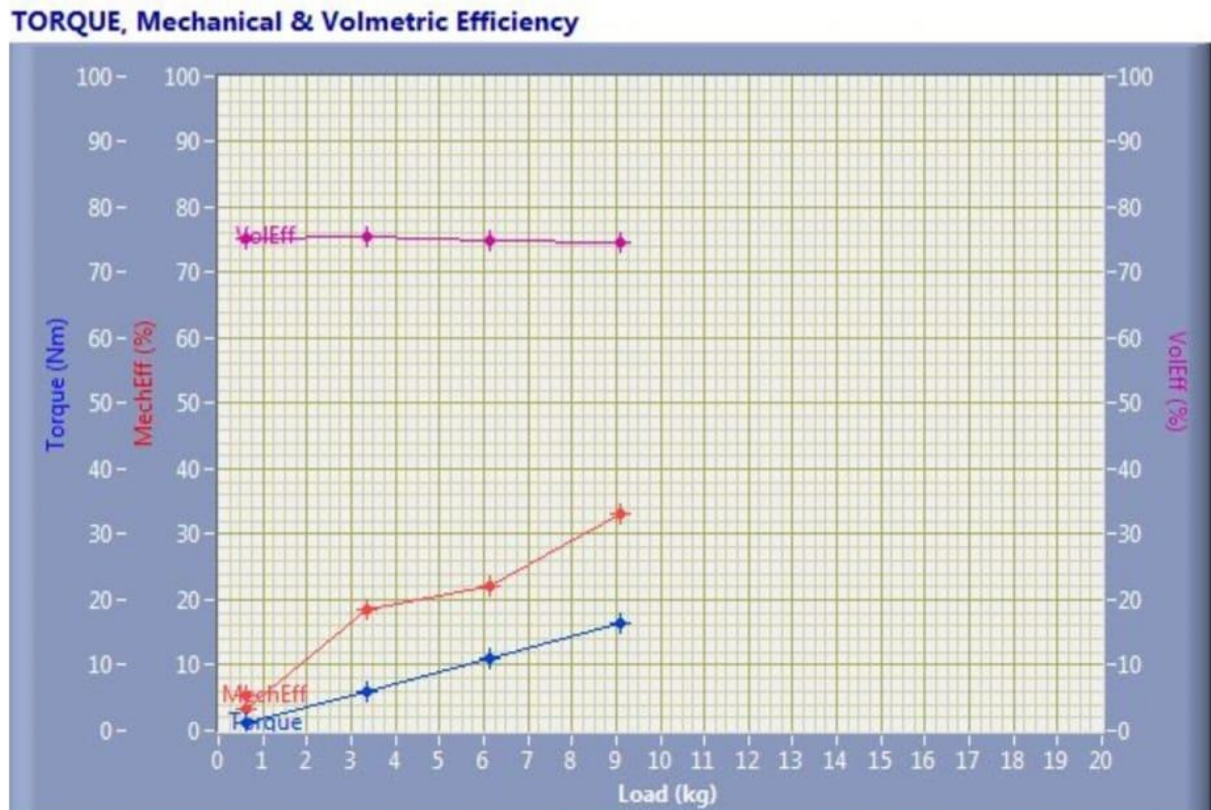


FIG 5.12: IC engine study for b15

TABLE 5.6 IC ENGINE STUDY DATA FOR B15

Speed (rpm)	Load (Kg)	Torque (Nm)	Mech Eff. (%)	Vol Eff. (%)
1525.00	0.61	1.11	5.30	75.32
1524.00	0.61	1.11	3.41	75.36
1523.00	3.35	6.08	18.56	75.46
1521.00	6.12	11.12	22.16	74.88
1518.00	9.09	16.49	33.14	74.72

The B15 fuel appears to produce good torque, but the mechanical and volumetric efficiencies are low. This suggests that the engine is losing a significant amount of power due to friction and other internal losses.

Indicated & Brake Thermal Efficiency

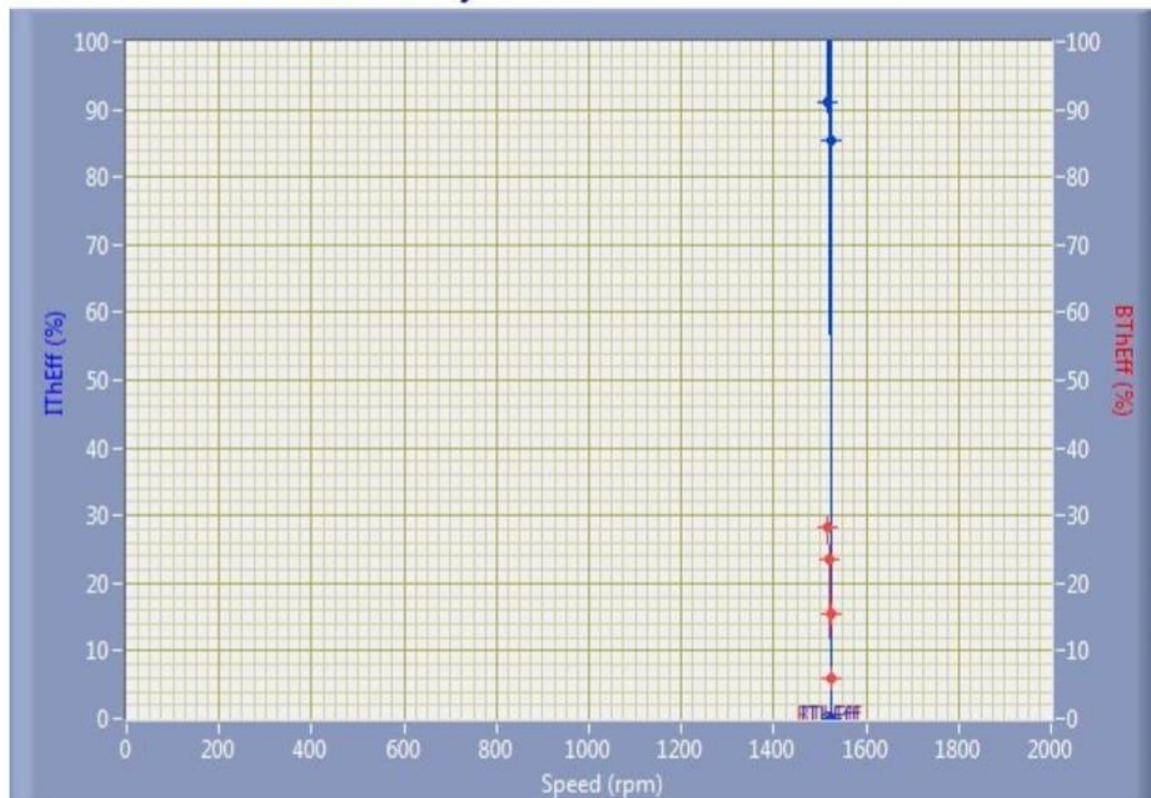


FIG 5.13: Brake thermal efficiency for b30

TABLE 5.7 BRAKE THERMAL EFFICIENCY FOR B30

Speed (rpm)	Load (kg)	IThEff (%)	BThEff (%)
1525.00	0.61	191.85	10.18
1524.00	0.61	298.80	10.19
1523.00	3.35	99.98	18.55
1521.00	6.12	114.58	25.39
1518.00	9.09	85.08	28.20

Indicated and brake thermal efficiency for B30 blends is studies for different loads ranging from 0.61 Kg to 9.09 Kg. In which the percentage efficiency of IThEff

decreases with increase in load whereas in BThEff the percentage efficiency increases with increase in load.

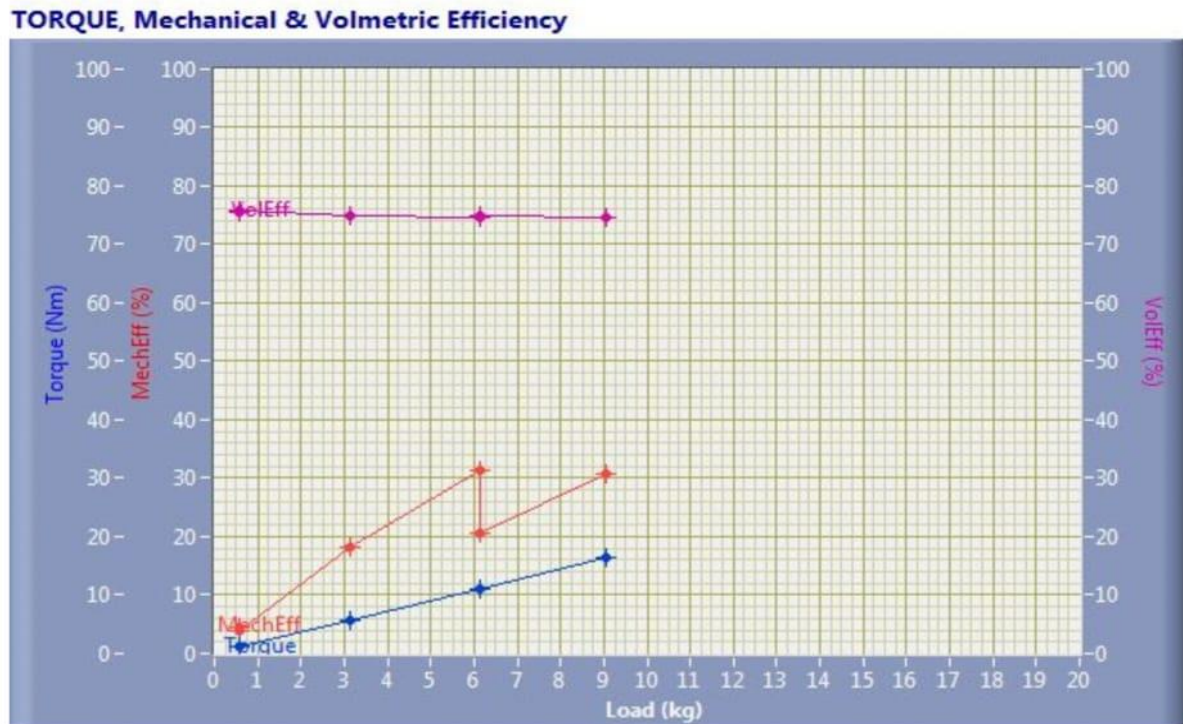


FIG 5.14: IC ENGINE STUDY FOR B30

TABLE 5.8 IC ENGINE STYDY DATA FOR B30

Speed (rpm)	Load (Kg)	Torque (Nm)	Mech Eff. (%)	Vol Eff. (%)
1524.00	0.59	1.08	4.55	75.51
1524.00	0.59	1.07	3.93	75.71
1526.00	3.12	5.65	18.22	74.98
1524.00	6.13	11.13	31.34	74.74
1520.00	6.12	11.10	20.57	74.82
1518.00	9.07	16.46	30.88	74.57

Torque is increasing as engine speed increases. At a low engine speed (around 1 on the x-axis), torque is around 20 Nm. As the engine speed increases to around

19 on the x-axis, torque increases to around 90 Nm. Mechanical efficiency is also increasing as engine speed increases. At a low engine speed (around 1 on the x-axis), mechanical efficiency is around 10%. As the engine speed increases to around 19 on the x-axis, mechanical efficiency increases to around 90%. Volumetric efficiency is also increasing as engine speed increases. At a low engine speed (around 1 on the x-axis), volumetric efficiency is around 10%. As the engine speed increases to around 19 on the x-axis, volumetric efficiency increases to around 80%. B30 biodiesel appears to be a viable alternative fuel with promising performance characteristics, especially at higher engine speeds

5.2.5 EMISSION ANALYSIS FOR VARIOUS BLENDS

Emissions from internal combustion engines using biodiesel are essential for assessing the fuel's environmental impact and contrasting it with regular diesel. It describes the steps taken to assess emissions while using biodiesel fuel in an engine. It indicates the parameters of the emissions that were measured, such as hydrocarbons (HC), carbon monoxide (CO), particulate matter (PM), nitrogen oxides (NO_x), and other pollutants. Give specifics about the test parameters, such as the load, engine speed, and composition of the biodiesel blend. Several factors, including feedstock, production technique, and additives, can affect the smoke density of biodiesel. Owing to its higher oxygen content and cleaner burning properties, biodiesel often emits less smoke than traditional diesel fuel. The precise blend, the conditions of combustion, and the testing methodology would all affect the smoke density values for biodiesel, though.

