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MICROWAVE ASSISTED SUNFLOWER OIL ETHYL ESTERPRODUCTION

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ABSTRACT

Ethanol is nont oxic biologically derivable alcohol often used to produce biodiesel, but it is not very effective as methanol because less activity in the conventional transesterification reaction. Hence microwave assisted ethanolysis process is attempted to study the biodiesel production from sunflower oil using ethanol and KOH. In this research work microwave, assisted transesterification setup was designed, fabricated and used for producing sunflower oil ethyl ester. In this experimental study, the microwave temperature for ethanolysis of sunflower oil was optimized with other process parameters kept unchanged. The synthesized ethyl ester was examined for physiochemical properties and compared with EN biodiesel standards. The results showed that 70°C was identified as optimum temperature with 92.7% sunflower oil ethyl ester yield when other parameters kept constant such as molar ratio of 6:1, reaction time 90 minutes, stirring speed at 500 rpm. 1%(w/w) KOH. The physiochemical characteristics results showed that the sunflower oil ethyl ester produced had appreciable properties within the EN standards.

INTRODUCTION

BIODIESEL

Biodiesel is a renewable fuel derived from vegetable oil or animal fats that can be added to conventional diesel to create a blend or used on its own. It is made from renewable resources that are largely plant-based, with their energy coming from the sun instead of fossil fuels. These feedstocks are diverted waste or byproducts from other industries, giving them new life. That helps biodiesel emit less greenhouse gases. In fact, biodiesel's lifecycle greenhouse gas emissions are up to 86% lower than those of petroleum diesel.Biodiesel often costs less than petroleum diesel. And it is a drop-in fuel that can be used in existing diesel vehicles and fueling infrastructure, giving diesel fleets a solution for reducing carbon emissions right away with their existing equipment. Compared with petroleum diesel, biodiesel fuel also enhances performance through higher Cetane, added lubricity and a cleaner burn that puts less stress in diesel particulate filters. Biodiesel is the fuel that can bring all these benefits to fleets, retailers and wholesalers.

BENEFITS OF BIODIESEL PRODUCED FROM RENEWABLE RESOURCES

Biodiesel is a renewable energy source, unlike other petroleum products that will vanish in years to come. Since it is made from animal and vegetable fat, it can be produced on demand and also causes less pollution than petroleum diesel.

CAN BE USED IN EXISTING DIESEL ENGINES

One of the main advantages of using biodiesel is that it can be used in existing diesel engines with little or no modifications at all and can replace fossil fuels to become the most preferred primary transport energy source. Biodiesel can be used in 100% (B100) or blends with petroleum diesel.

LESS GREENHOUSE GAS EMISSIONS

Fossil fuels, when burnt, release greenhouse gases like carbon dioxide in the atmosphere that raises the temperature and causes global warming. To protect the environment from further heating up, many people have adopted the use of biofuels. Experts believe that using biodiesel instead of petroleum diesel can reduce greenhousegases up to 78%.

BIODEGRADABLE AND NON-TOXIC

When biofuels are burnt, they produce significantly less carbon output and few pollutants. As compared to petroleum diesel, biodiesel produces less soot (particulate matter), carbon monoxide, unburned hydrocarbons, and sulfur dioxide. Flashpoint for biodiesel is higher than 150°C, whereas the same is about 52°C for petroleum diesel, which makes it less combustible. It is, therefore, safe to handle, store and transport.

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LITERATURE REVIEW

Shakinaz A. El Sherbiny, Ahmed A. Refaat et(al) stated that the Biodiesel production is worthy of continued study and optimization of production procedures because of its environmentally beneficial attributes and its renewable nature. Non- edible vegetable oils such as Jatropha oil, produced by seed-bearing shrubs, can provide an alternative and do not have competing food uses. These oils are characterized by their high free fatty acid contents. The authors showed that the application of radio frequency microwave energy offers a fast, easy route to this valuable biofuel with the advantages of enhancing the reaction rate (2 min instead of 150 min) and of improving the separation process. The methodology allows for the use of high free fatty acid content feedstock, including Jatropha oil.

Veera Gnaneswar Gude, Prafulla Patil et(al) explained about the review's principles and practices of microwave energy technology as applied in biodiesel feedstock preparation and processing. Analysis of laboratory scale studies, potential design and operation challenges for developing large scale biodiesel production systems are discussed in detail. Laboratory scale microwave applications in biodiesel production proved the potential of the technology to achieve superior results over conventional techniques. Short reaction time, cleaner reaction products, and reduced separation-purification times are the key observations reported by many researchers. Energy utilization and specific energy requirements for microwave-based biodiesel synthesis are reportedly better than conventional techniques.

