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Conventional and Intensified Transesterification Process of Biodiesel Production: A Review

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Abstract*— The dependency on fossil fuels is growing day by day with the need of drastic energy demand. Due to concern over diminution of fossil fuels, many researchers have focused on the renewable and unconventional sources of energy in the last decades. Alternative Fuels for transportation are one of those areas of research. In this paper, a concise review of various biodiesel production methods have been presented. First of all, conventional method is presented based on the transesterification reaction and later, a few novel techniques such as microwave irradiation aided, solar energy assisted, ultrasonic cavitation etc. have been depicted and the later part of this paper is an effort to discuss the use of solar energy in the biodiesel production to make it a feasible option for future research in the field of biodiesel production on a larger scale in a cost-effective manner.

Keywords: Biodiesel, Catalyst, Microwave Irradiation, Molar Ratio, Solar irradiation, Transesterification

I. INTRODUCTION

In the recent times, due to increasing concern over scarcity of conventional fuels, many researchers have focused on the development of the biofuels for various application. Biofuels are the biological combustibles commonly derived from biomass. The primary usage of biofuels are related to the applications of electricity generation, transportation, industrial process heat etc. If we talk about the feedstock for biodiesel, both edible and non-edible crops/seeds can be used and oil are extracted through the seeds by oil extraction process. Some of the previous researches depicts that producing biodiesel using edible crops is not a viable solution because it leads to the insecurity of the foods and the food prize also changes frequently [1-3]. So, non-edible oils may be a good source of producing biodiesel. Table 1 represents the various feedstock for biodiesel production.

Table 1. Feedstock for biodiesel

Edible Oils	Non-edible oils	Animal Fats	Other resources
Soybeans	Almond	Lard	Bacteria
Rapeseed	Babassu	Fish Oil	Algae
Canola	Brassica carinata	Tallow	Fungi
Safflower	Camelina	Poultry Fat	Micro algae
Barely	Cynara		Tarpenes
Coconut	Jatropha Curcus		Latexes
Copra or Khopra	Jatropha nana		Cooking oil (yellow grease)
Cotton Seed	Jojoba oil		Microalgae (Chlorella vulgaris)
Ground nut	Pongamiaglabra		
Oat	Laurel		
Rice	Lesquerella		
Sorghum	Mahua		
Wheat	Palm		
Winter rapeseed oil	Karanja		
	Rice bran		
	Sesame		

At present, biodiesel may not completely replace petro-fuels, still various merits of biodiesel enable it a viable solution. Biodiesel is an unconventional fuels which is biodegradable, causes less polluted exhaust gases. The content of sulphur and aromatic compounds are less in case of biodiesel and the Cetane number is more. Moreover, the environmental concern also emphasize the usage of biodiesel as it has less emission of pollution compounds such as unburnt hydrocarbon, carbon monoxide etc. [4] So, the degradation of environment and petro-fuel crisis can be provided a viable solution by means of biodiesel production. This paper is mainly about the biodiesel production process i.e. Transesterification – a chemical reaction in which a triglyceride (oil) reacts with the alcohol in the presence of a catalytic agent, alkyl ester and glycerol produces after the chemical reaction and this alkyl ester is known as the crude biodiesel which is further purified to test it into engine. Transesterification is a reversible process and methanol and ethanol are generally used as the alcohols in this methods [5]. The transesterification process is exemplified in the figure 1.