TABLE 5.9 EMISSION ANALYSIS FOR B15

Name of the gas	Load	Load	Load	Load
	0	3	6	9
CO	0.01	0.02	0.05	0.06
HC	1	1	2	3
CO ₂	0.40	0.5	1.20	0.8
O ₂	20.40	19.96	19.11	18.68
NO _x	23	25	53	62
SMOKE DENSITY	4.3	47.8	52.8	58.3

TABLE 5.10 EMISSION ANALYSIS FOR B30

Name of the gas	Load	Load	Load	Load
	0	3	6	9
CO	0.01	0.02	0.04	0.04
HC	2	2	3	3
CO ₂	0.70	0.90	1.20	1.9
O ₂	19.95	19.89	19.07	18.65
NO _x	5.7	58	62	64.32
SMOKE DENSITY	3.4	46.8	56.8	64.30

The above tables shows the amount of gas emitted for different loads of biodiesel input. It is done for two blends i.e. B15 and B30. The emission values of various gases were studied through the fine gas detector. The CO gas emission for B15 and B30 ranges within 0.01 to 0.06 and 0.01 to 0.04 for different loads respectively. Whereas the CO₂ gas emission for B15 and B30 ranges from 0.40 to 1.2 and 0.70 to 1.9 for the different loads respectively. As the emission range meets the standards it can be used as an alternative ecofriendly source.

CHAPTER 6

SUMMARY AND CONCLUSION

6.1 SUMMARY

Oil was extracted from duckweed using Solvent extraction technique by using various solvents like Hexane, Isopropyl alcohol and Ethanol. The extraction was carried out in Soxhlet apparatus. It was found that the best yield was obtained with the Isopropyl alcohol. The yield for different solvents were 28%,72%,54% respectively and the solvent is chosen on the basis of properties like density and specific gravity which plays a vital role for producing quality biodiesel. The characteristics of dried duckweed was also studied using FTIR, FESEM, EDAX and XRD. It was found that it has the essential to produce biodiesel. In XRD analysis it showed that the sample was crystalline.

Biodiesel was produced by transesterification process, In which the oil to methanol ratio was finalized on the basis of yield further it was optimized using different parameters such as temperature, time to obtain the optimized bio diesel. The biodiesel experiment was carried out using magnetic stirrer as it mixes uniformly. The optimized condition had the reaction time about 40 minutes, 45°C, catalyst [2 Wt %]. The properties of bio diesel was determined by ASTM and PMCC method. The properties are fire point, flash point, calorific value, density and kinematic viscosity. Internal combustion engine test and emission test was done for two blends B15 and B30 respectively. In engine test the brake thermal analysis, air flow, torque efficiency, mechanical efficiency was studied. In emission analysis various gases at different loads were analyzed using fine gas analyzer. The other characterization for bio diesel such as FTIR, GCMS was studied and it shows the presence of Ester and other functional groups. In GCMS it indicated the presence of various acids and methyl esters that shows that the biodiesel can be used as an alternative fuel

6.2 CONCLUSION

We conclude that the oil extracted from duckweed can be converted to duckweed bio diesel and it can be used in Internal combustion engines without any modifications. In engine combustion study the characteristics like brake thermal

efficiency, torque, mechanical and volumetric analysis were studied. BThEff the percentage efficiency increases with increase in load. At 1500 rpm, BThEff is around 30%. At 1500 rpm IThEff is around 85%. As speed increases BThEff and IThEff increases. The brake thermal efficiency increases as the speed (rpm) increases from 200 rpm to 1000 rpm. The brake thermal efficiency then starts to decrease as the speed (rpm) increases from 1000 rpm to 1800 rpm. The highest brake thermal efficiency observed in the graph is around 28.20% at 1518 rpm. In emission analysis gases like carbon monoxide, oxygen, carbon dioxide, nitrogen oxide and smoke density was detected using fine gas analyzer for different loads for various blends such as B15 and B30. The process can be scaled as future work.

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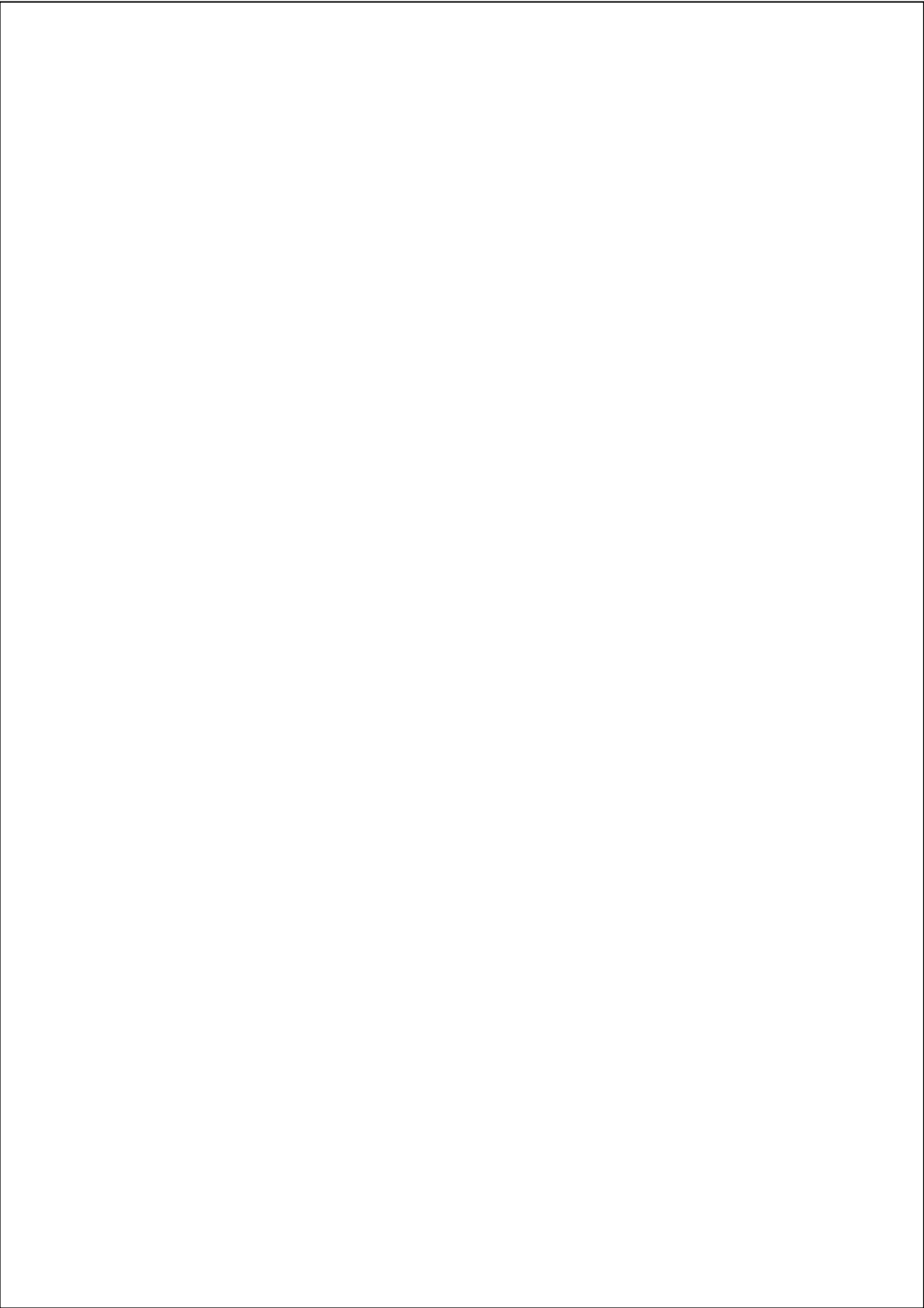
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ABSTRACT

Duckweed (Lemna minor) has garnered significant attention in recent years as a versatile resource with potential applications in biofuel production and scientific research. Duckweed is a floating aquatic plant that is abundant in ponds, lakes and other water resources. The duckweed oil was extracted by using Soxhlet apparatus with 3 different solvents such as Ethanol, Isopropyl alcohol and Hexane. It was found that the best yield was obtained with the Isopropyl alcohol. The obtained oil was converted to bio diesel by transesterification using homogenous catalyst, sodium hydroxide. The properties of duckweed biodiesel were determined according to ASTM D6751 for Free fatty acids, flash point, fire point, pour point, calorific value, density and kinematic viscosity. Engine test was carried with internal combustion engine and the total fuel consumption, specific fuel consumption, brake thermal efficiency and indicated thermal efficiency was found for B15 and B30 blends. Hence duckweed oil can be successfully converted to bio diesel and can be used in internal combustion engines without any modifications.

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LIST OF ABBREVIATIONS

NaOH	Sodium Hydroxide
KOH	Potassium Hydroxide
FAME	Fatty acid methyl ester
³⁴ FTIR	Fourier transform infrared radiation
FESEM	Field emission scanning electron microscope
⁴⁷ XRD	X ray diffraction
GCMS	Gas chromatography mass spectroscopy
ASTM	American society of testing materials
PMCC	Pensky-martenz closed cup
HCL	Hydrochloric acid
EDAX	Energy dispersive x ray analysis
SV	Saponification value
AV	Acid value
MW	Molecular weight

INTRODUCTION

1.1 BACKGROUND.

An investigation of the features of duckweed and its potential use for biofuel generation, along with the reasons behind it, usually comprise the background of the project. The following is a broad overview of the project's background.

Growing Need for Sustainable Energy Sources: Researching alternate and sustainable energy sources is becoming more and more important as worries about climate change and the depletion of fossil resources grow. As a viable way to lessen reliance on non-renewable resources and greenhouse gas emissions, biofuels—which come from renewable plant sources—have gained popularity and **Challenges in Biofuel Production:** Traditional biofuel crops, such as corn and sugarcane, have limitations in terms of land and water use, competition with food crops, and the potential for environmental degradation. As a result, there is a need to identify and develop more efficient and environmentally friendly sources of biomass for biofuel production.

The remarkable pace of growth and high starch and fat content of duckweed have made it a viable option for the manufacture of biofuel. A few of the drawbacks of conventional biofuel crops are addressed by these qualities, which make it a desirable option for the manufacture of bioethanol and biobutanol.

Duckweed provides further environmental benefits, including the ability to eliminate excess nutrients from water bodies, such as phosphate and nitrogen. This improves water quality and helps to alleviate problems like algal blooms. **Scientific Research and Model Organism:** Beyond its biofuel potential, duckweed is a valuable model organism for genetic and molecular studies. Its relatively simple genetic makeup allows researchers to investigate various biological processes and develop insights into plant genetics and physiology.

Different species and strains of duckweed are employed in tests based on the objectives of the study. The usual home for these strains is a collection of cultures. **Growth Media:** Duckweed cannot be grown without nutrient solutions or growth

media. Micronutrients, potassium, phosphorus, and nitrogen are among the important nutrients found in these solutions.

Duckweed is cultivated in ponds, tanks, or other regulated aquatic environments. Plastic, concrete, or liners may be needed for these systems' construction. Monitoring equipment for water quality is necessary to maintain ideal growth circumstances. This equipment includes turbidity metres, pH metres, and nutrient testing kits.

1.2 SOXHLET EXTRACTION.

There are various methods to extract oil from sources but when it comes to extracting aquatic plants the density differs from the other sources as the raw material has more starch and lipids content. Oil and lipid molecules can be extracted from a variety of botanical sources using the commonly used soxhlet extraction process. The other method is mechanical extraction which is widely used for extracting oil from coconut shells and groundnut but in this case solvent extraction is adopted. By continuously cycling solvents, target chemicals can be effectively extracted from solid matrices using the Soxhlet extraction technique. The extraction solvent is heated to a high temperature, vaporises, condenses, and is then circulated through the sample until the desired extraction is obtained. The success of this process depends on the solubility of the target chemicals in this solvent. Oil from seeds, nuts, plant tissues, and other biological materials can be extracted via Soxhlet extraction, which has an abundance of uses. With its excellent extraction efficiency, ease of use, and capacity for handling big samples, it has many benefits. Ecological restoration efforts by harnessing duckweed's nutrient-absorbing capabilities. Picking an appropriate organic solvent means finding one that is both immiscible with water and effectively dissolves the target compound, in this case, oil. Typical solvents for chemical extractions can be a combination of solvents, hexane, ethanol and isopropyl alcohol.

