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Experimental Investigation & Validation of Properties of Synthesized Biodiesel from Non-edible Oil Seeds

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Abstract— Fossil fuels are the most prominent energy source for rapid growth of nation's economy and fast developing industrialization since many decades. Stringent environmental norms and energy security has forced researchers to think for alternatives energy resources. Biodiesel is one of the alternatives that can replace conventional fossil fuel and can use in any conventional engine, without any design modifications. Several technologies are developed to reduce harmful gases emitted by the gasoline engine, but if fuel itself is not clean, then all these design modifications are not affordable and sustainable. So the main aim of this research is to carry out experimental investigation of properties of biodiesel to improve oil recovery and good quality of fuel from Jatropha, Cotton seeds and Undi oil using transesterification process. Also, it deals with validation of properties of synthesized biodiesel as per ASTM D-6751 standards. The result showed that modified biodiesel plant has oil recovery above 90% for jatropha, and undi oil, properties of synthesized biodiesel are also validated as per standards.

Index Terms— Biodiesel, Non-edible Oil, Transesterification Process, Oil Recovery, Quality Fuel.

1 Introduction

FOSSIL fuel consumption level is predicted to increase by 60% by 2030 mainly because of population growth, industrialization and exposure to better living standards. Stringent environmental norms are present day problem for all automotive manufacturers since diesel fuel is not capable of meeting standards. Also, technology to reduce harmful green house emissions is not affordable to customers. Biodiesel as fuel has the capability of meeting stringent environmental norms which are purely renewable and eco-friendly fuel. Biodiesel is renewable fuel consisting of short chain alkyl esters made by transesterification process from various edible, non-edible oil seeds, animal fats, and waste vegetable oils. Non edible oil seeds are at disputation in many countries since due to food security.

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Non edible oil is highly recommended since its Free Fatty Acid (FFA) is high, not suitable for human consumption and it has the capability of significant oil recovery. The government of India has taken initiatives to cultivate Jatropha in 1.72 million hectares of total land and land is already allocated to some public sectors. Considering draught conditions in Maharashtra and to provide some income source for farmers non-edible like Jatropha, Cottonseed and Undi are selected as feedstock oil in this research.

The Jatropha curcas Linnaeus plant is a hardy shrub that can grow on poor soils and areas of low rainfall. The oil content of Jatropha seed ranges from 25% to 30% by weight. Fresh Jatropha oil is slow-drying, odorless and color less oil, but it turns yellow after aging. The non-edible oil contains about 3-4 %wax and gum. De-waxing and degumming of plant oils are required not only for the smooth running of the CI engine but also to prevent engine failure even if plant oils are blended with diesel. It is, therefore, necessary to remove wax and gum from the fresh oil before it could be used in CI engine. [1-2].

Undi is a species of family Guttifereae (Clusiaceae), native to India, East Africa, South east Asia, Australia and South Pacific. Commonly it is called as Indian laure, Alexandrian Laurel, Beach Calophyllum. [3] The oil content of undi seed ranges from 55% to 60% by weight. The greenish yellow oil with disagreeable odour contains 28.08mg of KOH/gm FFA. The undi oil obtained from the Calophyllum seeds was used as an alternative to candlenut oil in lamps. Selected feedstock oil has the potential to grow in bare land and one rainfall is sufficient to yield seeds. Also, the life of selected feedstock plant is more than 100 years except for cotton plant which has a life of about 70years. [2-3]

Transesterification process is the conversion of ester and glycerol to form methyl ester (biodiesel) in the presence of a catalyst. So the selection of catalyst is one of the important parameters for transesterification process.[3] Reaction time is greatly affected by the type of catalyst used in transesterification process. Generally, homogeneous catalyst such sodium and potassium oxide are usually preferred at the wide scale. In this research homogeneous catalyst was replaced by heterogeneous catalyst aluminum trioxide which is ecofriendly and reusable for various batch production.[4] Also, it is of comparatively noncorrosive and non-toxic which is the important parameter for selection of material for the reactor vessel.

Lab scale studies were carried out to study existing process at Indian Biodiesel Corporation (IBDC), Baramati. Study of existing design at IBDC was carried out to set design conditions for commercial biodiesel production process. Biodiesel production process was studied and experimentation to set reaction time, temperature, pressure, molar ratio and stirrer speeds was done on lab scale basis. Many researchers have given priorities to optimize various parameters of biodiesel production process, but this research was extensively carry out to the effect of various design parameters involved in the design of biodiesel plant .

