

Jawaharlal Nehru Technological University Hyderabad-
University College of Engineering Science and Technology Hyderabad (Autonomous)
Department of Chemical Engineering
IV/IV Year B.Tech.

MAJOR PROJECT

PRODUCTION OF BIO DIESEL FROM WASTE COOKING OIL



Under the esteemed guidance of

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INTRODUCTION

- Biodiesel is a non petroleum based diesel fuel consisting of short chain alkyl esters.
- Biodiesel is a substitute to diesel fuel obtained from triglycerides of vegetable oils or animal fat.
- Biodiesel is a clean renewable fuel.
- Biodiesel can be produced from oils like:
 - soybean,
 - cotton seed,
 - palm
 - peanut
 - rapeseed and so on

Biodiesel can be produced from four methods. They are:

1. Direct Blending:

- In this process vegetables oils are directly diluted in diesel to reduce viscosity. The ratio of oil to diesel 1:10-2:10 was found successful.

2. Pyrolysis:

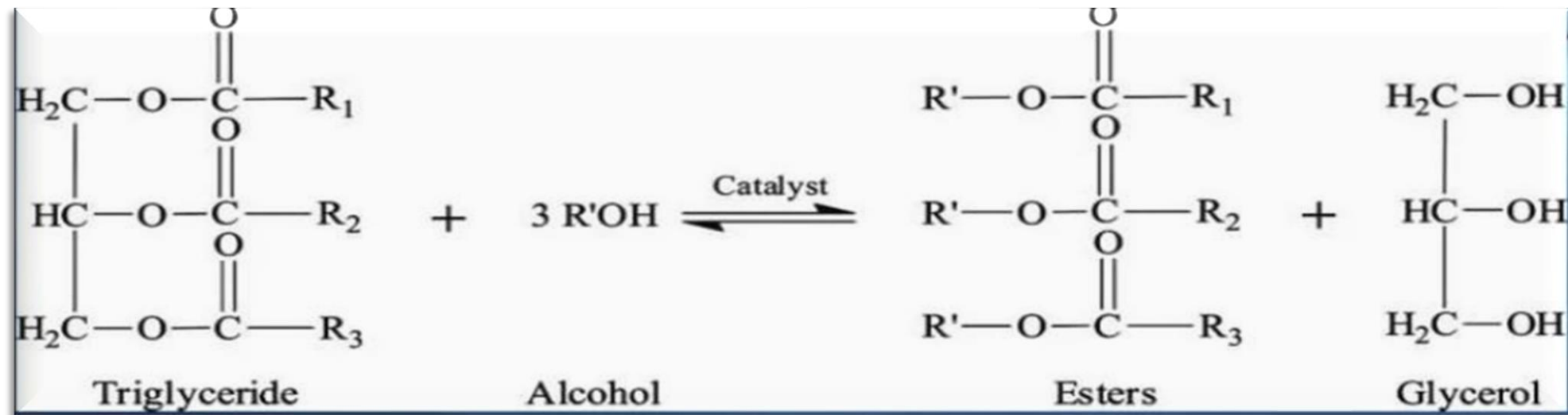
- It is the thermal decomposition of organic matter in the absence of oxygen with or without the presence of catalyst.
- This process decreases the density and viscosity of oils.

3. Microemulsions:

- It is defined as a thermodynamically stable, isotropic liquid mixtures of oil, water and surfactants
- This process increases cetane number.

Transesterification

- It is method of converting tryglycerides present in oil into methyl esters (biodiesel) by using alcohol and catalyst.
- In this process glycerine is used as by product which can be used in manufacturing of soaps, cosmetics and so on.
- It is the most common method to produce biodiesel.