SUMMARY OF LITERATURE REVIEW

- Transesterification process is the most effective process in biodiesel synthesize from vegetable oils.
- Methanol is very effective and frequently used alcohol in biodiesel production.
- Ethanol is non toxic, biologically derivable alcohol rarely used for biodiesel products due to its less reactivity than methanol.
- Biodiesel production using microwave assisted heating and ultrasonic assistedstirring may improve ethanol reactivity.

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RESEARCH GAP

- Limited studies are carried out using microwave assisted transesterificationprocess for biodiesel production with ethanol.
- Optimization of microwave heating temperature has not been studied forsunflower oil ethyl ester production.
- Low cost experimental setup for microwave assisted transesterification processwas not developed.

OBJECTIVES

Extensive literature review has been conducted and the key summery was also prepared based on the literature review, also research gaps were identified in the previous chapter. The main objective of this research work has been framed based on the research gap identified and listed below.

- Design and fabrication of low cost microwave assisted transesterification setup.
- Optimization of microwave reaction temperature for maximum yield of sunflower oil ethyl ester.
- Physiochemical characteristics of raw sunflower oil and sunflower oil ethyl este

MATERIALS AND METHODS

METHODOLOGY

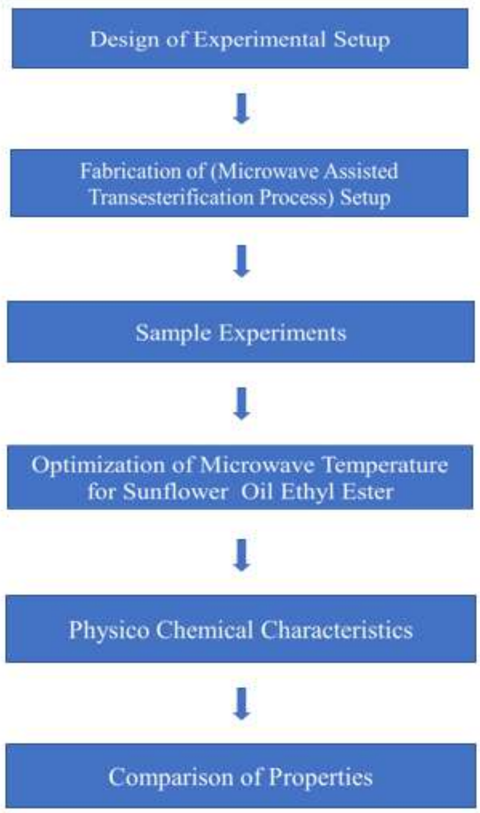

Figure 4.1. Flow chart of overall methodology

The overall methodology followed in this research work is shown in the figure no 4.1. Step-1 is dealing with the design of microwave assisted transesterification. In this all the required resources and their capacities

Industrial Engineering Journal
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with specifications were decided and procured. The performance of the fabricated experimental setup was ensured with some sample experiments and corrections were made in the fabrication setup functionality. Once experimental setup was made perfect the Optimization study in sunflower oil ethyl ester reaction temperature was carried out. The physiochemical characteristics of raw oil and sunflower oil ethyl ester were evaluated and compared with the existing literature.

MATERIALS FOR FABRICATION

SOLO MICROWAVE OVEN

A solo microwave is a basic microwave oven. It is great for uniform reheating. In solo ovens there's nothing except a magnetron — high voltage devices that produce ultra- high frequency (UHF) electromagnetic radiation. These electromagnetic rays penetrate the material kept inside the oven cavity and heat it up.

MECHANICAL STIRRER

Mechanical stirrer its laboratory equipment consisting of an electric motor that drives the blades ended metal rod immersed in the mixed liquid. Mixers are available with different speed and advancement, equipped with a digital display or without display- depending on the requirements. As we required a speed of 450 to 600 rpm, we went through the mechanical stirrer which is cost efficient.

CONDENSER

A condenser is a heat exchanger used to condense a gaseous substance into a liquid state through cooling. As the gas molecules releasing from round bottom flask coming towards the condenser, the reaction takes place inside the condenser through Cooling system. Gas molecules are turned into liquid state inside the condenser.

THREE MOUTH ROUND BOTTOM FLASK

Three neck flasks are used for a wide variety of applications where it is necessary to connect multiple components i.e., condensers, stirrers, etc., directly to the flask. Main
reaction takes place inside the flask, where one opening is fixed to the condenser. Other Opening is fixed to mechanical stirrer and last opening is closed with a rubber cork.