The quality of biodiesel is a key factor when it is used as an alternative to conventional diesel fuel. The quality of biodiesel fuel should meet ASTM D6751 standards within recommended limits so that it can be used in modern engines without modifications maintaining engine durability and maintainability. Another major problem of biodiesel fuel is poor oxidation stability which is of main concern behind not using biodiesel as fuel i.e. it alters its properties with time. Based on design modification done at IBDC result has shown good oxidation stability for set design conditions and parameters. Also, oil recovery above 90% of selected oil is achieved without affecting the quality of synthesized biodiesel. Design modifications were done considering economic and sustainability of biodiesel plant for various feedstock oil. However, it was found that undi oil had shown maximum oil recovery about 93% for designed biodiesel plant. Designed reactor vessel volume has shown good triglyceride conversion which is the important parameter of esterification process. Biodiesel synthesized from designed biodiesel plant has shows that 100% blending can be achieved as per ASTM D6751 standards.

2 METHODOLOGY

2.1. Design of Biodiesel plant

Biodiesel production process is carried out in three steps pretreatment, biodiesel production process, and purification. Sincethis project deals with design modifications of existing plant at IBDC to oil recovery and quality of fuel. Following are the design parameters which has maximum influence on oil recovery.

2.1.1 Design of Reactor Vessel

Biodiesel reactor vessel design was modified with eliminating pre-treatment vessel and carrying out esterification and biodiesel production process in same reactor vessel without affecting the quality of methyl ester produced.

i. Sizing of reactor unit

50 liters capacity of reactor vessel suitable for batch production system was designed. Sizing calculation for reactor vessel is done according to equation 1,

$$V = \frac{\pi D^2}{4} H \tag{1}$$

H/D = height to diameter ratio is taken as 1.5

H= 525 mm.

D= 350 mm

Therefore reactor vessel was designed to handle the volume of 50.511 liters of reactant volume

ii. Design conditions for reactor vessel

a. Design Pressure

According to standards, design pressure (internal) should be equivalent to the pressure set for the relief valve.

Internal pressure (gauge) (Pg) is taken as 25kpa, and hydrostatic pressure is calculated using Equation 2 assuming the average density of feedstock oil as 920 kg/m3 and liquid height approximately 500mm.

Hydrostatic pressure, $P_h = \rho g H$ (2)

= 920×9.81×0.5

= 4512.6 Pa

(Ph) = 4.5126 Kpa

Therefore as per standards design pressure is calculated as,

$$P_d = P_h + P_g \quad (3)$$

= 4.5126+25

Pd = 29.5126 Kpa.

b. Design Temperature

Generally, transesterification process is carried out at the temperature range of 60-120°C. Maximum operating temperature of reactor vessel was taken as 85°C with 10°C additional safety margin. Therefore design temperature was taken as 95°C which facilitates in the selection of design of heating element.

iii. Material Selection

Selection of material for biodiesel production process was done according to design conditions. Also material should be capable to withstand chemical reactivity during the process. SS304L material is selected according to manufacturer's recommendation for the design of reactor vessel and carbon steel for the structure of biodiesel plant structure.

iv. Selection of Design Stress

According to British standard PD500, the design stress or allowable stress σall of selected fabricating material SS304L at operating temperature 95°C is taken as 145 N/mm^2.

v. Calculation for reactor vessel thickness

Let,

σ_{all} = allowable tensile stress for cylindrical vessel shell =145 N/mm2

P_i = Design Pressure (internal pressure) = 29.512 Kpa

D_i = inner diameter of the cylindrical vessel shell = 350mm

t = thickness of the shell without corrosion allowance, mm

Therefore,
$$t = \frac{P_i \times D_i}{2 \times \sigma_{all} - P_i} \quad (4)$$

Therefore, t = 2 mm, the minimum allowable thickness was selected according to pressure vessel design standards.

vi. Design of end closure for reactor vessel

Considering economic feasibility and ease maintainability flat plate head closure with bolted cover was selected for designed reactor vessel. Minimum thickness required for plate was estimated using equation 5,

$$t = C_p D_e \sqrt{\frac{P_i}{\sigma_{all}}} \quad (5)$$

where,

C_p = design constant dependent on edge constant = 0.4

D_e = effective diameter = 0.390 mm

Therefore thickness of end closure was estimated to 4mm.