ABSTRACT

- Bio-diesel is produced from the waste cooking oil. Alkali catalyzed transesterification process of waste cooking oil with methanol is used to produce biodiesel. In this transesterification process, NaOH/KOH are used as alkaline catalyst and H₂SO₄ is used as acid catalyst. For the production of biodiesel waste cooking oil is preheated for 65⁰C and then methanol and 1 weight % NaOH/KOH/H₂SO₄ catalyst are added into the heated waste cooking oil by stirring. The reaction mixture is heated for 1 hour at 65⁰C by using a magnetic stirrer. After that reaction mixture is transferred into separating funnel
- In the separating funnel reaction mixture is allowed to settle for 90 minutes. After 90 minutes reaction mixture is separated into two layers. The bottom layer is glycerol and top layer is biodiesel. Obtained biodiesel is washed using hot water in 1:1 ratio at 75 to 80⁰C twice for purification of biodiesel. Yield of biodiesel is tested for methanol to oil ratios from 1:1, 1:2, 1:3, 1:4 & 1:5 ratios. Acid values calculated by using titrations, saponification values using reflux, flash and fire points are obtained by PENSKEY MARTIN FLASH AND FIRE APPARATUS and viscosity calculated by using REDWOOD VISCOMETER, iodine values and cetane numbers are calculated and compared with the literature.

LITERATURE REVIEW

Names of the authors	Title	Conclusions from the work
Sahar	Biodiesel production from waste cooking oil	<ul style="list-style-type: none"> Transesterification was done in the presence of alkali catalyst (KOH) and Fatty acid methyl ester (FAME) yield was 94% in the presence of 1% catalyst at 50 °C. Among the different mineral acids, H₂SO₄ gives the efficient esterification of FFAs with 88% conversion efficiency. When we use 1:3 methanol to oil ratio 94% FAME yield was achieved.
M. Chandrasekara	Waste Cooking Oil Based Biodiesel and Its Combustion	<ul style="list-style-type: none"> It is observed that biodiesel has similar combustion characteristics as diesel and also found that the base catalyst performs better than acid catalyst and enzymes.
Danushka Thilakarathne	Production of Biodiesel from Waste Cooking Oil in Laboratory Scale	<ul style="list-style-type: none"> Transesterification is commonly used to reduce the viscosity during the production of biodiesel. Widely used catalysts are acids and bases. However, the transesterification is dependent on the molar ratio of alcohol and the weight basis of KOH that we use as the catalyst.
N.Phan	Biodiesel Production from waste cooking oils	<ul style="list-style-type: none"> The effect of methanol to oil ratio , potassium hydroxide concentration and temperature on biodiesel conversion were investigated acid values of oil samples varied from 0.67 to 3.64mg KOH/g. Low level acid value is an advantage to use alkali catalyzed transesterification.

Names of the authors	Title	Conclusions from the work
Tuba Hatice Dogan	Biodiesel produced from waste cooking oil	<ul style="list-style-type: none"> Some of the physical properties such as kinematic viscosity, density, cloud point and pour point were examined. In addition, total polar material contents, heating values and acid values of biodiesel produced from waste cooking oils were analysed.
M. F. Elkady	Production of Biodiesel from Waste Vegetable Oil	<ul style="list-style-type: none"> This study investigated the use of KM micromixer in the production of biodiesel from waste vegetable oil. The effects of methanol/oil molar ratio, catalyst concentration, volumetric flow rates, and the presence of a cosolvent on the transesterification reaction were examined.
M. Hassania	Two-step Catalytic Production of Biodiesel from Waste Cooking Oil	<ul style="list-style-type: none"> A pre-treatment stage is performed to remove impurities including wastewater, salt and other impurities from WCO. Then FFA content was reduced using sulfuric acid as a catalyst in the process of esterification. finally TG is converted to biodiesel in potassium hydroxide catalyzed transesterification reaction.
Rummi Devi Saini	Conversion of Waste Cooking Oil to Biodiesel	<ul style="list-style-type: none"> Transesterification process is a commonly employed for its formation. Water produced during the esterification process may inhibit acid catalyst. It can be removed by stepwise reaction mechanism. Methanol is the most suitable alcohol because of its low cost and easy separation from biofuel.