BORON SILICATE GLASS AND TEFLON IMPELLER

Borosilicate glass has a wide variety of uses ranging from cookware to lab equipment, as well as a component of high-quality products such as

Industrial Engineering Journal
ISSN: 0970-2555
Volume : 52, Issue 4, April : 2023

implantable medical devices and devices used in space exploration. Teflon impeller is rotors used in machinery. The action of this Teflon impeller increases the velocity and pressure of the fluid, and also serves to direct it.

THERMOCOUPLE

Thermocouple is a temperature-measuring device consisting of two wires of different metals joined at each end. One junction is placed where the temperature is to be measured, and the other is kept at a constant lower temperature.

TEMPERATURE CONTROLLER

Temperature Controller is a device that is used to control a heater or other equipment by comparing a sensor signal with a set point and performing calculations according to the deviation between those values. The controller takes an input from a temperature sensor and has an output that is connected to a control element such as a heater. Controller in a temperature control system will accept a temperature sensor such as a thermocouple or RTD as input and compare the actual temperature to the desired control temperature, or set point.

FABRICATION PROCESS

The basic design is to make 2 cavities on the top of the oven in order to connect the condenser in one and to immerse stirrer in another. In addition to this the third cavity is added at the time of fabrication for the temperature controller in order to maintain a balanced temperature in the flask for long period of time during experiment. After making cavities on the top of the oven the mechanical stirrer has been fabricated and replaced with boron silicate glass rod and Teflon impeller, it is inserted through the middle cavity. Thermocouple is connected to temperature controller for maintaining required temperature which is inserted thorough the last cavity. Third cavity is used to insert condenser and it is inserted in third mouth of the flask.

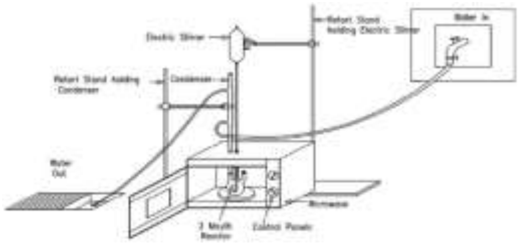

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Figure 4.2. Modified Microwave Oven

MATERIALS FOR BIODIESEL PROCESS

Vegetable oil was collected from the

Industrial Engineering Journal
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Volume : 52, Issue 4, April : 2023

local stores in padur, Chennai, India. Pellet form laboratory grade potassium hydroxide with 85% purity and 99% pure ethanol were used in this experimental work. The experimental setup consists of a 250 ml capacity borosilicate glass reactor with three necks, speed governable mechanical stirrer with carrying glass rod and Teflon impeller, temperature controller of range 0°C to 650°C and a thermocouple.

MICROWAVE ASSISTED TRANSESTERIFICATION PROCESS

Transesterification reactions were carried out in the presence of KOH catalyst (1.0% and 1.5% by weight of oil) at various reaction temperatures (60°C and 70°C). The stoichiometry for the reaction is 6:1 alcohol to oil. The catalyst was dissolved in ethanol and the resulting solution was added to the oil. This reaction mixture was heated inside microwave oven with temperature controller with the reaction time of 90 minutes. The reaction was timed as soon as the transesterification temperature was achieved by microwave.

OPTIMIZATION OF MICROWAVE REACTION TEMPERATURE

The Microwave reaction temperature for sunflower oil ethyl ester production was optimized using conventional optimization method. In this method all other experimental setup were kept constant such as 1 percent W/W catalyst 90 minutes reaction time, 500 rpm stirring speed, 6:1 molar ratio of oil to ethanol. Microwave reaction temperatures were varied from 40°C to 80°C with ten degree step increment.

PHYSICOCHEMICAL AND FATTY ACID PROPERTIES OF RAWOIL AND SUNFLOWER OIL ETHYL ESTER

The physicochemical properties of the raw sunflower oil and sunflower oil ethyl ester are measured using different parameters such as Viscosity, Density, Acid value, Flash point, Cloud point, Pour point, and Specific gravity.