2.1.2. Design of Mixing/ agitator mechanism

The design of mixing mechanism is a critical parameter since it affects reaction time need to complete the transesterification process and ultimately quality of biodiesel fuel. Mechanical agitator or stirrers are commonly used for mixing purposes but with the aid of an electric motor. Considering economic feasibility and safety constraints as mechanical agitators are more prone to ignition and electric spark it may cause the explosion. Mechanical agitators were replaced by Jet mixing mechanism as shown in fig has significant oil recovery in comparatively less reaction time. The design of Jet mixing mechanism includes calculation of various design parameters such as jet nozzle diameter d_i, critical velocity V_c to estimate jet velocity and minimum circulation rate Q_c.

i. Jet nozzle diameter

According to [3] geometry of single side entry nozzle was selected according to equation 6,

$$0.25 \le \frac{H}{D} \le 1.5 \quad (6)$$

where,

H = Liquid height in tank

D = Diameter of reactor vessel = 350 mm

According to [3] it was reported that liquid height in the tank should be within 0.25 to 1.5 times diameter of the reactor vessel. Higher H/D ratio is recommended for better mixing ability. Therefore the liquid height of reactor H was estimated to 490mm. Dimensions selected for jet mixing mechanism are shown in Fig.1,

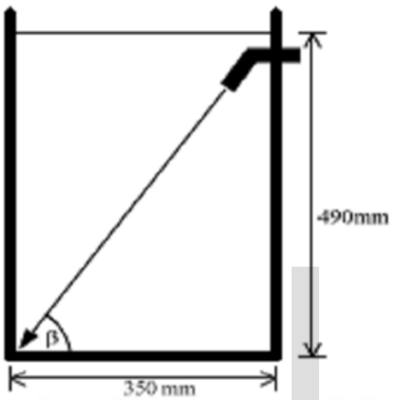

Fig.1 Dimension selected for jet mixing mechanism.

X as shown in Fig 2 was calculated using geometrical relationship, $X = \sqrt{H^2 + D^2}$

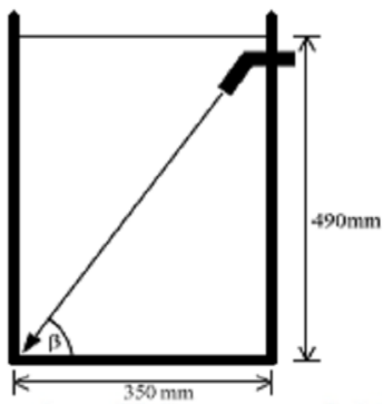

Fig 2 Jet length X for side entry

Therefore X = 609mm. According to above relationship and jet nozzle diameter, dj is estimated using relationship given in equation, is selected within 1.36 and 10.88mm. Therefore diameter of the nozzle is selected as 10mm.

ii. Critical jet velocity Vc

The critical velocity Vc which is necessary to estimate jet velocity is estimated using relation 7,

$$V_c = \left[\frac{2gGH\left(\frac{\rho_2 - \rho_1}{\rho_2}\right)}{\sin^2\theta} \right]^{0.5} \quad (7)$$

where,

$\theta = (\beta + 5)^{o}$,

β = angle of inclination of jet nozzle to horizontal

ρ_2 = density of heavy liquid

ρ_1 = density of light liquid

G = constant based on stratification data for jet mixing

Therefore Vc is estimated as 5.21 m/s.

iii. Minimum liquid circulation rate Qc

Minimum circulation rate Qc was estimated using relations (8)

Therefore, circulation pump selected for pumping capacity equivalent to Qc = 2.213 m^3/ hr.

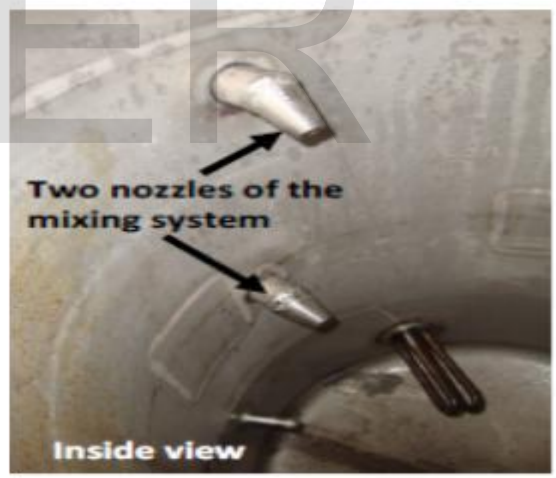

Fig 3 Jet nozzle arrangements for reactor vessel

2.1.3. Design of heating element

Generally, a temperature range of 55-120°C is desired to carry out transesterification process, and temperature has the significant effect on total reaction time needed to complete the process. Also, it is ensured that properties of synthesizes biodiesel should meet norms while selecting appropriate temperature range. A heating element has been designed to meet temperature requirement of transesterification process. Considering controllability and safety of the system, it was recommended to use the electric heating system to meet process requirement as shown in Fig 4.