Selection of raw materials

WASTE COOKING OIL

- Initially the vegetable oil which is used consist of unsaturated fatty acids as we use the oil for number of times this unsaturated fatty acids converts into saturated fatty acids.
- In our requirement we need to have some unsaturated fatty acids, so we need to have the waste cooking oil which is not used for more than 5times.

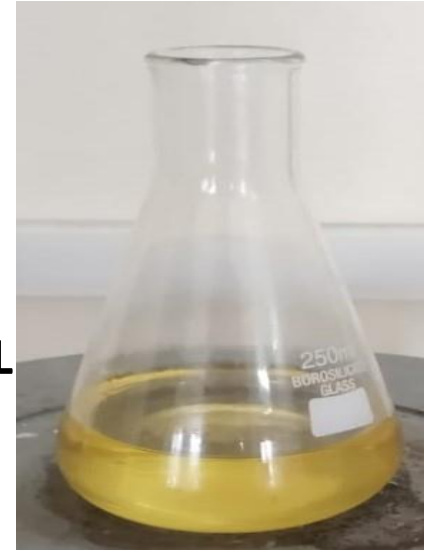
CATALYST

- Different types of catalysts such as acids and bases in which bases act as better catalyst compared to acids because of high conversion efficiency and acid catalysts which leads to hinderance in transesterification
- Bases like KOH OR NaOH are used but KOH is used as a better catalyst due to the better yield percentage.

ALCOHOL

- Though methanol gives high yield and takes lesser time where as ethanol is derived from bio-based resources.

**LESS USED
COOKING OIL**

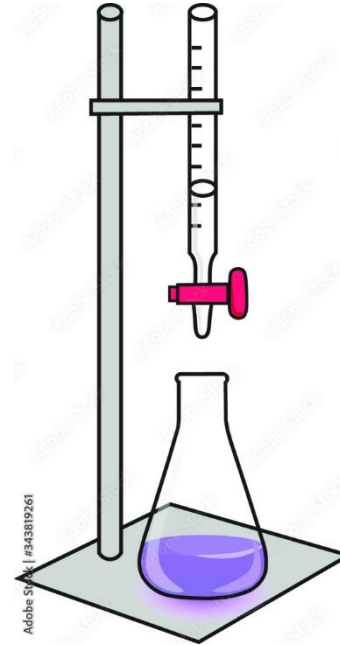


**HIGHLY USED
COOKING OIL**



PROCEDURE

- The FFA has been estimated with the procedure of titration using phenolphthalein as indicator and KOH as base
- FFA is less than 5% so we use direct transesterification method
- Initially pretreatment is done to the WCO and it is filtered using nylon filter cloth
- Initially the filtered oil is heated at 100°C to remove moisture on the heating pan.



Titration



Filtration

- 100 ml of methanol and NaOH (1.1% of weight of oil) is weighed and dissolved in a beaker to make the solution.
- When the sample oil reaches 60-65°C the make-up solution is added to it and the conical flask is enclosed to avoid the evaporation losses.
- 100 ml of Sample oil (WCO) in a 250ml conical flask is subjected to heating on the heating pan and temperature is set at 65°C.



Transesterification

- Magnetic stirrer and bead is used for continuous stirring to maintain uniform temperature and concentration throughout the reaction process.
- Constant temperature is maintained throughout the experiment period i.e., between 60-65°C.
- This experiment is allowed to happen for 60 minutes. Then the solution is cooled to the room temperature.

- Cooled solution is transferred into the separating funnel using funnel with filter paper in it.
- It is kept undisturbed for 60 to 90 minutes, then we can observe two immiscible liquids separated based on their density difference.
- The top liquid is Bio-Diesel (Product) and the bottom liquid is Glycerol (By-product).
- By opening the valve situated at the bottom of separating funnel glycerol is separated leaving our desired product bio-diesel in it.