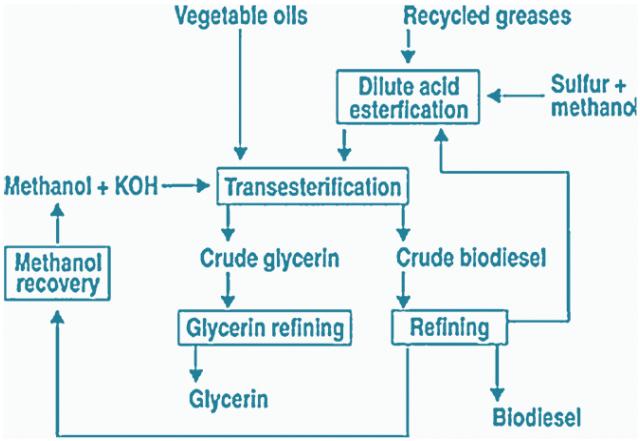

Fig. 1. Illustration of Transesterification process

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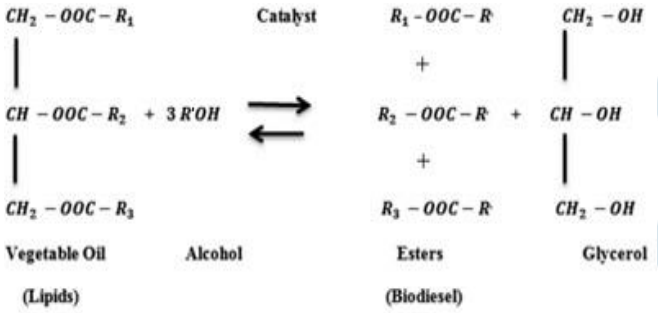
This paper presents an overview of conventional transesterification process for biodiesel production. However, recent researches are focused to intensify the transesterification process by various innovative techniques such as microwave irradiations, ultrasonic cavitation, solar energy assisted methods etc. These methods are also covered in this manuscript.

II. OVERVIEW OF CONVENTIONAL TRANSESTERIFICATION PROCESS

The chemical reaction which takes place in the transesterification process is shown below. In this reaction edible or non-edible oils which are extracted from the seeds, chemically are the forms of carboxylic acid which reacts with the alcohol to produce Esters form and Glycerol. Catalysts taking part in the reaction are just accelerate the reaction but do not chemically react with the reactants.



Previously, many researchers have worked on this basic transesterification process and the transesterification process can be categorized as follow [6]:

A. Base catalyst transesterification:

In this type of transesterification, free fatty acid content of the feedstock oil less than 1.0 wt%. KOH and NaOH are most commonly used as catalyst in the homogeneous base transesterification process. This transesterification is the easiest method includes least steps in biodiesel formation process. The biodiesel formation is enhanced with the increase in the reaction temperature. However, the reaction temperature should be sufficiently less than the boiling point of methanol (alcohol). The excessive increase in temperature for higher reaction time may cause saponification. Base catalytic transesterification is not a better option when feedstock oil with higher value of FFA content is used. Also, water used for purification can cause more soap formation after decomposing triglycerides into FFA and diglycerides or glycerides. Previous research depicts that KOH is better option as base catalyst when compared to NaOH, as it results in faster reaction and less soap formation.

B. Acid catalyst transesterification

In this type of transesterification, homogenous acid catalyst such as HCl & H_2SO_4 are used and chemical reaction takes place between feedstock oil and alcohol to form glycerol and fatty acid alkyl esters. This type of transesterification is not popular in the industrial applications as it cause corrosion to the surface of metallic vessel also it is a comparatively slower process and supports formation of metal oxide. The deployment of acid catalytic agent involves more steps of neutralization and purification. However, acid catalyst based transesterification is a viable option in case when the feedstock oil has higher free fatty acid content (more than 1.0 wt%).

Jatropha is one of the most popular feedstock oil, Berchmans et al. described the biodiesel production process from crude jatrophacurcas seed oil which is an oil with higher free fatty acid (FFA). The yield of biodiesel produced might be decreased due to Alkali base catalyst, so, acid pretreatment is done prior to reaction with base catalyst. In this work, the authors reported 90% yield for biodiesel i.e. methyl ester form of the jatropha oil [7].

C. Heterogeneous catalyst transesterification

In this process, the chemical reaction takes place between the feedstock oil and an alcohol to form glycerol and biodiesel in the presence of a heterogeneous catalyst i.e. oxides of base reinforced on a large surface area (CaO, TiO_2 and MgO etc.), Calcium oxide (CaO) is the superior heterogeneous catalyst as it remains non-consumed in the chemical reaction and it can withstand severe conditions. This process has advantage of no soap formation but a co-solvent should be added to make certain mass transfer between reactants.