The solvent flask is positioned underneath the extraction chamber of the Soxhlet extraction device after it has been built. The solvent in the flask vaporises when heated, rising into the condenser as a result. After being cooled by the condenser, the vapour condenses back into liquid and returns to the extraction chamber. It

starts to extract the oil from the solid sample in the thimble as the liquid solvent builds up in the extraction chamber. A continuous cycle is used in the Soxhlet extraction process: when the solvent in chamber the extraction chamber reaches a specific level, it syphons into the solid sample, extracts the oil, and then, because of the temperature difference, drops back into the flask. For a lengthy length of time, this cyclic process keeps the solvent flowing through the solid sample, thereby extracting the oil. Even in situations when the chemical of interest is present in relatively low concentrations in the solid material, the Soxhlet extraction process is intended to guarantee complete extraction of the component. Over time, as the extracted oil dissolves in the circulating solvent, it builds up in the flask. The oil-solvent mixture flask is taken out of the device once a enough amount of oil has been extracted, ending the extraction process. The extracted oil must be separated from the solvent in the following step. This is usually accomplished by evaporation, in which the extracted oil is left behind after the solvent evaporates. The solvent can be effectively removed using methods like basic distillation or rotary evaporation. Oils, lipids, and other organic substances are frequently extracted from solid materials in laboratories using the Soxhlet extraction method. It has a number of benefits, including as excellent extraction efficiency and batch processing capabilities. But it can take a while, and you have to be very careful about safety, especially when handling organic solvents.

1.3 TRANSESTERIFICATION.

The transesterification reaction required to produce it can be run on nearly any size, from a small laboratory scale using a few litres of oil to a huge industrial scale that can produce millions of litres of biodiesel annually. Nonetheless, the direct use of vegetable oils and oil mixes is widely regarded as impracticable, despite the potential benefits of commercialization. Some of its most evident disadvantages include its high viscosity, fatty acid composition, and free fatty acid concentration; other disadvantages include gum production from oxidation and polymerization during storage and burning; and carbon deposits and thickening of lubricating oil. due to the finite supply of fossil fuels, an appealing fuel. Biodiesel is synthesised chemically from vegetable and animal fats, which are renewable

feedstock, and monoalkylesters of long chain fatty acids. By reacting oil or fat with a monohydric alcohol in the presence of a catalyst, a process known as transesterification yields the matching monoalkyl esters. With sodium hydroxide acting as an alkaline catalyst, this article presents experimental results on the synthesis of fatty acid methyl esters from sunflower oil. These vegetable oils were used to generate biodiesel, and the factors influencing its yield and properties were investigated. A three-hour reaction period, a 0.5–1.5 weight percent catalyst concentration, and a 1:3–1:9 oil-to-methanol molar ratio were the variables that were examined. The best yield % was found using a based on the findings obtained.

1.3.1 CATALYST.

Catalysts are essential to the manufacturing of biodiesel because they aid in the transesterification reaction, which changes triglycerides—which are present in oils and fats—into fatty acid methyl esters (FAMES), which are the main ingredients of biodiesel. Typically, triglycerides and an alcohol (like methanol or ethanol) are reacted in the presence of a catalyst during the transesterification reaction.

Catalysts can be categorized into various types based on their nature, mechanism, and application. Here are some common types of catalysts:

a. Homogeneous Catalysts:

- Sodium Hydroxide (NaOH): A strong base commonly employed in biodiesel production.
- Potassium Hydroxide (KOH): Another strong base often used as a catalyst.

b. Heterogeneous Catalysts:

- Solid Base Catalysts: Examples include calcium oxide (CaO) and barium oxide (MgO), which are solid catalysts.
- Enzymes: Lipases are biological catalysts that can be used for enzymatic transesterification in biodiesel production.

c. Acid Catalysts (for acid-catalyzed transesterification):

- Sulfuric Acid (H₂SO₄): Utilized in the acid-catalyzed method, particularly for low-quality feedstocks.

d. Enzymatic Catalysts:

Lipases: These enzymes can catalyze transesterification reactions in a specific and environmentally friendly manner.

The choice of catalyst depends on factors such as feedstock quality, reaction conditions, and desired product purity. Homogeneous catalysts are more commonly used in traditional biodiesel production, while heterogeneous catalysts and enzymes may offer advantages in specific applications or for higher-quality biodiesel production.

1.3.2 METHANOL.

Triglycerides are the main building blocks of vegetable oil, animal fats, and microalgae. Methanol reacts strongly with these compounds to speed up the transesterification process, which produces biodiesel. A greater degree of freedom in catalyst selection and optimization processes is afforded by the vast spectrum of catalysts with which methanol can react, primarily in transesterification reactions. Methanol helps to improve the purity and quality of the final product by reducing unwanted byproducts or side reactions during the biodiesel synthesis process. Environmental advantages come from the usage of methanol to make biodiesel, which lowers greenhouse gas emissions as compared to petroleum fuel.

1.4 BIOFUEL.

As the need for energy rises and non-renewable resources run out, it is imperative that sustainable energy sources be developed. There is a lot of interest in biodiesel as a fossil fuel substitute since it is environmentally benign and sustainable. However, the costly and inefficient previous biodiesel production process has limited its widespread adoption.

Reduction in Greenhouse Gas Emissions: In general, diesel derived from petroleum emits more greenhouse gases than biodiesel. By lowering carbon dioxide and other harmful pollutants that contribute to global warming, it can help moderate climate change. When considering lifecycle greenhouse gas emissions, biodiesel derived from leftover vegetable or animal fats can be substantially less

than petroleum fuel. Diversification of Fuel Sources: There are hazards associated with supply disruptions, geopolitical instability, and price volatility when driving or using energy primarily from petroleum-based fuels. By increasing energy security and lowering reliance on finite fossil fuels, biodiesel provides a means of fuel diversification.

Various renewable resources can be used to make biodiesel, such as animal fats, algae, and even recycled cooking oil. Vegetable oils include soybean, canola, and palm oil. Reducing the demand on non-renewable fossil fuel supplies, these resources can be grown or obtained sustainably and are replenishable.

Encouraging Rural Development and Agriculture: By giving farmers the chance to cultivate certain energy crops or turn agricultural waste into fuel, biodiesel production can boost rural economies. In agricultural regions in particular, this can boost local economies and generate jobs. Better Air Quality: Burning biodiesel results in lower emissions of sulphur oxides, particulate matter, and aromatic hydrocarbons than burning conventional diesel fuel.

Petroleum diesel can be easily replaced with biodiesel, which is a suitable drop-in alternative for diesel engines and infrastructure that are already in place. Mixing different amounts of biodiesel with regular diesel (B5, B20, or greater blends) is a standard procedure that facilitates adoption gradually without requiring major adjustments to fleets of vehicles or infrastructure.

1.4.1 ADVANTAGES OF BIOFUEL.

The production of biofuels has the potential to boost rural economies by generating employment and giving farmers, landowners, and biofuel producers more revenue options. Sustainable land management techniques, rural development, and money generation for agricultural communities can all be achieved through the cultivation of biomass feedstocks and bioenergy crops.

Compatibility with Current Infrastructure: In many cases, existing engines, cars, and transportation infrastructure may be used to blend biofuels like ethanol and biodiesel with conventional petroleum fuels without requiring significant improvements or adjustments. Due to their compatibility, biofuels may be widely

used without requiring large infrastructure investments, allowing for a more gradual shift to renewable fuels.

Advances in biotechnology, agronomy, bioengineering, and process optimisation are fuel by the development and marketing of biofuels. Research and development investments in biofuels help to increase feedstock quality, biomass conversion technologies, and sustainable bioenergy production methods, which benefits the biofuels business as well as adjacent industries.

The production of biofuels on a local or regional scale can lead to energy independence and a decrease in dependency on remote energy sources. Communities may develop value-added goods, eliminate energy losses connected to fuel distribution and supply chains in transportation, and maximise their own renewable energy potential by leveraging locally accessible biomass resources.

1.4.2 Disadvantages of Biofuel.

Competition for resources such as land and water between energy and food crops is one of the main issues with biodiesel production. Land use disputes, deforestation, environmental degradation, and higher food costs might result from large-scale biofuel production that depends on food crops like maize or soybeans.

The significance of developing non-food biomass sources and sustainable feedstock sourcing for biofuel production is emphasised in this article. The proliferation of biofuel feedstock production has the potential to cause alterations in land use, such as clearing forests, destroying natural habitats, and converting natural ecosystems into agricultural land. Soil erosion, biodiversity loss, and disruption of ecosystem services are just a few of the detrimental effects these changes may have on the environment. Plans for land use and sustainable land management are crucial.

A number of variables, including climate, soil quality, agricultural methods, and crop yields, can affect the availability and productivity of biomass feedstocks for the production of biodiesel. In order to meet major energy demands, large land areas must be dedicated to the growth of some bioenergy crops, which may have lower energy outputs than fossil fuels.

Developing high-yielding feedstock varieties, streamlining farming techniques, and boosting agricultural output are crucial approaches to overcome this restriction. The mixture is allowed to separate once the reaction is finished, with the higher-density glycerol at the bottom and the lower-density biodiesel at the top. Improved performance and a high cetane number can be obtained along with cost-effective and time-efficient biodiesel production through the use of the transesterification method. While biodiesel produces fewer emissions than diesel, it still has an impact on the environment because of its production process, especially when it comes from non-renewable sources. Environmental waste management problems can occasionally arise from glycerol overproduction.

1.5 PROPERTIES OF BIOFUEL.

Chemical Composition: The main component of biodiesel is fatty acid methyl esters (FAMES), which are produced from the triglycerides found in animal or vegetable fats. Depending on the feedstock utilised to produce it, biodiesel can have a different precise composition.

Viscosity: In diesel engines, fuel flow and atomization can be impacted by the viscosity of biodiesel, which is generally higher than that of petroleum diesel. However, variables like temperature and feedstock mix affect how viscous of the biodiesel.

Cetane Number: This is a metric used to assess how well diesel fuel ignites. When compared to petroleum diesel, biodiesel typically has a higher cetane number.

Oxidative Stability: When it comes to oxidation and degradation during handling and storage, biodiesel is more vulnerable than petroleum diesel due to its poorer oxidative stability. To extend the shelf life of biodiesel and stop gums and deposits from forming, antioxidants and stabilisers can be added.

Lubricity: Due to its lower concentration of sulphur compounds, which function as lubricants, biodiesel offers superior lubricating qualities than ultra-low sulphur diesel (ULSD). Because biodiesel is more lubricating, it can shield the fuel system.

The temperature at which a fuel releases enough vapours to continue burning once it is ignited is known as its "fire point." Usually, it is greater than the flash point—the temperature at which, in the presence of an ignition source, fuel vapour

can briefly ignite. Because it represents the temperature at which a fuel can burn continuously when it is lit, as opposed to only flashing briefly, the fire point is a crucial safety criterion.

Fire point: When compared to petroleum diesel, biodiesel often has a greater fire point. This is so because the chemicals found in biodiesel tend to have higher molecular weights and need more energy to vaporise and continue burning. As a result, when it comes to fire safety, biodiesel is preferred over petroleum-based diesel.

Calorific value: Biodiesel's precise composition of fatty acid methyl esters (FAMES) and other additives that may impact its energy content, as well as the feedstock used in its manufacturing, can all affect the fuel's calorific value.

The calorific value of biodiesel is comparatively high to petroleum diesel, with an average calorific value of about 37 to 42 megajoules per kilogramme (MJ/kg). As biodiesel obtained from various feedstocks or manufacturing procedures has slightly different energy levels, the precise calorific value of biodiesel might vary depending on its chemical makeup.

Density: The feedstock utilized, the method of production, and the presence of any additions or contaminants can all affect the density of biodiesel. However, at standard temperature and pressure (STP), which is defined as 25 degrees Celsius (77 degrees Fahrenheit) and 1 atmosphere of pressure, the density of biodiesel generally ranges between 0.86 and 0.90 grams per millilitre (g/mL). The densities of biodiesel blends, like B20, which is composed of 80% petroleum diesel and 20% biodiesel, will range between that of pure biodiesel and petroleum diesel; the precise figure will depend on the mix ratio.

Pour point: The lowest temperature that allows biodiesel to continue flowing or pumping without hardening or becoming too viscous is known as the pour point. Since it affects the functionality of vehicles and equipment that run on biodiesel, it is a crucial attribute, particularly in colder climates or during the winter. Various factors, including the feedstock used in the production process, any added materials or contaminants, might affect the pour point of biodiesel. The pour point range of biodiesel is typically between -9°C and -15°C (16°F and 5°F). While pure

biodiesel has a somewhat lower pour point, some blends, like B20 (20% biodiesel, 80% petroleum diesel), may have a slightly greater pour point.