Fig.4 Arrangement of electric heating element

2.1.4. Design of settling unit

Settling unit or storage tank design was modified to equip with air sparging system to carry out biodiesel washing process. The maximum volume of settling tank was designed to handle 50 liters of reactants. It was recommended to design settling tank with additional 10 % volume (i.e 5 liters) to accommodate liquid variations during biodiesel washing process.

2.1.5. Design of air sparging system

Air sparging system designed for settling/ storage tank is used for biodiesel washing process. It consists of mesh-like tube structure which can be submerged horizontal to the bottom of the vessel and a vertical tube mesh from outside as shown in fig. The complete system was fabricated using 152.4mm and 76.2mm SS304L tubes with 2mm holes drilled to the bottom side of tube mesh to allow escaping pressurizes air as tiny bubbles as shown in Fig 5.

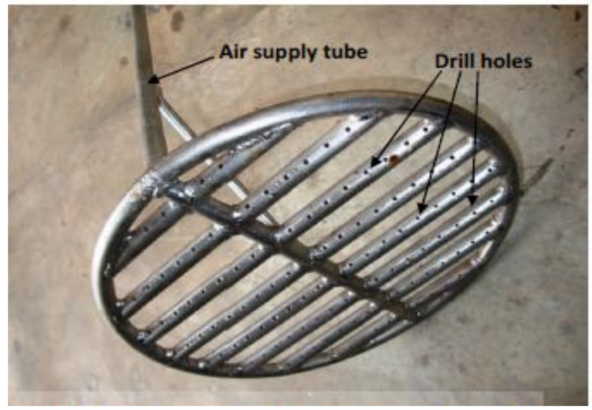

Fig. 5 Tube structure for air sparging system

2.1.6. Selection of piping system

A piping system suitable for design conditions for transferring reactants was selected replacing stainless steel pipes with flexible clear PVC tubing. It allowed transparency to allow operator visually inspect process, and it is flexible for assembling and dismantling whenever required.

2.2. Biodiesel production process

Due to design modifications esterification is carried out in reactor vessel inspite of pretreatment vessel. Esterification process is carried out to reduce free fatty acid (FFA) value of selected oil. Jatropha oil first heated to 50°C then 1.7% (by wt. of oil) sulfuric acid was to be added to heated oil and methyl alcohol about 1:8 molar ratio (by wt of oil) added afterward in 50 litres designed reactor. The reaction started with stirring speed about 950 rpm and temperature was controlled at 55-60°C for 60 min with regular analysis of FFA every after 25-30 min. Finally, the FFA was reduced upto 1.5% then the excess methyl alcohol was removed by distillation, and esterified oil was transferred into settling tank. The trace quantity of moisture was formed in this step, which was removed. The major obstacle to acid catalyzed esterification for FFA is the water formation. Water can prevent the conversion reaction of FFA to esters from going to completion [28]. Afterward, the esterified oil was now used to carry out transesterification process [29].

1.2wt % of aluminum trioxide was added in the reactor vessel to dissolved with methyl alcohol (8 mol of that oil) at 40 0C; then this mixture was slowly added to heated oil & reaction started for 90 min with stirring speed about 650 rpm and at 55-60°C temperature. After reaction completion i.e. when FFA reduced up to 0.7% the transesterified oil was again transferred to settling/storage tank. The three distinct layer of methyl ester, glycerol, and unreacted oil were formed. The glycerol settled at the bottom of storage tank due to gravity and jatropha methyl ester were settled at the top of the storage tank. The unreacted oil settled in between glycerol and methyl ester layer. The glycerol was separated manually. Then the product i.e. jatropha methyl ester was washed with the help of air designed air sprager to remove dissolved sulphuric acid, sodium, methyl alcohol and glycerol. The wasted biodiesel was dried over anhydrous sodium sulphate. The purified methyl ester was preceded for quality testing.The undi methyl ester and thumba methyl ester was synthesized as per protocol using aluminium trioxide as a strong base catalyst and methyl alcohol [17], [20]. Same process was carried out to synthesize biodiesel from cotton seeds oil and undi oil.

2.3. Experimental Set-up at IBDC

Designed 50 liters capacity of biodiesel plant with suitable design modifications at IBDC is shown in Fig 6

Fig 6 Designed Biodiesel Plant at IBDC

3. Results And Discussion

Biodiesel synthesized from modifications in the design of Biodiesel plant has shown significant results as discussed below.