D. Supercritical methanol transesterification

In this type of transesterification process, the catalyst is not used in the chemical reaction, biodiesel is produced by means of reaction between feedstock oil (triglyceride) and alcohol at supercritical condition i.e. higher pressure and temperature. The yield of biodiesel formed is high in this case and there is no formation of soap. This process is insensitive to free fatty acids and water, so pretreatment is not required in this case. However, this process is not economically good and operating conditions are also severe.

E. Enzymatic transesterification

In case of enzymatic transesterification, lipases enzymes are utilized as catalyst in the biodiesel formation. This process is environmental friendly process amongst all types of transesterification. In this process, the reaction temperature is kept low around 40°C and there is no formation of glycerol. The process is insensitive to water and FFA content of oil, so the yield of biodiesel formed is greater in this process. Slow rate of reaction and high cost are the demerits of this process.

The ease of producing biodiesel by means of transesterification make it one of the effective and popular method. However, conventional transesterification requires a lot of pretreatment and post-treatment processes.

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Pretreatment process includes esterification with acid catalyst (H_2SO_4) in case of feedstock with FFA more than 2.5 wt%. Post-treatment process includes the purification and quality control of the crude biodiesel and the process flow chart is illustrated in the figure 2. The advantages and disadvantages of various types of transesterification process is tabulated in the table 2.

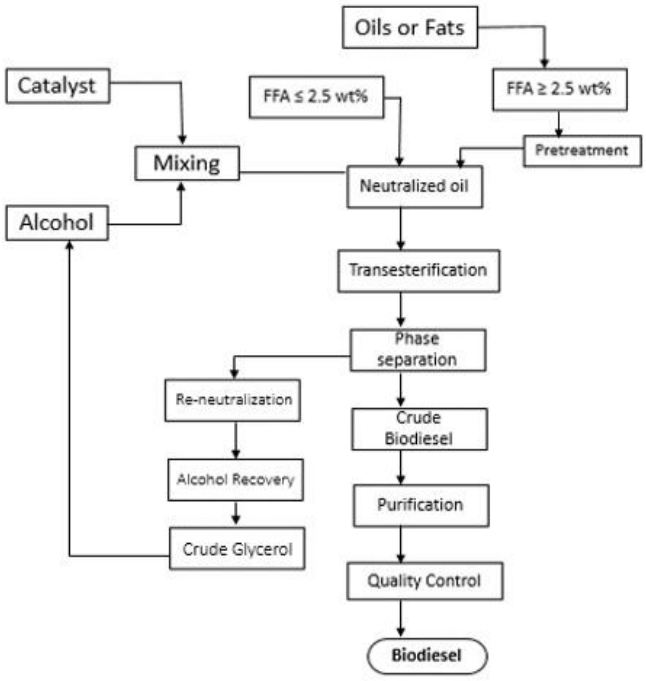

Fig. 2. Flow chart of Conventional Transesterification

Table 2. Comparison of different catalyst based Transesterification process [6]

Type	Example	Advantages	Disadvantages
Alkali Homogeneous	NaOH, KOH	High catalytic activity, low cost, favorable kinetics, modest operation conditions	Low FFA requirement, anhydrous conditions, saponification, emulsion formation, more waste water from purification, disposable
Alkali Heterogeneous	CaO, $CaTiO_3$, $CaZrO_3$, CaO–CeO_2, $CaMnO_3$, $Ca_2Fe_2O_5$, KOH/Al_2O_3, KOH/NaY, Al_2O_3/KI, ETS-10 zeolite, alumina/silica supported K_2CO_3	Noncorrosive, environmentally benign, recyclable, fewer disposal problems, easily separation, higher selectivity, longer catalyst lifetimes	Low FFA requirement, anhydrous conditions, more wastewater from purification, high molar ratio of alcohol to oil requirement, high reaction temperature and pressure, diffusion limitations, high cost
Acid Homogeneous	Concentrated sulphuric acid	Catalyze esterification and transesterification simultaneously, avoid soap formation	Equipment corrosion, more waste from neutralization, difficult to recycle, higher reaction temperature, long reaction times, weak catalytic activity
Acid Heterogeneous	carbon-based solid acid catalyst, carbohydrate-derived catalyst, Vanadyl phosphate, niobic acid, sulphated zirconia, Amberlyst-15, Nafion-NR50	Catalyze esterification and transesterification simultaneously, recyclable, eco-friendly	Low acid site concentrations, low Micro-porosity, diffusion limitations, high cost
Enzymes	Candida antarctica fraction B lipase, Rhizomucor mieher lipase	Avoid soap formation, nonpolluting, easier purification	Expensive, denaturation