Settling of bio-fuel and glycerol according to their density

- This biodiesel is subjected to purification (washing) process in which the product and deionised water are mixed in 1:1 volume ratio at 60-65 degree Celsius
- Now the suspension of bio-fuel and water is left overnight for settling
- After over night settling washed water and bio-fuel are separated/settled according to their density
- Then the washed liquid is drained using the valve situated at the bottom of the separating funnel
- Then washed bio-fuel is dried using air pump at 60-65 degree Celsius



Settling of Bio-fuel
& water

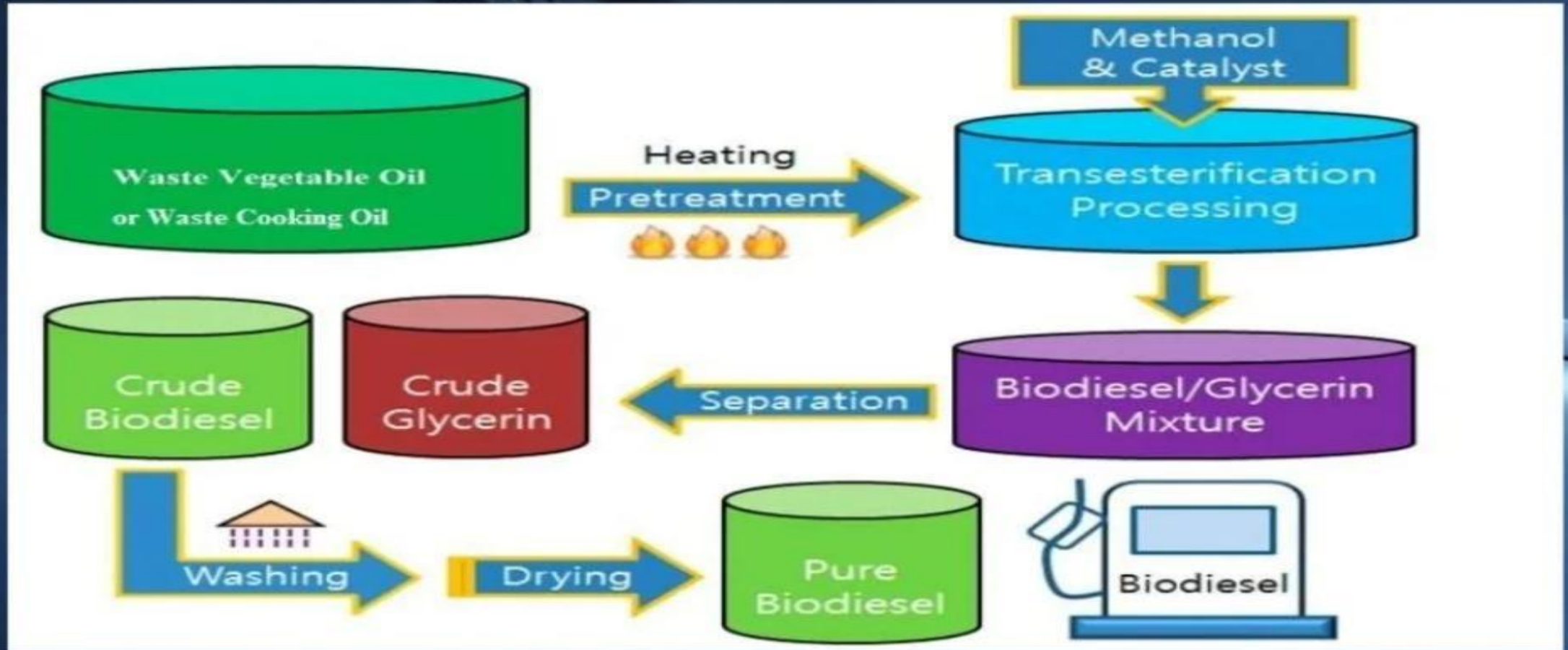


Bio-fuel and washed
water suspension

- This procedure is repeated for methanol to oil ratio i.e., 1:1, 1:2, 1:3, 1:4, 1:5.
- The product is then carried out for qualitative and quantitative analysis.

$$\text{Yield (\%)} = \frac{\text{amount of biodiesel produced}}{\text{amount of WCO used}} \times 100$$