Flash point: When an ignition source is present and certain test circumstances are met, the lowest temperature at which the fuel's vapor can momentarily ignite is known as the biodiesel flash point. For the handling, moving, and storage of biodiesel, it is a crucial safety factor. An estimated range of 120°C to 160°C (248°F to 320°F) is the flash point range for biodiesel. Conventionally, testing protocols like ASTM D93 or ISO 2719 are used to determine the flash point. These experiments include gradually heating a small sample of biodiesel and frequently passing a small flame over the sample's surface. The flash point of a sample is the temperature at which the vapor above it briefly ignites.

1.6 APPLICATIONS OF BIODIESEL.

Biodiesel is a renewable and eco-friendly fuel substitute that can be used in a variety of industries. Typical uses for biodiesel include:

Transportation Fuel: With minimal to no modification needed, biodiesel can be used directly in diesel engines to replace petroleum diesel. It can be utilised in a variety of vehicles, including cars, lorries, buses, trains, boats, and agricultural equipment, and blended with petroleum diesel in different ratios (such as B5, B20, or greater blends).

Heating Fuel: In commercial, industrial, and domestic heating systems, including space heaters, boilers, and furnaces, biodiesel can be used in place of heating oil.

Production of electricity: Biodiesel can be used as fuel for combined heat and power (CHP) systems and stationary diesel generators, which produce both heat and power. Microgrids, distributed energy systems, and renewable energy installations with intermittent or limited access to grid electricity are among the many settings where biodiesel-fueled generators find frequent application. Applications in the Marine and Aviation Industries: Biodiesel can be utilised as fuel for maritime vehicles, including ships, boats, and ferries, as well as in aviation applications, like aviation biofuels or as a blend element in jet fuel. Even though the marine and aviation industries utilise biodiesel in very small amounts when compared to other forms of transportation, continued research and development

efforts are being made to increase its application and compatibility with marine and aviation engines. Industrial and Commercial Applications: Biodiesel can be employed as a feedstock or precursor in a number of industrial processes, including those that create surfactants, lubricants, bioplastics, and other materials and chemicals derived from plants. Glycerine produced from biodiesel, a

CHAPTER - 2

LITERATURE REVIEW

- **GH Niawanti, N N Octavia et al. (2018)** Using a Soxhlet device and 500 mL of 96% ethanol as a solvent, the study extracted the leaves of Averrhoa bilimbi. Because bioactive chemicals are soluble in ethanol and are both non-toxic and biodegradable, ethanol was employed as a solvent. After being gathered, the leaves were cut and dried at 60 °C until their weight stabilised. After 24 hours of drying, the leaf's moisture content was 48.67%. Examining the impact of extraction time on leaf ethanol extract yield was the aim of the study. The extraction process was run of total flavonoids were produced, which was the highest yield. Because of the ideal extraction time of 360 minutes, the yield was lowered.
- **Haifeng Su et al. (2014)** The family of aquatic plants known as duckweed (Lemnaceae) has the potential to serve as the feedstock for the next generation of alternative energy sources, however not much information about turning duckweed into higher alcohols has been published. We looked into the fermentation procedure used to extract higher alcohols from duckweed. The production of higher alcohols with high energy yields through bioconversion by *Clostridium acetobutylicum*, mutant yeast strains, and bioengineered. Duckweed acid hydrolysate was used to create butanol and total solvent at concentrations of 12.03 and 20.03 g/L through fermentation by *C. acetobutylicum*.
- **Kamrun Nahar et al. (2019)** As a potential substitute for natural gas and crude oil, which are finite resources on which Bangladesh is heavily dependent, duckweed has great promise as a feedstock for ethanol and biogas production. The energy needed for long-term economic expansion cannot be met by the available reserves. The burning of biofuels produces significantly less hydrocarbons, and particulate matter than that of fossil fuels, making them harmless and biodegradable substitutes. Over millions of hectares of lakes, rivers, and ponds make up Bangladesh's prospective sites for phytoremediation.

- Gui Li Yang et al. (2022)** Based on biomass, biofuels are seen as the most promising alternative due to the increasing shortage of conventional energy sources and the corresponding serious environmental challenges. The manufacturing processes and synthesis mechanisms of this essential raw material for biomass energy are the subject of growing investigation. Owing to its many benefits, such as quick biomass buildup, high starch content, high biomass conversion efficiency, and sewage remediation, duckweed has been utilised as a premium new
- Nazim Muradov et al. (2014)** One of the most extensively explored topics in the world is phytoremediation, which offers an appealing option for the bioremediation of water pollution. It is based on the capacity of aquatic plants to recycle nutrients. An affordable and environmentally beneficial solution to the problem of water pollution and the creation of value-added products is the following application of the biomass from the remediation for the production of fuels and petrochemicals.
- Md Arman Arefin et al. (2020)** Aquatic plants have been deemed concerning. Still, one new energy source is the creation of biofuel from aquatic plants. So, along with their physicochemical characteristics, this research provides a thorough overview of the production of biofuel from floating aquatic plants. This research aims to assess some of the key aquatic plants' capacity to produce biofuel. Also, some viable techniques for producing biofuel from aquatic plants are presented in this research for use in future energy production. This study examines five main categories of floating aquatic plants: azolla, water hyacinth, water fern, water lettuce, and duckweed. It also examines the process of producing biofuel from these plants using transesterification, pyrolysis, hydrolysis, and torrefaction. Also, this essay assesses ideal.
- Wei-Hein Chen et al. (2016)** The investigation focused on the effects of extraction solvents on the yields of product fractions and the makeup of the bio-oils that resulted from hydrothermally processing duckweed at 350°C for 30 minutes. A total of ten solvents were used, comprising nonpolar solvents like petroleum ether and cyclohexane as well as polar solvents like isopropanol, ethyl acetate, dichloromethane, diethyl ether, dichloroethane,

benzene, and carbon disulfide. Bio-oil yields were often greater in the extraction solvents with high relative polarity values.

- **Asaad H. Sayer et al. (2023)** One potential substitute that could help provide energy security is biodiesel made from renewable resources. By using direct transesterification, this study attempted to create biodiesel from duckweed plants and *Oedogonium* algae. The considered duckweed plant was given 5 g of dry biomass, and to extract fatty acid methyl esters (FAME), the algae was submerged in 50 mL of a 10:1:1 v/v/v solvent mixture of methanol, hydrochloric acid, and chloroform. Utilising FT-IR and GC-Mass spectrometry, the isolated components were collected for characterization.
- **Natalia Politaeva et al. (2018)** In this work, the same polar solvent (ethanol) is used in combination with different non-polar solvents (methanol-ethanol, chloroform-ethanol, hexane-ethanol, diethyl ether-ethanol, and petroleum ether-ethanol) at varying volume ratios to investigate the effects of different solvent extraction systems on the recovery of lipids extracted from the dry biomass of *Lemna minor*. Finding a Soxhlet extraction technique that uses a variety of solvents to improve lipid recovery while reducing the extraction time is the aim.
- **Amalate Ann Obuebite et al. (2022)** By using pyrolysis, duckweed was converted into bio-oil and its properties were determined. With the use of pyrolysis, the study's objective is to create and evaluate bio-oil from duckweed. A thermocouple attached to the reactor monitored the temperature, and a stopwatch tracked the amount of time until the final drop of product was observed from the system. This study used the pyrolysis method to produce bio-oil from duckweed. A fix-bed reactor was connected to condensers, and heat was applied from a gas hob.
- **Yulia Smyatskaya, Natalia Politaeva et al. (2018)** The literature contains earlier research on the makeup of higher aquatic plants' lipid content. In the current work, ethanol is used as the polar solvent, and different non-polar solvents, such as methanol, chloroform, hexane, diethyl ether, and petroleum ether, are used at different volume ratios to study the effects of different solvent extraction systems on the recovery of lipids extracted from

CHAPTER 3

AIM AND SCOPE

3.1 AIM.

- To determine the best solvent for the extraction of duckweed oil using various solvents.
- To convert the duckweed oil to duckweed biodiesel using transesterification process.
- To determine the fuel properties according to ASTM and PMCC standards.
- To conduct engine test for the prepared biodiesel.

3.2 SCOPE.

- Developing effective processes to transform duckweed biomass which is considered as waste i.e., turned into biofuels is the project's main goal. Developing extraction techniques to turn lipids into fuel that can be used is part of this.
- The project's scope includes future work of scaling up the process for commercial purpose.
- Biofuels can replace the fossil fuels in near future and it is to be noted that from where the source of the biofuel comes from, it is appreciable if it is obtained from natural resource like in this project it is obtained from the aquatic plants that is Duckweed which is a promising candidate for the production of biofuel.

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CHAPTER – 4

MATERIALS AND METHODS

4.1 MATERIALS AND CHEMICALS.

The materials used for the producing biodiesel are duckweed obtained from water resources. Sodium hydroxide used as the catalyst and methanol are used for the transesterification process. The primary process used to convert oil into biodiesel (fatty acid methyl esters, or FAMES) is transesterification, which is where methanol plays a critical role in the manufacturing of biodiesel. In the process of making biodiesel. Triglycerides found in oils are converted into fatty acid methyl esters,

which are the primary ingredients of biodiesel, through a process known as the transesterification reaction. Catalysts like sodium hydroxide (NaOH) must be used with an alcohol, usually methanol for the production of biodiesel.

4.2 EXTRACTION OF OIL FROM DUCKWEED.

Duckweed is a promising feedstock for producing bio fuel. It is obtained from sources like ponds, lakes. The obtained duckweed is in wet condition and its weight is around 1000 grams.



FIG 4.1 DUCKWEED



FIG 4.2 DRYING OF DUCKWEED

The obtained duckweed is spreaded over a thin cloth and got it dried in presence of sunlight. The resulting dried product is measured and the dry weight is around 32 g.

1000 grams (Wet duckweed) ➡ 32 grams (Dried duckweed)

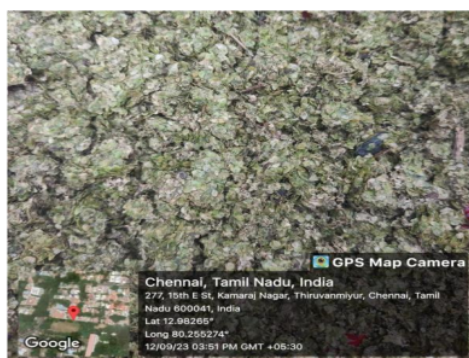
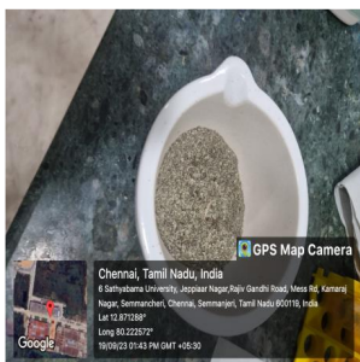


FIG 4.3 CRUSHING OF DUCKWEED

The dried duckweed is separated into 3 samples each sample contains of 10 grams and it is packed in a filter paper by crushing it into fine powder using mortar

and pestle for solvent extraction. The solvent used for extraction are ethanol, isopropyl alcohol and hexane. The boiling point of the solvents are 78.7°C, 82.5°C, 68.7°C respectively. The extraction is done by using an equipment called Soxhlet apparatus. This equipment is used to extract oil from the duckweed.

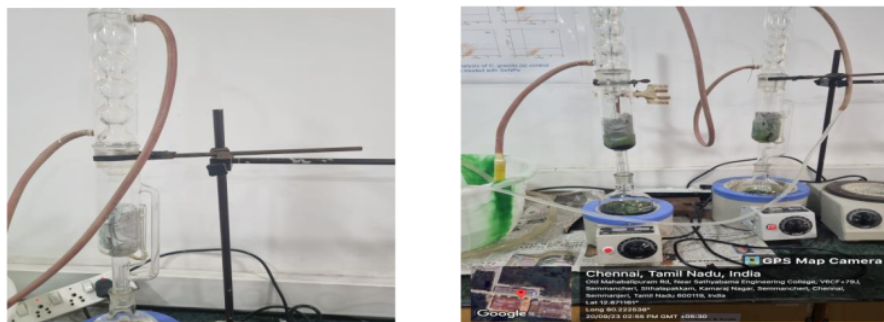


FIG 4.4 SOXHLET APPARATUS

The apparatus in and outflow connected with the water tub as condensation and vaporization process happens simultaneously. The solvent is poured in a round bottom flask and the solvent taken for extraction weighs of 300ml. The extraction is done for around 4 to 5 hours. Icepack has been added to the water tub periodically.