III. TECHNIQUES TO INTENSIFY THE TRANSESTERIFICATION PROCESS

Microwave Irradiation based transesterification process

Chen et al. presented the process of biodiesel production from waste cooking oil along with a comparative analysis of convention transesterification and microwave assisted transesterification. The process in the microwave assisted biodiesel production is much faster in comparison to conventional process. Microwave assisted method was able to produce a maximum of 97.9% yield of biodiesel at 750 W microwave power, 6:1 methanol to oil ratio, 0.75 % by weight catalyst (CH_3ONa) and reaction time of 3 minutes. However, the conventional method could produce a maximum yield of 96.6% [8]. Azcan and Danisman conduction similar kind of comparative analysis for biodiesel production from cottonseed oil. The catalyst used in this work was KOH and methanol was used as alcohol. In this work, the conversion yield was attained in the range of 89.30% – 92.80% at reaction temperature around 60°C, KOH concentration 1.5 wt% for both type of process. However, the reaction time was only 7 minutes in microwave irradiation assisted technique compared to 30 minutes in conventional method [9].

Yaakob et al. conduced the experiments for biodiesel formation from jatrophacurcas oil using a microwave oven for the required heating. The maximum yield of biodiesel produced was reported 86.3% at 30:1 methanol to oil ratio, reaction temperature 55˚C and 4 wt% NaOH as catalyst. The reaction time was drastically decreased for microwave assisted method [10].

Kamath et al. discussed the biodiesel formation from feedstock oil with high FFA content i.e. Pongamiapinnata which is a non-edible oil. A double-step method was used to produce biodiesel from Pongamiapinnata using batch microwave irradiation technique and around 90% biodiesel was produced at process parameters – 10:1 alcohol to oil molar ratio and 1 wt% KOH [11].

Sherbiny et al. deliberated the microwave irradiation method to produce biodiesel from jatropha oil. In this experiment, same optimum reaction conditions to the microwave irradiation technique as in the case of conventional method. The maximum yield was reported 97.4% at reaction temperature 60˚C, molar ratio 7.5:1, 1.5 wt% KOH and reaction time of 2 minutes [12].

Kanitkar et al. discussed a batch microwave heating system for transesterification process using rice bran oil and soybean oil as feedstock. The authors compared the yield for different alcohol i.e. ethanol and methanol. The results indicated that the reaction time is reduced in case of microwave heating. It was discussed that methanol as an alcohol in the biodiesel production is better in terms of performance and effectiveness [13].

Hsiao et al. deliberated the microwave-assisted transesterification using soybean oil as feedstock and Nano

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powder CaO (Calcium Oxide) as catalyst. The experimental results depicted that a qualitative yield of biodiesel can be gained 96.6% approximately with the use of Nano-CaO in microwave assisted transesterification method [14].

Priambodo et al. used a Panasonic NN-SF550 (Power: 1000W) microwave reactor system to conduct intensified transesterification process from waste cooking oil. In this work, a heterogeneous base catalyst SrO was used and reaction time was varied from 40 second to 180 seconds. The methanol to oil molar ratio was maintained at 6:1 and reaction temperature at 80˚C. The supreme yield of biodiesel was found 93% yield at SrO concentration 1.85 wt% and reaction time of 3 minutes [15].