FLOW DIAGRAM FOR BIODIESEL PRODUCTION



ANALYSIS OF BIODIESEL

Determination of Acid value:

- Mass of potassium hydroxide (KOH) in milligrams that is required to neutralize one gram of oil/fat/FAME is known as “acid value”. Units are mg KOH/gm of substance.
- For the determination of free fatty acid content, 0.5 g of biodiesel, 10 mL ethanol and 1–2 drops of phenolphthalein were used as indicator.
- The mixture was titrated against 0.1 N KOH and change in colour was noted.
- The FFA content was expressed g/100 g as oleic acid and calculated by

$$\text{FFA} = V \times N \times \frac{28.2}{w}$$

$$\text{Acid value} = \text{FFA (\%)} \times 1.989, \text{ mg KOH/gm of oil}$$

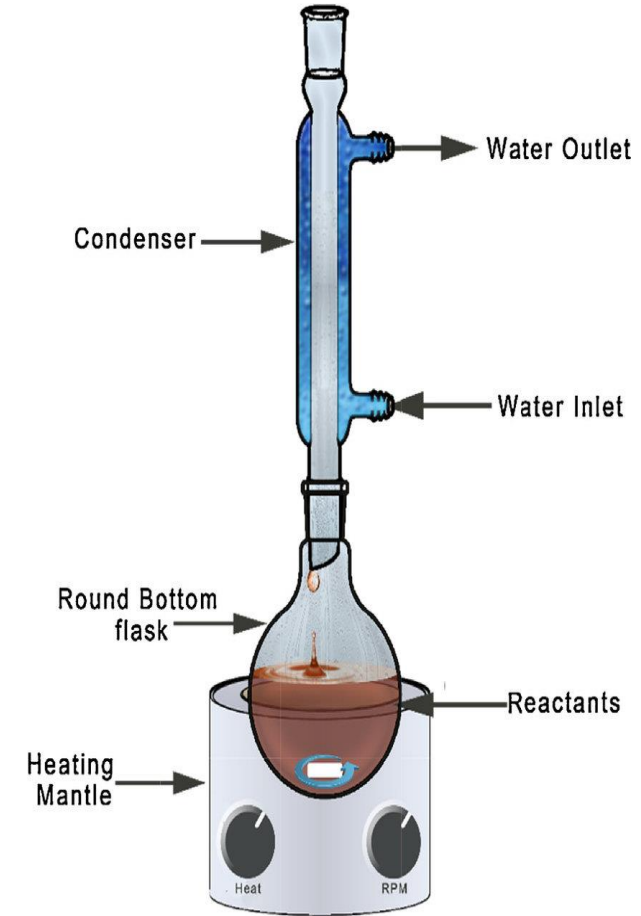
Where, V=burette reading

N = normality of NaOH/KOH used (0.1N)

w = weight of sample oil.

Determination of Saponification value:

- “Saponification value” represents the number of milligrams of KOH or (NaOH) required to saponify one gram of fat under the conditions specified. It is a measure of the average molecular weight of all the fatty acids present in the sample as triglycerides.
- For the determination of saponification value, the 0.5 g biodiesel and 20 mL of 0.5 N alcoholic (ethanol) KOH was mixed.
- The mixture placed in round bottom flask, refluxed and heated at 40 °C until clear solution, an indicated of saponification reaction.



- After cooling the contents, phenolphthalein was added as indicator and mixture was titrated against 0.5 N HCl until the pink colour disappeared.

- The saponification value was determined using relation,

$$\text{Saponification value} = (\text{B}-\text{S}) \times \text{N} \times \frac{56.1}{w}$$

Where, B and S are the blank and sample values (HCl)

N is normality of HCl

w is weight of biodiesel.