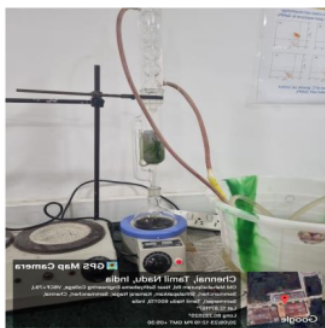


FIG 4.5 SOLVENT EXTRACTION



FIG 4.6 ROTARY EVAPORATOR

The extracted oil is then proceeded to separate out the oil from the solvent this is done by using vacuum evaporator. The temperature in the vacuum evaporator has been set so that oil from the solvent can be separated. It is done around for 20 minutes. Now we have the duckweed oil separated from solvent.

The oil weighs for 3 different solvents such as ethanol(10.009ml), isopropyl alcohol (28.25 ml) and hexane (7.441ml). So among the three solvents used for extraction, the extraction done using ethanol solvent gave more oil than the other 2 solvents.



FIG 4.7 OIL EXTRACTED FROM DIFFERENT SOLVENTS

The yield of the oil is calculated on the basis of amount of oil obtained from the extraction. Among the three solvents used for extraction, in terms of quantity the isopropyl alcohol gave more yield. The other properties of the oil such as specific gravity and density of the oil is studied to finalise the oil for further production of biodiesel. Specific gravity of the oils are calculated using Picnometer i.e, specific gravity bottle in which the empty bottle weight is measured (w_1) and the bottle is filled with water and weight is measured and mark it as (w_2) and the bottle is filled with the oil samples and the weight is measured (w_3), (w_4), (w_5) respectively

Specific gravity = Density of oil / Density of water

Specific gravity = $(w_2 - w_1) / (w_3 - w_1)$

The specific gravity of the oil obtained from ethanol, isopropyl alcohol, hexane solvents are (0.86), (0.74), (0.97) respectively. Specific gravity is a relative quantity therefore the oil with a higher specific gravity results in producing poor diesel. The density of the biodiesel is 1.006g/cc which meets the ASTM standards for producing quality biodiesel.

4.3 PROPERTIES OF DUCKWEED OIL.

4.3.1 DETERMINATION OF ACID VALUE OF OIL:

The quantity of free fatty acids contained in an oil is indicated by its acid value. In the food and pharmaceutical industries in particular, it is a crucial characteristic for the examination of oils and fats.

The acid value is commonly represented as the milligrams of sodium hydroxide (NaOH) needed to neutralize one gram of oil's worth of free fatty acids. Higher acid values signify a higher level of rancidity or deterioration, making it a gauge of the oil's quality and freshness.

A standardized NaOH solution can be used for titration, one of the analytical techniques that can be used to determine the acid value. It is a crucial criterion for determining if oils are suitable for a range of uses, such as cooking.

Chemicals required: Ethanol, NaOH, Indicator (phenolphthalein)

Materials required: Oil sample, burette with stand, hot plate, weighing balance.

- Prepare 0.1N NaOH solution.
- Add few drops phenolphthalein indicator to 50 ml Ethanol.
- Titrate this against NaOH to neutralize.
- Neutralized ethanol is added with 10g of oil and it is heated using hot plate.
- After 20 mins add few drops of indicator and titrate against 0.1 NaOH
- Note initial and final burette readings.

Acid value = $40 \times 0.1 \times (F.V - I.V) / \text{Weight of the sample}$

Acid value = $40 \times 0.1 \times 1.6 / 10$

Acid value of the oil = 0.64

4.3.2 DETERMINATION OF FFA OF OIL:

The term "free fatty acids" (FFAs) describes the unesterified fatty acids that are found in oil. Since they are not connected to any other molecule, like glycerol, they are referred to as "free." FFAs naturally occur in oil as a result of oxidation, hydrolysis, or other degrading processes.

Particularly in the food sector, the amount of FFAs in an oil is a significant determinant of its quality. Increased levels of FFAs can cause food products to

have bad Flavors, have a shorter shelf life, and have less nutritional content. Consequently, reducing FFAs is crucial to preserving the stability and quality of oils.

The acid value determination method, which quantifies the quantity of KOH needed to neutralize the FFAs, is frequently used to test FFAs in oil.

Free fatty acid is calculated by : Acid value of oil/2

The obtained FFA of oil is 0.32. It means that the extracted duckweed oil is to be proceeded with single step process i.e, transesterification.

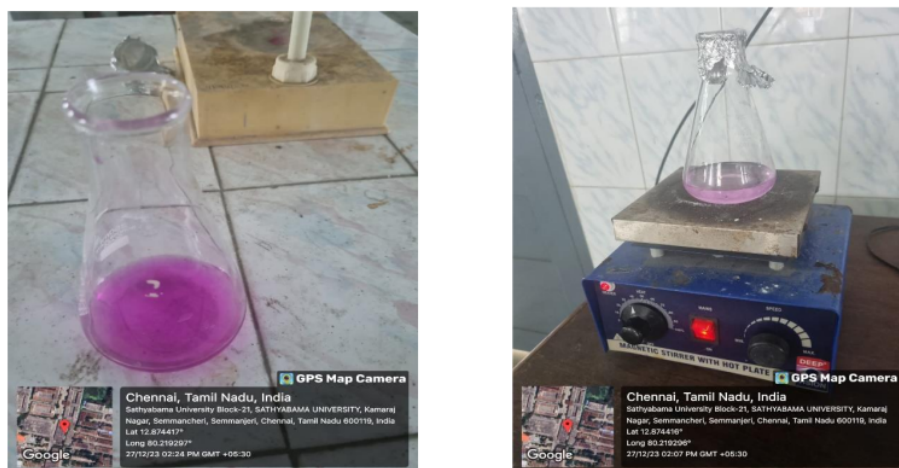


FIG 4.8 ACID VALUE DETERMINATION

4.3.3 DETERMINATION OF SAPONIFICATION VALUE OF OIL:

The amount of alkali required to saponify a specific amount of fat or oil is indicated by the oil's saponification value. The amount of ²⁶potassium hydroxide (KOH) milligrams needed to saponify one gram of oil is how it is expressed.

The amount of lye (potassium or sodium hydroxide) required to convert a given volume of oil into soap is calculated in the soap industry using the saponification value. Additionally, it offers details regarding the fatty acids' average molecular weight in the oil.

Chemicals required: Absolute ethanol, KOH, HCL, Indicator.

- Materials required: Reflux column, Weighing balance, Burette with stand, Oil sample.
- Measure 200 ml ethanol add 1.67 g of KOH and add 1g of granulated aluminium foil and transfer it into round bottom flask and heat it using heating mantle with reflux column on top.
- Using rotary evaporator Ethanolic KOH is separated.
- Prepare a sample of blank and oil.
- Titrate against 0.1N HCl. Note initial and final readings.
- Blank sample : $(50-0) + (39-20) = 69 \text{ ml}$
- Oil sample : $(17.5 - 11.5) = 6$
- Saponification value = $28.05 * (V_b - V_s) / W_s$
- $S.V = 28.05 * (69 - 6) / 5.2$
- Saponification value of oil = 283



FIG 4.9 REFLUX COLUMN



FIG 4.10 SAPONIFICATION VALUE DETERMINATION

4.4.4 DETERMINATION OF MOLECULAR WEIGHT OF THE OIL:

$$M.W = 56.1 \times 1000 \times 3 / (S.V - A.V)$$

$$M.W = 56.1 \times 1000 \times 3 / (283 - 0.6)$$

Molecular weight of the extracted oil = 596 g/mol.

4.4 PREPARATION OF BIODIESEL.

Biodiesel is prepared in different ratios and in different conditions by changing the parameters. The experiment is carried out in a magnetic stirrer set up with a condensation column fixed so that it prevents the solvent from evaporating. Obtaining and getting ready the reactants required for the transesterification reaction is the first stage. The feedstock in this case would usually be vegetable or animal fat, the alcohol would be methanol (or ethanol), and the catalyst would be sodium hydroxide (NaOH).

Reactant mixing involves combining methanol and vegetable (or animal) oil in a reaction vessel with the catalyst present. The vessel is set on a hot plate for a magnetic stirrer and has a magnetic stir bar within. The transesterification reaction

is sped up by heating the mixture to a certain temperature, usually between 50°C and 60°C. Heating increases the rate of reaction and reduces the oil's viscosity.

The magnetic stir bar inside the reaction vessel spins quickly due to the magnetic field created by the magnetic stirrer. In addition to ensuring complete mixing of the reactants, this stirring movement also facilitates effective contact between the alcohol, catalyst, and oil.

The transesterification reaction produces fatty acid methyl esters (FAMES) and glycerol when the oil's triglycerides combine with the alcohol in the presence of the catalyst. Triglycerides are better converted to biodiesel thanks to the swirling action, which also helps to improve reaction kinetics.

Reaction Monitoring: Temperature, pH, viscosity, and other variables are usually measured to track the transesterification reaction's development over time. The mixture is permitted to settle after the reaction is finished in order to separate the biodiesel (FAME) layer.

4.4.1 Experimental calculation for biodiesel:

Molecular weight of the oil = 596 g/mol

Molecular weight of the methanol = 32.04 g/mol

Density of the oil = 1.006 g/cc

Density of the methanol = 0.791 g/cc

For 1:3 ratio.

1 mol of oil : 3 mol of methanol

$$1 \times 596.04 = 3 \times 32.04$$

$$596.04 = 96.12$$

$$596 / 1.006 = 96.12 / 0.791$$

$$592.48 = 121.51$$

40 ml of oil = x ml of methanol

X = 8.2 ml methanol required for transesterification.

Mass of methanol = Density of methanol * Volume of methanol

$$\text{Mass} = 0.791 * 8.2 = 6.486$$

Mass of the oil = 40.24

Total mass = 46.24

Catalyst at 3 Wt % $X/X + 46.24 = 3 / 100$

X = 1.43 grams required for transesterification.

For 1:6 ratio.

$$596.04/1.006 = 6 * 32.04/0.791$$

$$592.48 = 243.034$$

40 = X ml of methanol

X = 16.40 ml

Total mass = 53.21

Catalyst at 3 Wt % = 1.65 grams.

For 1:9 ratio.

$$592.48 = 9 * 32.04/0.791$$

X = 24.611 ml of methanol

Total mass = 59.7

Catalyst at 3 Wt % = 1.84 grams

For 1:12 ratio.

$$592.48 = 12 * 32.04/0.791$$

X = 32.81 ml

Total mass = 66.19

Catalyst = 2.24 g

For 1:15 ratio.

$592.48 = 15 \times 32.04 / 0.791$

X=40.9 ml

Total mass = 72.54

Catalyst = 2.44g

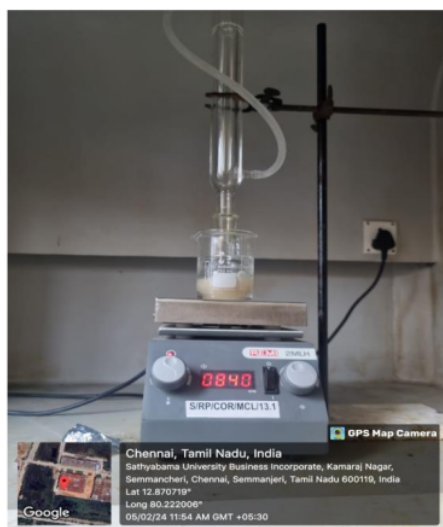


FIG 4.11 TRANSESTERIFICATION SETUP

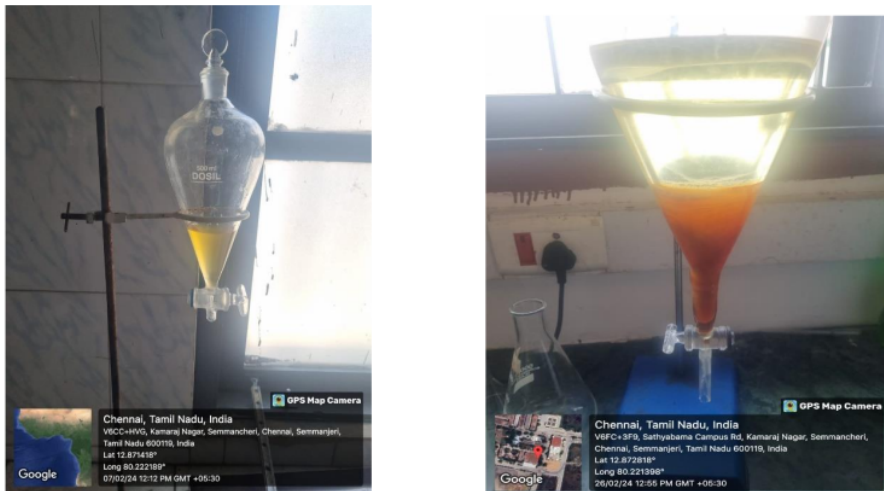


FIG 4.12 BIODIESEL

4.4.2 OPTIMIZATION OF BIODIESEL.

In order to increase productivity, yield, and product quality while lowering expenses and having a minimal negative impact on the environment, biodiesel production must be optimized in a number of ways. Make sure that the alcohol-to-

oil ratio, reaction temperature, reaction time, catalyst type, and concentration are all optimized for the transesterification reaction. The yield, purity, and conversion efficiency of biodiesel are influenced by these factors.