Gude et al. also discussed the intensified transesterification process using the microwave irradiation technique. In this work, following merits were observed by the authors [16]:

- Energy consumption was less.
- The reaction time was substantially reduced
- Less amount of solvent required
- The by-product formation reduced which resulted into more yield of biodiesel
- The effectiveness and quality of biodiesel produced microwave irradiation assisted method increases

So, it can be said that microwave irradiation assisted technique of biodiesel production can be better option for biodiesel production on a commercial level. The schematic diagram for the process of microwave assisted method is illustrated in figure 3, in which two reservoirs of oil and methanol are used to supply these to microwave over by means of pumps, transesterification take place in the microwave oven and products of the results are extracted in the product reservoir. The two layers appear in the product reservoir, above is the crude biodiesel and the other is glycerol which is separated from crude biodiesel. Crude biodiesel is the purified to remove moisture through various methods like water washing etc.

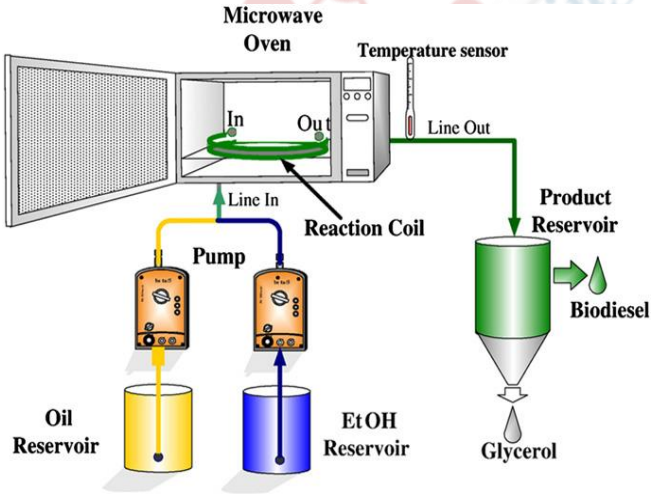

Fig. 3. Schematic of Microwave irradiation based transesterification

A few work related to microwave assisted transesterification are tabulated in the table 3.

Table 3. Summary of work related to Microwave assisted transesterification

Feedstock oil	Process Parameters	Microwave Reactor System	Outcomes	References
Waste Cooking Oil	Catalyst used: CH_3ONa Methanol to Oil ratio: 3–15:1 Stirring speed: 600 rpm Time: 1–6 min	Microwave Synthesis Reactor (Power: 200–750 W)	97.9% yield of biodiesel at 750 W microwave power, 6:1 methanol to oil ratio, 0.75 wt% catalyst, Reaction time 3 minutes.	Chen et al. [8]
Waste Frying Oil	$NaOCH_3$- 0.5 – 1.5 wt % Time: 3–9 min Temp: 55–65 °C	Start S model microwave	90.04–98.85% yield	Azcan and Yilmaz [17]
Waste cooking oil	Time: 40–180 s Temp: 80 °C Methanol to Oil ratio: 4–10:1 SrO: 1.4–3.25 wt %	Panasonic NN-SF550 Power: 1000 W	93% yield at M:O- 6:1, SrO:1.85 wt % Time: 3 min	Priambodo et al. [15]
Waste cooking oil	Batch:30 g Box-Behnken model was used	Modified Domestic Microwave	Optimum power: 325 W Time: 200 s Temp:70 °C KOH wt %-1 g/g 97% yield	Thirugnanasam bandham et al. [18]
Waste cooking oil	Esterification Oil to methanol ratio: 0.1 – 1 Time: 10–60 s Power: 100–400 W Transesterification M:O-5:1 NaOH: 2 wt % Temp.: 65 °C	Central composite rotatable design was used for DOE	Esterification Methanol to Oil ratio: 19.57:1 Time: 35 s Power: 250 W FFA reduced: 0.082% Transesterification Yield: 94.6%	Supraja et al. [19]
Waste cooking oil	KOH wt %: 0.5–1.5% Methanol to Oil ratio: 3–9:1 Time: 30–90 s Temp.: 65–85 °C	Domestic microwave oven	95% conversion KOH wt %: 1% Methanol to Oil ratio: 6:1 Temp. 75 °C Time:60 s	Selvaraj et al. [20]