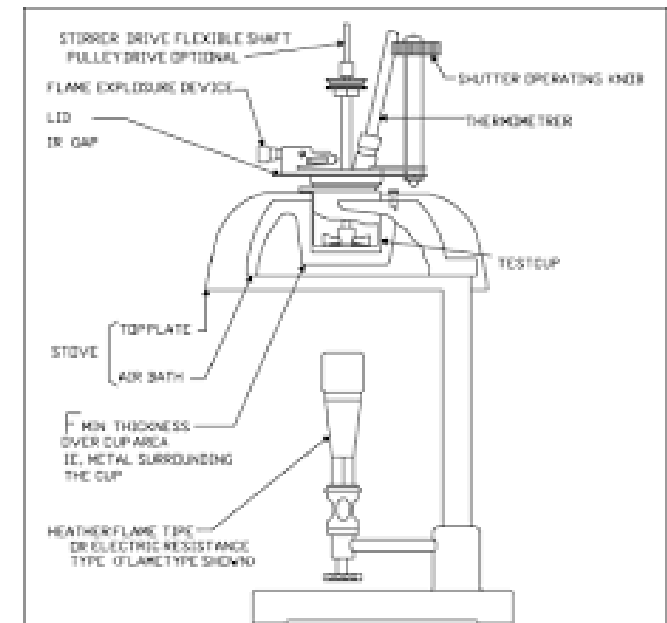


Flash and Fire point

- The flash point of a volatile material is the lowest temperature at which it can be vaporized to form an ignitable mixture in air.
- The fire point of a fuel is the temperature at which the vapour produced by that given fuel will continue to burn for at least 5 seconds after ignition by open flame.
- The flash and fire point of bio-diesel was found to be 112°C and 120°C respectively.



pensky martins apparatus

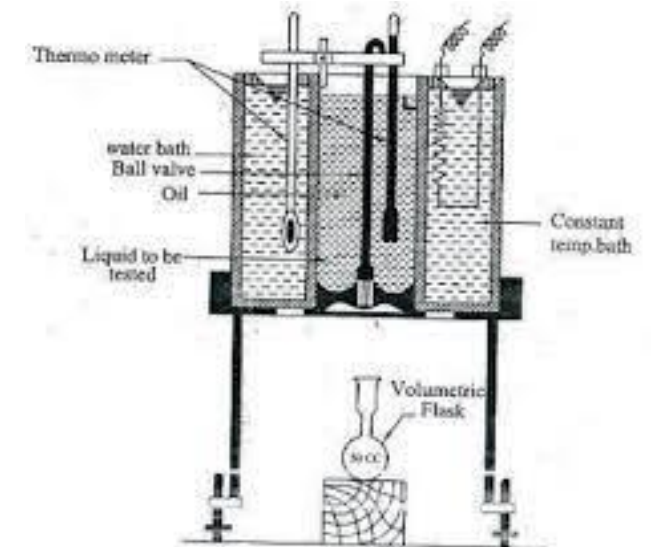


viscosity

- Viscosity: the resistance of a fluid (liquid or gas) to a change in shape or movement of neighbouring portions relative to one another.
- Redwood Viscometer is used for the determination of redwood viscosity and which can be converted to Kinematic viscosity also. It is also used for observing the effect of temperature on viscosity by plotting graph.