3.1. Effect of internal pressure on reactor vessel thickness

Reactor vessel used for biodiesel production process designed to withstand pressure between 20-40 N/m^2. However, increase of internal pressure has to incorporate with the increase in thickness of pressure vessel. Designed reactor vessel with 29.515 N/m^2 pressure is optimum and safe against design conditions set for the biodiesel plant.

3.2. Effect of Reactor Volume on Triglyceride Conversion

Reactor vessel volume has the significant effect on triglyceride conversion i.e. conversion FFA on methyl ester. According to ASTM standards, triglyceride conversion should be not more than 0.30 (wt%), designed reactor vessel has meet norms for selected oil from different feedstock as shown in Fig. 7

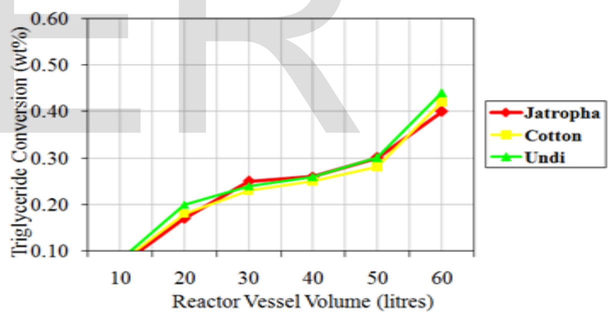

Fig 7 Reactor volume (litres) vs. Triglyceride conversion (wt%)

3.3. Effect of Modifications of mixing mechanism.

Replacement of mechanical agitators with jet mixing mechanism has reduced total reaction time required to carry out biodiesel production process. Synthesizes biodiesel was obtained in just 55 minutes of reaction times without affecting the quality of fuel. Also, it was not necessary to equip separate motor which ultimately led to cost saving.

3.4. Effect of Modification in the design of settling/storage tank.

Air sparging system designed for storage tank unit has reduced settling time and loss of oil recovery during washing process was completely eliminated. Due to which results has shown up to 90% oil recovery for selected feedstocks.

3.5. Oil Recovery

Oil recovery of existing plant at IBDC was 70-75% and due to design modifications incorporated oil recovery for selected feedstocks oil is increased as shown in Table 1,

Table 1 Oil Recovery of Designed Biodiesel Plant

Sr..No	Non-edible Oil Seeds	Oil Recovery (%)
1	Jatropha	90
2	Cotton	85
3	Undi	93

3.6. Validation of Synthesized Biodiesel as per ASTM standards

Validation of synthesized biodiesel is the important parameter in deciding the quality of synthesized. Results have shown that approximately all properties of synthesized biodiesel are as per norms.

Table 2 Properties of Synthesized Biodiesel

Properties	ASTM Standards	Properties of synthesized biodiesel Methyl Ester		
		Jatropha	Undi	Cotton
Density (g/cm^3)	0.860-0.900	0.872	0.892	0.87
Viscosity (cst)	3.5-5	3.82	3.87	3.8
Calorific Value (MJ/kg	35-45	38	37.18	37
Flash point (°C)	160-190	164	170	184
Fire point (°C)	160-190	171	165	172
Cetane Number	51	48	50	55

4. CONCLUSION

Design reactor vessel is safe against design conditions and has shown good triglyceride conversion rate for selected capacity. Also, the cost associated with selection of material is comparatively low since the SS304L material is cheaply available with manufacturer due to its good corrosive properties. Also, SS304L is the material which does not take into account corrosion allowance for the design of reactor vessel. Modification in mixing mechanism has reduced settling time required to obtain methyl ester. Also, good oxidation stability up to 8 hours is achieved using jet mixing mechanism. Electric heater employed is capable of controlling process temperature within limits with the aid of temperature controller. Design plant is compact since the pre-treatment vessel was completely eliminated carrying out esterification and transesterification process in the main reactor vessel.

Biodiesel plant designed at IBDC has shown the increase in oil recovery for selected non-edible oil seeds like Jatropha (90%), Undi (93%) and cotton seeds oil (85%) using transesterification process. Undi oil has shown recovery up to 90%. Also, heterogeneous catalyst (Aluminium trioxide) used for designed plant is reusable and eco-friendly, which is one of the additional benefits in economical concern. Also, properties of synthesized biodiesel are validated as per ASTM D6751 standards which show that 100% blending of synthesized biodiesel is possible in the conventional engine without any design modifications.

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