From the below table, particular ratio 1:12 is tried with change in parameters like time, temperature and in catalyst weight percentage to obtain the optimized biodiesel. The one with the highest yield among the ratios is tried with different experiments. The final output is proceeded to characterization to know more in detail.

S. no	Ratio	Oil (ml)	Methanol (ml)	Mass of methanol	Mass of Oil	Catalyst Wt. %	Catalyst in (g)	Time in min	Temp °C	Yield %
1	1:3	40	8.2	4.0341	40.2	3	1.26	45	60	42
2	1:6	40	16.4	8.068	40.2	3	1.38	45	60	49.6
3	1:9	40	24.611	12.11	40.2	3	1.51	45	60	63
4	1:12	40	32.81	16.11	40.2	3	1.63	45	60	74
5	1:12	40	32.81	16.11	40.2	1	1.45	45	60	72
6	1:12	40	32.81	16.11	40.2	1	1.45	30	45	76
7	1:12	40	32.81	16.11	40.2	2	1.58	40	45	78
8	1:12	40	32.81	16.11	40.2	2	1.58	30	45	74

9	1:12	40	32.81	16.11	40.2	3	1.58	60	60	70
10	1:15	40	40.9	20.15	40.2	3	1.76	45	60	54.2
11	1:18	40	49.22	24.20	40.2	3	1.88	45	60	61

TABLE 4.1 OPTIMIZATION OF BIODIESEL.

CHAPTER 5

RESULTS AND DISCUSSIONS

5.1 PHYSIOCHEMICAL PROPERTIES OF DUCKWEED.

5.1.1 FTIR.

28 Fourier transform – infrared radiation spectroscopy The Fourier transform infrared spectroscopy (FTIR) is a valuable tool for identifying organic compounds, polymers, and functional groups in a variety of samples. It does this by measuring the interaction of materials with infrared light.

Once the functional groups have been identified, several characteristics of the sample, including its chemical composition, structure, purity, and even some physical attributes, can be inferred. An infrared absorbance (or transmittance) vs wavenumber (or wavelength) plot results in the resulting spectrum. It is possible to

identify the functional groups present in the sample by comparing the peaks and troughs in the spectrum to recognized spectral patterns found in databases or literature.

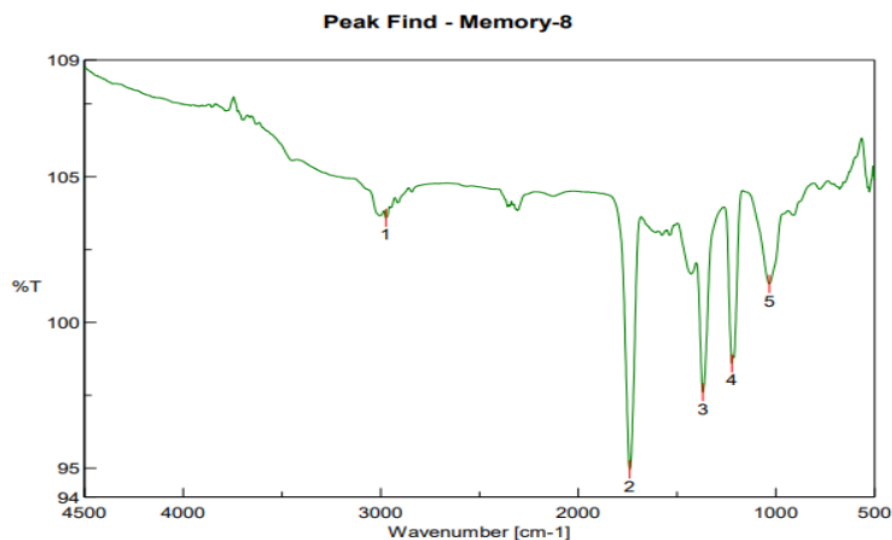


FIG 5.1 FTIR FOR DUCKWEED

From the above graph peak level at different position indicates the functional groups present in it.

- 2973.7 shows the presence of C-H stretch with variable intensity.
- 1739.48 shows the presence of C=O Ketone with strong intensity.
- 1369.21 shows the presence of C-O stretch with strong intensity.
- 1222.65 shows the presence of Alkyl ketone stretch with strong intensity.
- 1032.69 shows the presence of C-F stretch with strong intensity.

5.1.2 FESEM.

Field Emission Scanning Electron Microscopy, or FESEM for short, is a high-resolution imaging method that probes the nanoscale structure and surface morphology of materials. The morphology of biodiesel, specifically the size, shape, and distribution of the particles or aggregates present, can be understood through the application of FESEM analysis.

Using an electron beam, the FESEM device examines the sample's surface. Fine surface details can be seen in images produced with a very high resolution and magnification by detecting secondary electrons emitted from the surface.

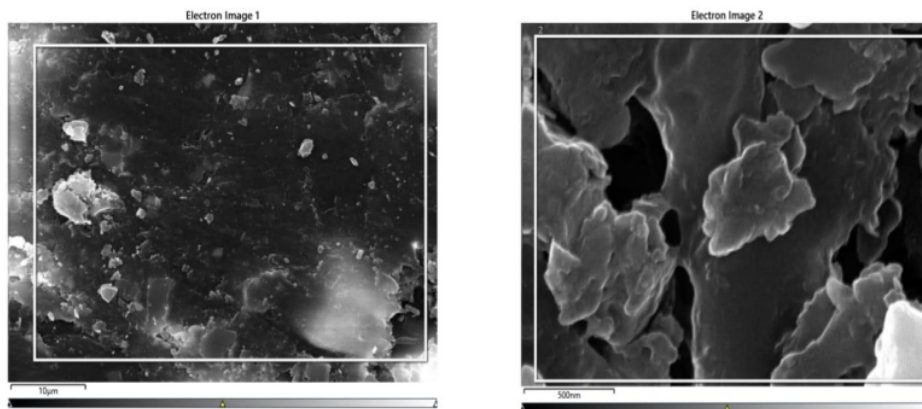


FIG 5.2 SEM IMAGES OF DUCKWEED

Given that duckweed is a plant, it is likely that the powder's particles are asymmetrical and uneven. The shapes and distribution of particle sizes are seen in the FESEM image. The morphology of the cell wall is visible in the FESEM image as the resolution is good enough.

5.1.3 EDAX.

EDAX is an analytical method that measures the energy of X-rays released when electrons interact with a sample. It is frequently used in conjunction with scanning electron microscopy (SEM) to ascertain the elemental composition of a sample. It is utilized for elemental analysis in a variety of applications, such as geology and materials characterization.

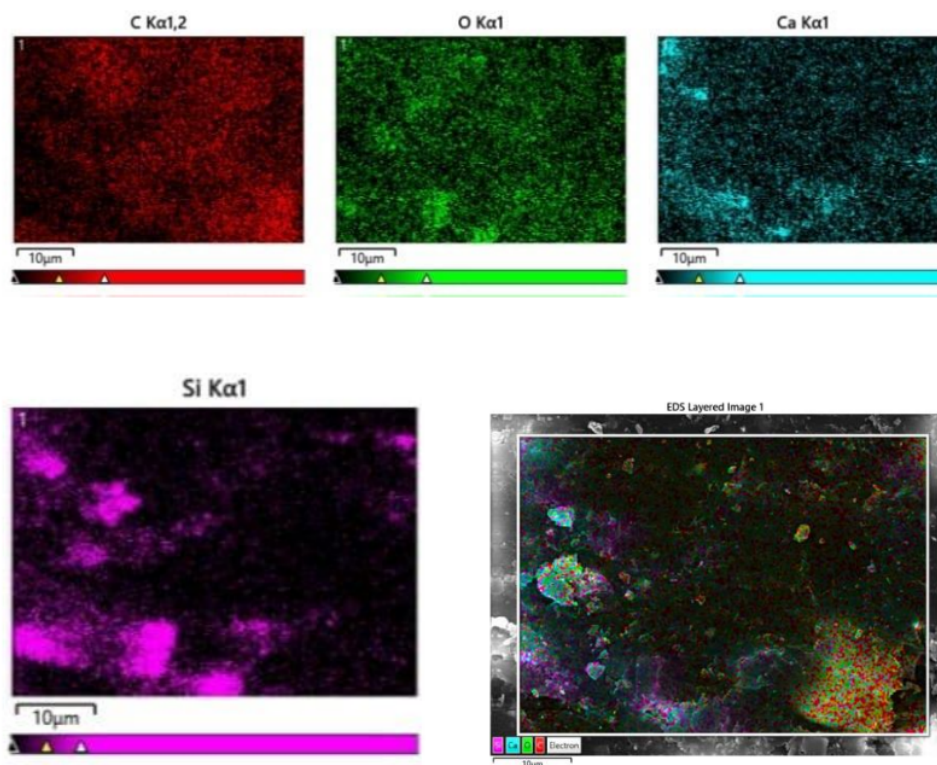


FIG 5.3 EDAX FOR DUCKWEED

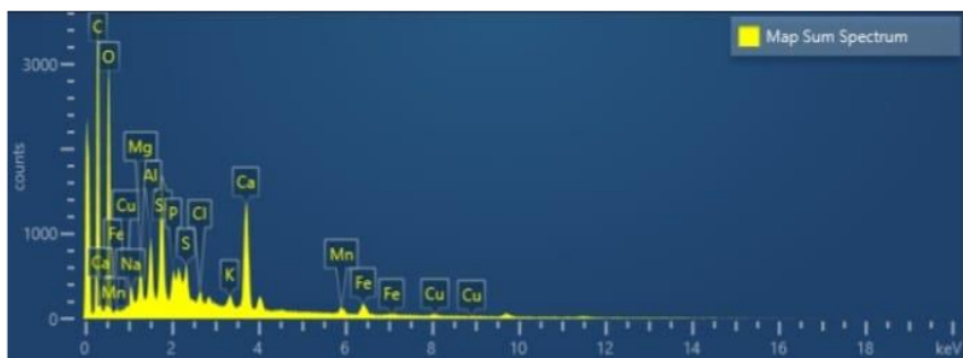


FIG 5.4 EDAX MAPPING FOR DUCKWEED

TABLE 5.1 ELEMENTAL COMPOSITION IN DUCKWEED

Map Sum Spectrum				
Element	Line Type	Weight %	Weight % Sigma	Atomic %
C	K series	53.31	0.58	64.01
O	K series	33.72	0.47	30.40
Na	K series	0.64	0.05	0.40
Mg	K series	1.01	0.04	0.60
Al	K series	1.30	0.04	0.70
Si	K series	2.49	0.05	1.28
P	K series	0.36	0.04	0.17
S	K series	0.61	0.04	0.27
Cl	K series	0.36	0.03	0.15
K	K series	0.36	0.03	0.13
Ca	K series	3.88	0.07	1.40
Mn	K series	0.55	0.05	0.14
Fe	K series	1.07	0.06	0.28
Cu	K series	0.32	0.08	0.07
Total		100.00		100.00

The above table shows the list of chemical elements present in duckweed. This result is obtained as the result of EDAX.

5.1.4 XRD.

An effective method in materials science for figuring out a crystalline material's atomic and molecular structure is X-ray diffraction (XRD) examination. Indexing the diffraction peaks to ascertain the lattice parameters and crystal symmetry, fine-tuning the parameters to match the experimental data.