Redwood viscometer-1

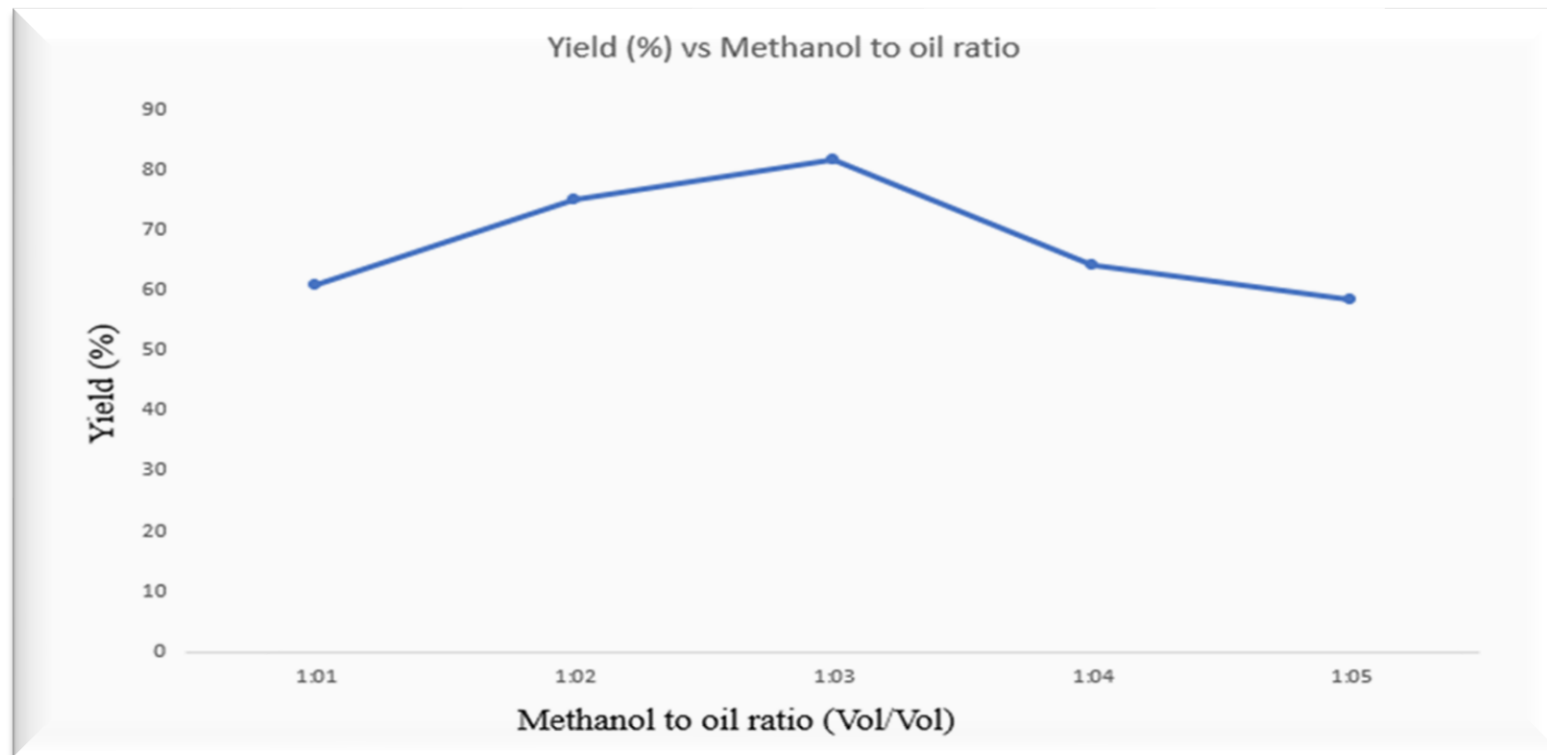


RESULTS AND DISCUSSIONS

- The calculated FFA value of the waste cooking oil is 2.483% which is less than 5% so, only trans-esterification process is required for the conversion of biodiesel.
- We have synthesized biodiesel by varying methanol to oil ratio from 1:1, 1:2, 1:3, 1:4, 1:5.
- The highest FAME yield (81.6%) was achieved at 1:3 methanol to oil ratio.
- Further increase in methanol concentrations decreased the FAME yield.
- The higher methanol to oil ratio might affect the solubility of glycerin and resultantly, FAME yield was decreased.
- Important influencing parameters for transesterification include methanol to oil ratio, catalyst dose, temperature and reaction time