Ultrasonic assisted production of biodiesel

Pal et al. conferred the biodiesel formation from waste cooking oil using power ultrasound and hydrodynamic cavitation technology. This technology augmented the quality of the biodiesel production from waste cooking oil. The maximum conversion yield was reported above 90% at process parameters – methanol to oil molar ratio 4.5:1 and catalyst concentration 0.5 wt%. The reaction time was also adequately decreased [21].

Koberg et al. described the biodiesel production from microalgae biomass of Nannochloropsis. In this work, SrO was used as catalyst and the comparative analysis was done between microwave and ultrasonic irradiation technique. The result displayed that microwave oven related technique is more efficient than the ultrasonic assisted technique. In reaction time of 5 minutes, the conversion yield was reported 37.1% for microwave oven technique and only 20.9% for ultrasonic technique [22].

Lin et al. offered ultrasonic energy assisted biodiesel formation from waste frying oil. Due to high FFA content of the feedstock oil, the acidic homogeneous catalyst H_2SO_4 was used and its concentration was varied from 1 % to 3 5 by weight. Alcohol to oil molar ratio was varied from 6:1 - 11:1. Further, the transesterification took place for triglycerides in waste frying oil having FFA content less than 2% by weight

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in presence of alkali-base catalyst NaOH. (acid value < 2.0) were transesterified with oil to alcohol molar ratio of 6:1. The alkali-base catalyst NaOH was used (1.0 wt%) in this second step. The yield of fatty acid methyl esters (FAME) i.e. biodiesel produced can be augmented using the ultrasonic mixing. Around 97.1% yield of biodiesel was attained in this process [23].

The schematic diagram of the ultrasonic assisted process is illustrated in the figure 4.

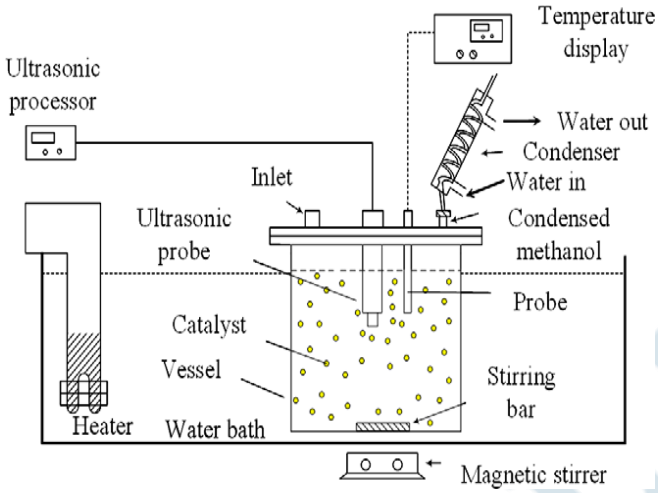

Fig. 4. Illustration of ultrasonic assisted biodiesel production

Solar Energy based Transesterification

Widayat et al. proposed a method of biodiesel formation from waste cooking oil using solar energy. In this method, a Fresnel lens was used to provide heat required for transesterification process. The authors compared the biodiesel yield at same reaction parameters. The Fresnel lens assisted method was able to produce maximum yield of biodiesel 99.18% at reaction time of 5 minutes, reaction temperature about 60°C, catalyst concentration 2.5 wt%, at the same conditions, the conventional transesterification was able to produce only 21.3% of biodiesel. The reaction time need to be increased up to 70 minutes with temperature maintained at 60°C to gain yield up to 97% in the conventional transesterification. Thus, it can be demonstrated that the assistance of solar energy is a viable option in intensifying the transesterification process [24].