RESULTS AND DISCUSSIONS

FATTY ACID PROFILE OF RAW SUNFLOWER OIL

Industrial Engineering Journal
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Fatty acid composition	Value (%)
C14:0	0.09
C16:0	6.33
C18:0	3.45
C18:1	21.64
C18:2	66.74
C18:3	0.09
C20:0	0.23
C20:1	0.13
C22:0	0.72
Other	0.04

Table 5.1. fatty acid composition

PHYSICOCHEMICAL PROPERTIES OF RAW SUNFLOWER OIL

PROPERTY	SUNFLOWER OIL
Viscosity	42.60
Density	0.921
Acid value	0.33
Flash point	272
Cloud point	-6.5
Pour point	-18
Specific gravity	0.913

Table 5.2. Characteristics of Raw Sunflower Oil

The physicochemical properties of raw oil were estimated and presented as follows. The density of oil at 15 °C was 0.921 g/cm3, the kinematic viscosity at 40 °C was measured as 42.60 mm2 /s, the estimated acid value of the oil was 3.32mg KOH/g, and the free fatty acid value was determined as 1.96%. Free fatty acid content more than2.5% in the oil leads to high saponification reaction with KOH during ethyl ester synthesis; hence, biodiesel synthesis is to be carried out in two steps. The iodine value of the oil was estimated at 64.3 g iodine/100g. The physical appearance of the oil was clear brownish-yellow, and the molecular weight was determined as 873.31 g/mol. The ester content in the SOEE is 91.8% which is 4.4% lesser than the minimum limit of EN14214 standard. The density of SOEE is 0.872 g/cm3, which is within the limitations of biodiesel standards. The kinematic viscosity is 5.24 mm2 /s, which is within the maximum limit of biodiesel requirements and somewhat higher than SOEE. The acid value is 0.32 mg KOH/g, which is considerably within the maximum limit of biodiesel standards and nearly double the SOEE. Other properties, such as heating value, pour point and flash point are within biodiesel requirements. The fatty ester profile of SOEE, as determined by GC. These are other common biodiesel fuels, including rapeseed ethyl esters (REE), soybean oil ethyl esters (SEE), and palm oil ethyl esters (PEE). The main ethyl ester was linoleic acid (55.2%), followed by oleic (33.2%), palmitic (7.0%) and stearic (3.5%) acids. The present GC analysis supported the fact that SOEE produced under optimized

Industrial Engineering Journal
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Volume : 52, Issue 4, April : 2023

protocol had its free and total glycerol contents with in ASTM D 6751 specifications. Non-optimized ethanolysis experiments yielded impure SOEE with sizeable residual amounts of free and total glycerol. The quality of optimized SOEE was verified and determined.

OPTIMIZATION OF SUNFLOWER OIL ETHYL ESTER

The Optimization of Sunflower oil Ethyl Ester was done under different temperatures and the yield was calculated for each temperature. At 40°C the yield was less when compared to other temperatures i.e. 68.7%., At 50°C the yield was 73.2%, At 60°C and 70°C the yield was 91.5% and 92.8% respectively. The yield was recorded highest at 70°C. As the temperature increases to 80°C the yield will be reduced to 82.4%.

Graph 5.1. Optimization of Sunflower Oil Ethyl Ester

Figure 5.1. Sunflower Oil Ethyl Ester

PHYSICOCHEMICAL PROPERTIES OF SUNFLOWER OILETHYL ESTER

Property	Method	SOEE
Specific gravity (15 °C)	D 287	0.87±0.03
Kinematic viscosity (40 °C, mm 2 /s)	D 445	4.75±0.02
High heating value (MJ/kg)	D 4868	45.8±1.1
Flash point (°C)	D 93	172.0±2.9
Cloudpoint (°C)	D 2500	1.0±0.1
Pour point (°C)	D 97	-3.0±0.3
Acid value (mg KOH/g)	D 664	0.26±0.03
Free glycerin (%)	D 6584	0.014±0.001
Total glycerin (%)	D 6584	0.204±0.010
Combustionpoint (°C)	D 92	179.0±2.8

Table 5.3. Properties of Obtained SOEE

CONCLUSION

Results of present study demonstrated that the optimum conditions elucidated for the ethanolysis of sunflower oil were: 6:1 molar ratio of sunflower oil to ethanol, 60 1Creaction temperatures, and 1.00% (w/w) KOH catalyst. The optimized conditions provided Sun flower oil ethyl ester in high yield (92.8%). The fuel properties of Sun flower oil ethyl ester measured were also within prescribed ASTM D 6751 specifications. Thus indicating that sun flower ethyl ester is acceptable as a substitute for other common biodiesel and petrodiesel fuels for combustion in compression- ignitions (diesel) engines.

FUTURE SCOPE

- Optimization other parameters of molar oil ratio etc
- Engine performance of Sunflower oil ethyl ester in unmodified diesel engine
- Engine performance of Sunflower oil ethyl ester with some nano addition

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