Yield (%) for different methanol to oil ratio

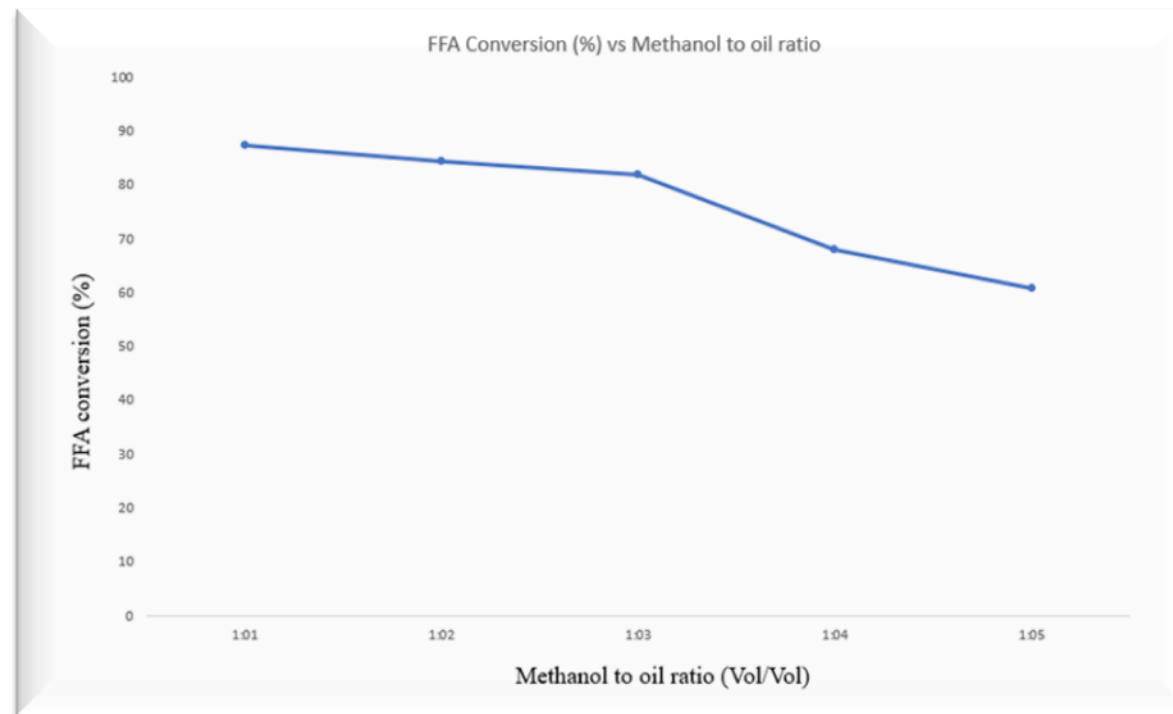
Methanol to oil ratio	Weight of WCO used (gm)	Weight of bio-diesel produced after washing (gm)	Yield (%)
1:5	89.07	51.945	58.32
1:4	71.69	45.97	64.12
1:3	53.31	43.5	81.6
1:2	37.686	28.244	74.95
1:1	18.35	11.157	60.8



YIELD(%) TO METHANOL TO OIL RATIO

Free Fatty Acid conversion (%)

Methanol to oil ratio	Saponification value of biodiesel (mgKOH/g)	Acid value (mgKOH/g)	FFA conversion (%)
1:1	319	0.62	87.4
1:2	308.2	0.769	84.41
1:3	304.7	0.89	81.99
1:4	149	1.57	68.12
1:5	218	1.93	60.93



Fatty Acid Conversion to methanol to oil ratio

- The FFAs conversion was increased to 87.4% from 60.93% as the methanol ratio increased to 1:5 from 1:1.
- The maximum FFA conversion of 87.4% was achieved at methanol to oil ratio of 1:3

Standard parameters of biodiesel

- Saponification value - less than 312 (mgKOH/g)
- Acid value - less than 0.8 (mgKOH/g).

CONCLUSIONS

- Alkali catalyzed transesterification process of waste cooking oil with methanol is used to convert biodiesel from waste cooking oil.
- The Waste Cooking Oil was successfully converted into biodiesel production
- Yield of biodiesel is tested for methanol to oil ratios from 1:1 to 1:5 ratios.
- Highest yield is obtained at 1:3 methanol to oil ratio. Acid values, saponification values are calculated and compared.
- FFA conversion % is studied for all samples with different methanol to oil ratio.

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THANK YOU...