Agee et al. has proposed the transesterification assisted by solar thermal energy. For this purpose, a large satellite dish (81 × 55 cm^2) with a reflective coating over it, was used as solar heat source and the chemical reactions of transesterification process were performed on the medium and small scale. In this work, the soybean oil was used and both methanol and ethanol were used as alcohol. This setup could be operated properly at solar intensity more than 400 W/m^2 and could produce around 2.5 liters of biodiesel in the reaction time of around 60 minutes [25].

Authors in [26] discussed the sonochemical deposition method to propose an advanced carbon supported catalyst SrO/C. In this work, solar energy was utilized for the heat required in the reaction and three type of oils were used – waste cooking oil, Canola and Soybean. The SrO/C catalyst enhanced the process of biodiesel synthesis significantly in comparison to the biodiesel synthesis process with SrO catalyst. The results depicted that the yield of biodiesel from waste cooking oil was found 98.5% by weight in the reaction time around one hour, temperature 46°C, methanol to oil molar ratio 6:1 and 7.1 wt% of the SrO/C catalyst. The catalyst can be reused four times in the subsequent transesterification process without considerable decrease in the yield of biodiesel. Similarly, the catalytic activity was enhanced by 5-times with soybean oil and more than 4-times with canola oil as feedstock. In this way, the SrO/C catalyst proved to be an economically viable catalyst and incorporation of solar heat omitted the need of electrical energy supply to make this a viable technology.

Gupta et al. described a biodiesel synthesis transesterification method from hempseed oil in an energy efficient way. For this purpose, a Fresnel lens was used in place of conventional heater to provide the heat required and a comparison was done between conventional heating assisted and solar heating assisted transesterification. The results depicted that a maximum of 97.37% yield of biodiesel can be gained in case of transesterification assisted by solar heat via Fresnel lens concentrator. The reaction parameter were 4.5:1 alcohol to oil ratio, 60°C temperature, catalyst concentration 0.9 wt% and reaction time of 4 minutes. At the same parameters, the conventional heating transesterification could provide only 21.3% of biodiesel. To acquire around 97% yield of biodiesel, conventional heating assisted transesterification should be processed at 60°C temperature, 6:1 alcohol to oil ratio, catalyst concentration 1.05 wt% and speed of magnetic stirring 300 rpm for a duration of 70 minutes. The properties of biodiesel produced were as per the standards of American Chemical Society for the Materials (ASTM) D6751. The schematic diagram of the setup used in this work are shown in figure 4. [27]

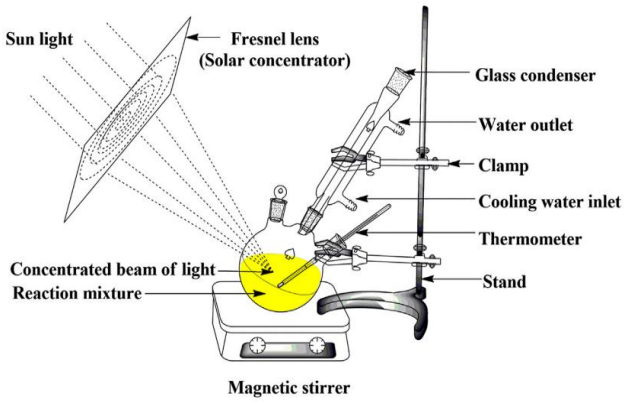

Fig. 5. Use of Fresnel lens in a solar assisted transesterification [27]

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León et al. discussed the economic viability of a solar assisted supercritical transesterification process to produce biodiesel from waste vegetable oil. The supercritical conditions omitted the need of catalyst due to which the steps in the purification of biodiesel were reduced. However, the need of higher quantity of alcohol and more energy to maintain high pressure and temperature in the process make it problematic in perspective of economic viability. The authors installed a trial plant in Mexicali, Baja California and it was demonstrated that the payback period for trail plant in less than five years with the internal rate of return of 31.7% [28]. So, the utilization of solar energy in the biodiesel production through transesterification promotes the application of renewable sources of energy and minimize the dependency on fossil fuels and electricity. This solar assisted transesterification is a clean, renewable and novel technique which can enhance the economic viability of the biodiesel production and implementation of this technique at large scale can results in numerous benefits in the field of alternative fuels in IC engine. The biodiesel produced from solar assisted transesterification method also have better properties when compared to biodiesel produced from convention method. Pal et al. presented the experimental work on the solar assisted synthesis of biodiesel from cottonseed oil in which the setup used (figure 6) was able to produce biodiesel in a short span of time and more than 90% yield of biodiesel was reported at alcohol to oil molar ratio equals to 6:1, reaction temperature 70°C, catalyst 1.0 wt%. The products of the transesterification reaction Cottonseed oil methyl ester (CSOME) and glycerol are shown in figure 7 [29]. The CSOME is separated from the glycerol.