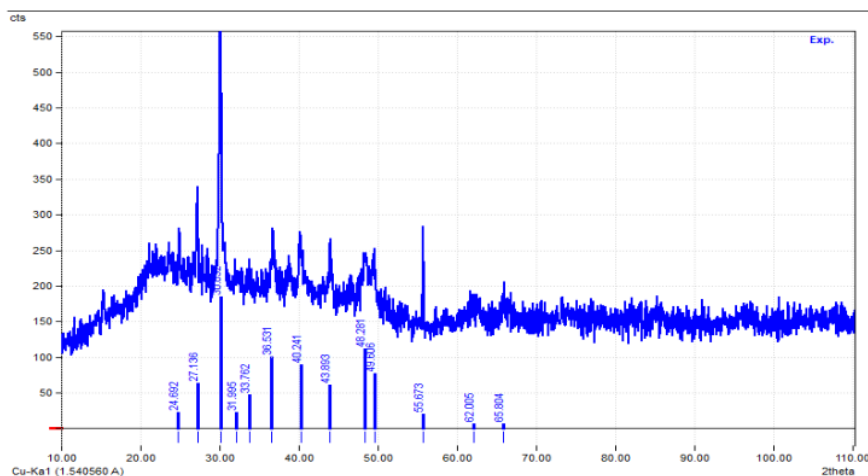


FIG 5.5 XRD FOR DUCKWEED

Peak List					
No.	2theta [°]	d [Å]	I/I0 (peak height)	Counts (peak area)	FWHM
1	24.69	3.6026	184.33	21.93	0.2356
2	27.14	3.2834	372.78	62.73	0.3333
3	30.05	2.9711	1000.00	183.64	0.3637
4	32.00	2.7949	58.37	21.90	0.7431
5	33.76	2.6526	80.19	45.86	1.1328
6	36.53	2.4577	173.13	100.02	1.1443
7	40.24	2.2392	215.83	88.07	0.8082
8	43.89	2.0610	155.59	60.33	0.7680
9	48.28	1.8834	174.14	111.08	1.2531
10	49.61	1.8362	107.92	75.75	1.2530
11	55.67	1.6496	318.36	18.93	0.1178
12	62.00	1.4955	97.81	5.82	0.1178
13	65.80	1.4180	99.33	5.91	0.1178

Integrated Profile Areas		
Based on calculated profile		
Profile area	Counts	Amount
Overall diffraction profile	585319	100.00%
Background radiation	499298	85.30%
Diffraction peaks	86021	14.70%
Peak area belonging to selected phases	0	0.00%
Unidentified peak area	86021	14.70%

Peak Residuals		
Peak data	Counts	Amount
Overall peak intensity	802	100.00%
Peak intensity belonging to selected phases	445	55.43%
Unidentified peak intensity	357	44.57%

FIG 5.6 PEAK REPORT OF XRD

XRD spectrum indicated in the above graph shows well defined and intense peak at 30.02 indicates the emergence of crystalline.

5.2 CHARACTERIZATION OF BIODIESEL.

5.2.1 FTIR.

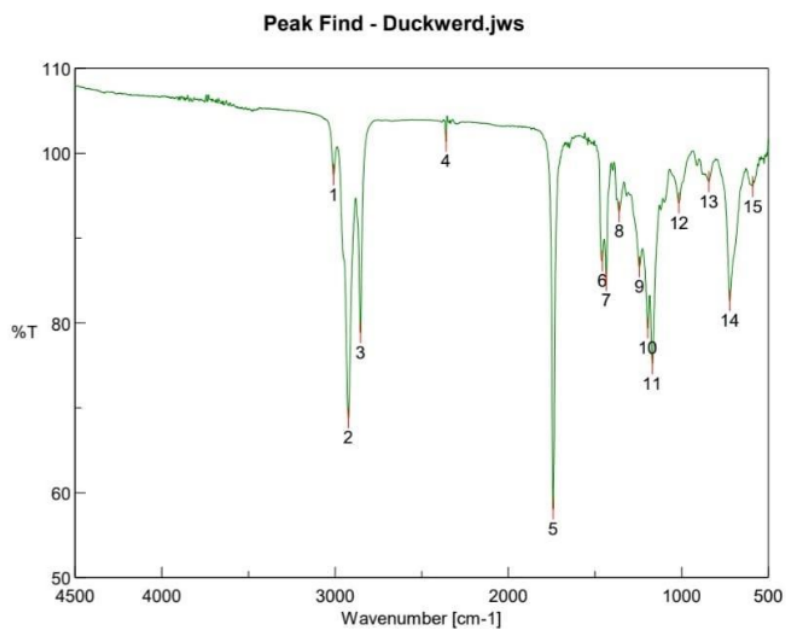


FIG 5.7 FTIR FOR BIODIESEL

[Result of Peak Picking]

No.	Position	Intensity	No.	Position	Intensity
1	3008.41	97.4442	2	2923.56	68.8111
3	2853.17	78.8325	4	2360.44	101.385
5	1741.41	58.0784	6	1457.92	87.2999
7	1435.74	84.9783	8	1361.5	93.1005
9	1243.86	86.605	10	1195.65	79.3903
11	1169.62	75.1517	12	1016.3	94.1431
13	843.704	96.6588	14	722.211	82.6245
15	591.075	96.0583			

FIG 5.8 PEAK REPORT OF FTIR

From the above graph peak level at different position indicates the functional groups present in it.

- 3008.41 shows the presence of C-H Stretching group with medium intensity.
- 2923.56 shows the presence of O-H Stretching with strong broad.
- 1741.41 shows the presence of C=O stretching (Esters) with strong intensity.
- 1169.62 shows the presence of C-O stretching (Esters) with strong intensity.
- 591.5 shows the presence of S=O sulfonate stretching with strong intensity.

5.2.2 PROPERTIES OF BIODIESEL.

TABLE 5.2 PROPERTIES OF BIODIESEL

S. No	Parameters	Results
1	Density (g/cc)	0.86
2	Kinematic Viscosity @ 40 (°C)	3.8
3	Flash point by PMCC method (°C)	154.2
4	Fire point by PMCC method (°C)	172
5	Pour point by ASTM method (°C)	-4.4
6	Calorific value (MJ/Kg)	35.5

The above table shows the properties of optimized biodiesel. The values of parameters meet the standards according to ASTM D6751-1

5.2.3 GCMS.

Gas Chromatography-Mass Spectrometry (GC-MS) analysis is a widely employed method for the evaluation and verification of biodiesel's quality. Typically, specialist software is used to process the results from GC-MS analysis. By comparing the mass spectra of the sample components with those of established standards or databases, this program assists in the identification of the various chemicals included in the biodiesel sample. Additionally, it measures how much of each

ingredient is present in the sample. By giving manufacturers precise information about the chemical makeup of biodiesel, GC-MS analysis enables them to monitor product quality and optimize production procedures.

GCMS ANALYSIS OF DUCKWEED OIL.

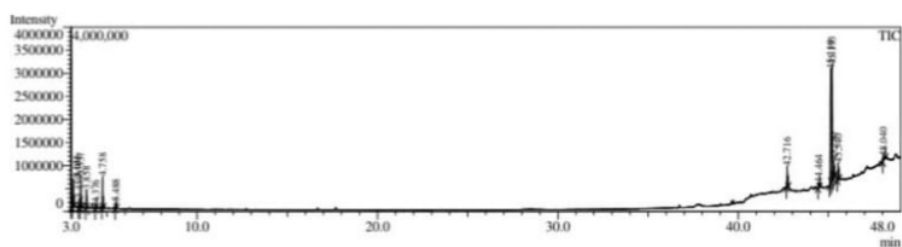


FIG 5.9 GCMS FOR DUCKWEED OIL

TABLE 5.3 PEAK REPORT OF GCMS FOR DUCKWEED OIL

Peak Report TIC				
Peak#	R.Time	Area	Area%	Name
1	3.036	1108362	4.34	Ethylbenzene
2	3.141	1269472	4.98	BENZENE, 1,2-DIMETHYL-
3	3.459	362705	1.42	BENZENE, 1,2-DIMETHYL-
4	3.537	1475516	5.78	2-PENTANETHIOL, 2-METHYL-
5	3.858	837886	3.28	2-Aminoethanol, N,O-diacetyl-
6	4.376	227273	0.89	Pentane, 3-ethyl-2,4-dimethyl-
7	4.758	1536711	6.02	3-Hexen-2-one
8	5.488	291651	1.14	BENZENE, 1,2,3-TRIMETHYL-
9	42.716	1017351	3.99	Glycidol stearate
10	44.464	243164	0.95	9,12-Octadecadienoic acid (Z,Z)-, 2,3-dihydr
11	45.119	6725170	26.36	14-METHYL-8-HEXADECYN-1-OL #
12	45.193	8209109	32.17	9-Octadecenoic acid, 1,2,3-propanetriyl ester
13	45.280	866450	3.40	cis,cis-7,10,-Hexadecadienal
14	45.540	890324	3.49	Glycidol stearate
15	48.040	454572	1.78	9-Octadecenoic acid (Z)-, 2,3-dihydroxyprop
		25515716	100.00	

The above GCMS result for duckweed oil demonstrates the presence of octadecadienoic acid and other components present in it.

GCMS ANALYSIS OF DUCKWEED BIODIESEL.

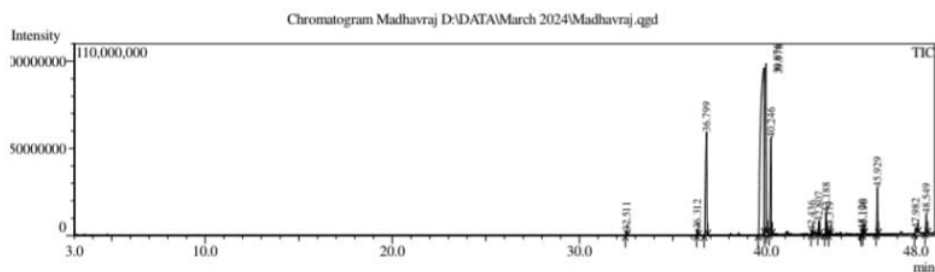


FIG 5.10 GCMS OF BIODIESEL

TABLE 5.4 PEAK REPORT OF GCMS FOR BIODIESEL.

Peak Report TIC				
Peak#	R.Time	Area	Area%	Name
1	32.511	5531425	0.22	TETRADECANOIC ACID, METHYL ESTER
2	36.312	8468037	0.34	9-Hexadecenoic acid, methyl ester, (Z)-
3	36.799	264521271	10.61	HEXADECANOIC ACID, METHYL ESTER
4	39.876	1286550895	51.59	Methyl 10-trans,12-cis-octadecadienoate
5	39.978	560926666	22.49	9-OCTADECENOIC ACID, METHYL ESTER
6	40.246	185679793	7.45	Methyl stearate
7	42.436	4179393	0.17	OXIRANEOCTANOIC ACID, 3-OCTYL-, 1
8	42.807	16961138	0.68	cis-11-Eicosenoic acid, methyl ester
9	43.188	30451199	1.22	EICOSANOIC ACID, METHYL ESTER
10	43.379	4863741	0.20	OXIRANEOCTANOIC ACID, 3-OCTYL-, 1
11	45.120	6042643	0.24	BICYCLO[10.1.0]TRIDEC-1-ENE
12	45.196	5892554	0.24	9-Octadecenoic acid, 1,2,3-propanetriyl ester
13	45.929	68391835	2.74	Methyl 20-methyl-heneicosanoate
14	47.982	14622919	0.59	9,12-OCTADECADIENOIC ACID (Z,Z)-, 2
15	48.549	30878970	1.24	TETRACOSANOIC ACID, METHYL ESTER
		2493962479	100.00	

The above table and peaks present in the graph demonstrates the presence of methyl ester which is the main component of biodiesel and confirms whether the oil is converted into biodiesel and this is attained by transesterification process.

21 This confirms the presence of methyl esters and acids present in the duckweed biodiesel. As duckweed oil is converted to biodiesel, the triglycerides it contains undergo transesterification, which yields these methyl esters of fatty acids.

5.2.4 INTERNAL COMBUSTION ENGINE STUDY

Internal combustion engines (ICEs) running on biodiesel fuel would be the subject of a study that would examine a number of topics including performance, emissions, and engine optimisation. The IC engine study for various blends [B15, B30] are studied.

Break thermal efficiency (BTE) is a crucial metric for assessing an internal combustion engine's efficiency. It calculates the usable work production in relation to the engine's total energy input. When evaluating the performance of engines using various fuels, BTE is a crucial parameter to consider.

Calculate the amount of heat created when the biodiesel in the engine burns. This entails taking the biodiesel fuel's energy content and combustion efficiency into account. Incomplete combustion and heat loss to the environment are losses that 46 are taken into account by the combustion efficiency. Utilizing the torque and rotational speed, compute the engine's work output. With the engine displacement volume and the torque-speed connection, one may compute the amount of work performed in a single engine cycle. When operating under similar conditions, compare the break thermal efficiency of conventional diesel fuel and biodiesel fuel. The relative effectiveness of utilizing biodiesel as a fuel in internal combustion engines is revealed by means of this comparison. An evaluation of the mechanical efficiency of biodiesel usually entails comparing the performance of machinery or engines running on biodiesel fuel to those running on regular diesel fuel. The purpose of this investigation is to determine how biodiesel affects the system's mechanical efficiency.