Fig. 6. Solar assisted transesterification

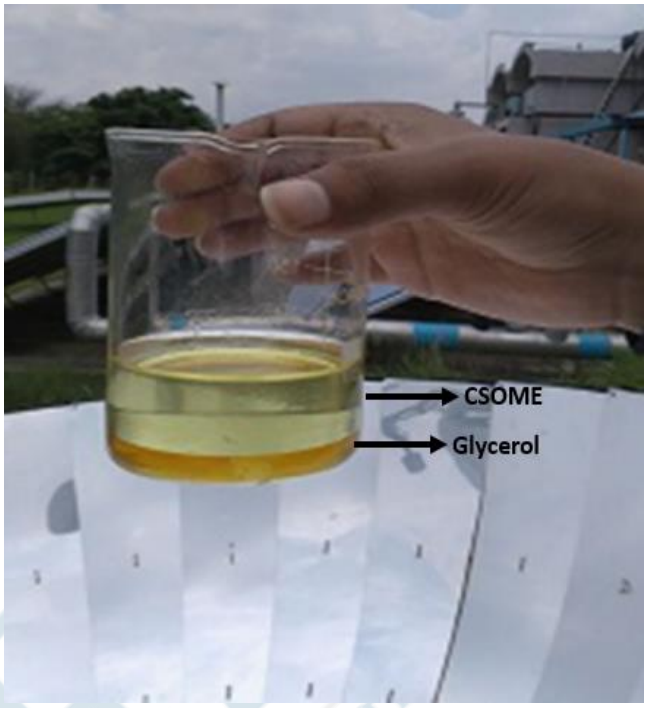

Fig. 7. Biodiesel and Glycerol produced

IV. GAPS IN THE LITERATURE REVIEW

The previous work related to transesterification for biodiesel production from various feedstock has been extensively done. The optimization of the biodiesel production has also been widely discussed in the previous research. Still there are some areas which need to be focused in the future research related to the biodiesel production:

i. The uncertainty analysis should be performed as it is an experimental work. The solar assisted method is the novel technique, so it should be validated using the uncertainty analysis.
ii. The current technique of the optimization must be implemented to augment the yield of biodiesel.
iii. The economic analysis should be performed to promote the commercialization of the biodiesel.
iv. Biodiesel formation method other than transesterification should also be discussed.
v. The problems related to feedstock supply and management should be elaborated.
vi. The effect of noise factors in case of intensified transesterification (in solar assisted or microwave assisted method), should be discuss to maximize yield.

V. CONCLUSION

In this paper, a comprehensive review of conventional and intensified transesterification process to produce biodiesel, have been presented. Conventional transesterification was classified as per the type of catalyst used in the process. A few novel techniques such as microwave irradiation aided,

solar energy assisted, ultrasonic cavitation etc. have been depicted. The microwave irradiation assisted method augmented the quality and yield of biodiesel produced in comparison on conventional method and reaction time was also substantially reduced in this method. However, the cost of production may increase. The transesterification process using ultrasound cavitation techniques appeared to be effective and rapid in comparison of conventional transesterification. The conversion yield obtained for methyl ester was high which make this process viable for industries. The later part of this paper is an effort to discuss the use of solar energy in the biodiesel production to make it a feasible option for future research in the field of biodiesel production on a larger scale in a cost-effective manner. The heating required for the chemic reaction can be supplied by means of solar collector in place of electrical heater.

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