Examining the torque production of an engine or piece of equipment when running on biodiesel fuel as opposed to regular diesel fuel is known as torque analysis for biodiesel. Because it indicates the rotating force produced by the engine's drivetrain, torque is an important metric for evaluating an engine's mechanical performance. To see the engine's torque output over its operating range, plot torque curves for both conventional diesel fuel and biodiesel.

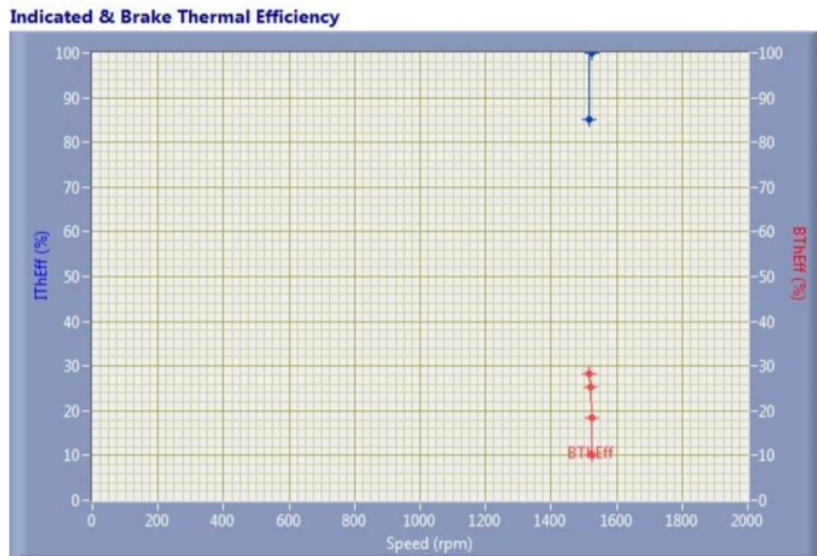


FIG 5.11 BREAK THERMAL EFFICIENCY FOR B15

TABLE 5.5 BREAK THERMAL EFFICIENCY DATA FOR B15

Speed (rpm)	Load (kg)	IThEff (%)	BThEff (%)
1525.00	0.61	191.85	10.18
1524.00	0.61	298.80	10.19
1523.00	3.35	99.98	18.55
1521.00	6.12	114.58	25.39
1518.00	9.09	85.08	28.20

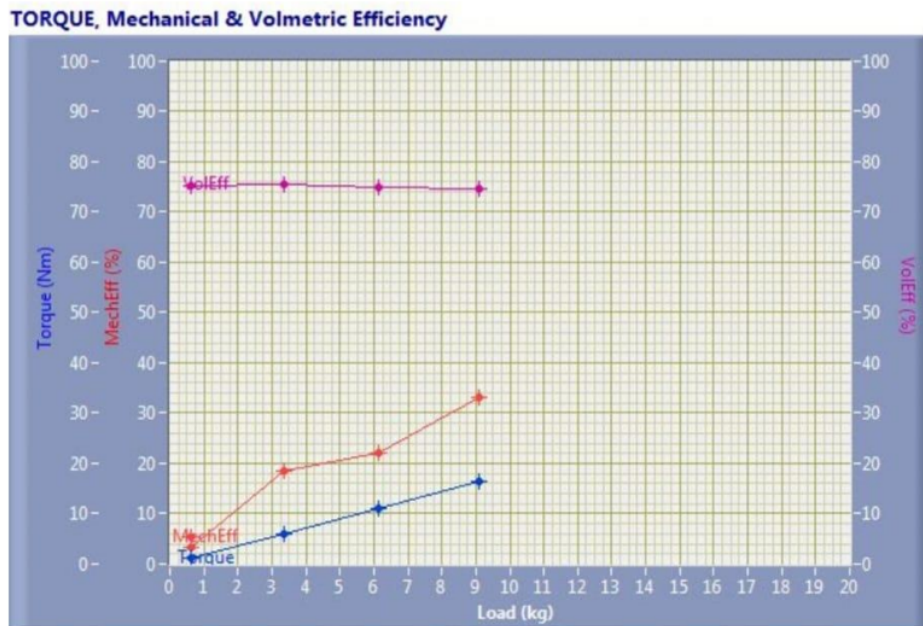


FIG 5.12 IC ENGINE STUDY FOR B15

TABLE 5.6 IC ENGINE STUDY DATA FOR B15

Speed (rpm)	Load (Kg)	Torque (Nm)	Mech Eff. (%)	Vol Eff. (%)
1525.00	0.61	1.11	5.30	75.32
1524.00	0.61	1.11	3.41	75.36
1523.00	3.35	6.08	18.56	75.46
1521.00	6.12	11.12	22.16	74.88
1518.00	9.09	16.49	33.14	74.72

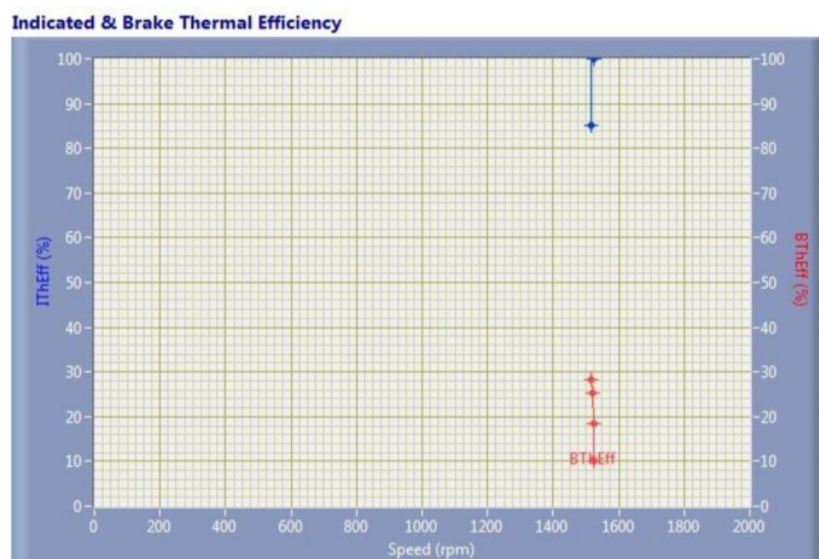


FIG 5.13 BREAK THERMAL EFFICIENCY FOR B30

TABLE 5.7 BREAK THERMAL EFFICIENCY FOR B30

Speed (rpm)	Load (Kg)	Torque (Nm)	Mech Eff. (%)	Vol Eff. (%)
1525.00	0.61	1.11	5.30	75.32
1524.00	0.61	1.11	3.41	75.36
1523.00	3.35	6.08	18.56	75.46
1521.00	6.12	11.12	22.16	74.88
1518.00	9.09	16.49	33.14	74.72

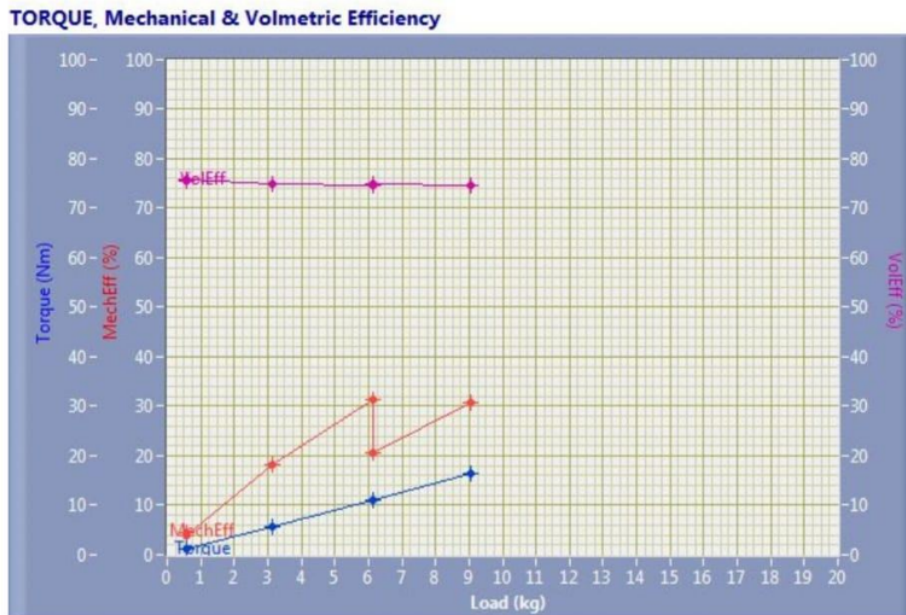


FIG 5.14 IC ENGINE STUDY FOR B30

TABLE 5.8 IC ENGINE STYDY DATA FOR B30

Speed (rpm)	Load (kg)	IThEff (%)	BThEff (%)
1525.00	0.61	191.85	10.18
1524.00	0.61	298.80	10.19
1523.00	3.35	99.98	18.55
1521.00	6.12	114.58	25.39
1518.00	9.09	85.08	28.20

5.2.5 EMISSION ANALYSIS FOR VARIOUS BLENDS.

Emissions from internal combustion engines using biodiesel are essential for assessing the fuel's environmental impact and contrasting it with regular diesel. It describes the steps taken to assess emissions while using biodiesel fuel in an

engine. It indicates the parameters of the emissions that were measured, such as hydrocarbons (HC), carbon monoxide (CO), particulate matter (PM), nitrogen oxides (NO_x), and other pollutants. Give specifics about the test parameters, such as the load, engine speed, and composition of the biodiesel blend.

TABLE 5.9 EMISSION ANALYSIS FOR B15

Name of the gas	Load 0	Load 3	Load 6	Load 9
CO	0.01	0.02	0.05	0.06
HC	1	1	2	3
CO ₂	0.40	0.5	1.20	0.8
O ₂	20.40	19.96	19.11	18.68
NO _x	23	25	53	62
SMOKE DENSITY	4.3	47.8	52.8	58.3

TABLE 5.10 EMISSION ANALYSIS FOR B30

Name of the gas	Load 0	Load 3	Load 6	Load 9
CO	0.01	0.02	0.04	0.04
HC	2	2	3	3
CO ₂	0.70	0.90	1.20	1.9
O ₂	19.95	19.89	19.07	18.65
NO _x	5.7	58	62	64.32
SMOKE DENSITY	3.4	46.8	56.8	64.30

The above tables show the amount of gas emitted for different loads of biodiesel input. It is done for two blends i.e B15 and B30. The emission values of various gases were studied through the fine gas detector. As the emission range meets the standards it can be used as an alternative eco friendly source.

SUMMARY AND CONCLUSION.

6.1 SUMMARY.

Oil was extracted from duckweed using Solvent extraction technique by using various solvents like Hexane, Isopropyl alcohol and Ethanol. The extraction was carried out in Soxhlet apparatus. It was found that the best yield was obtained with the Isopropyl alcohol. The yield for different solvents were 28%,72%,54% respectively and the solvent is chosen on the basis of properties like density and specific gravity which plays a vital role for producing quality biodiesel. The characteristics of dried duckweed was also studied using FTIR, FESEM, EDAX and XRD. It was found that it has the essential to produce biodiesel. In XRD analysis it showed that the sample was crystalline. Biodiesel was produced by transesterification process, In which the oil to methanol ratio was finalized on the basis of yield further it was optimized using different parameters such as temperature, time to obtain the optimized bio diesel. The biodiesel experiment was carried out using magnetic stirrer as it mixes uniformly. The optimized condition had the reaction time about 40 minutes, 45°C, catalyst [2 Wt %]. The properties of bio diesel was determined by ASTM and PMCC method. The properties are fire point, flash point, calorific value, density and kinematic viscosity. Internal combustion engine test and emission test was done for two blends B15 and B30 respectively. In engine test the break thermal analysis, air flow, torque efficiency, mechanical efficiency were studied. In emission analysis various gases at different loads were analysed using fine gas analyser. The other characterisation for bio diesel such as FTIR, GCMS was studied and it shows the presence of Ester and other functional groups. In GCMS it indicated the presence of various acids and methyl esters that shows that the biodiesel can be used as an alternative fuel

6.2 CONCLUSION

We conclude that the oil extracted from duckweed can be converted to duckweed bio diesel and it can be used in Internal combustion engines without any modifications. The process can be scaled as future work.

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