

**MAJOR PROJECT-II**

**SOLAR ASSISTED BIODIESEL PRODUCTION FROM SESAME  
OIL**

Submitted in partial fulfilment of the requirement for the award of degree of

**Master of Technology**

In

**Renewable Energy Technology**

Submitted by

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## **DECLARATION**

I, hereby declare that the work which is being presented in this dissertation, titled “**SOLAR ASSISTED BIODIESEL PRODUCTION FROM SESAME OIL**” towards the partial fulfilment of the requirements for the award of degree of **Master of Technology** with specialization in **RENEWABLE ENERGY AND TECHNOLOGY** from Delhi Technological University Delhi, is an authentic record of my own work carried out under the supervision of **Dr. R.S Mishra**, Professor and Head of Mechanical Engineering Department and **Dr. Amit Pal**, Associate Professor, Mechanical Engineering Department, Delhi Technological University, Delhi.

The matter embodied in this dissertation record has not been submitted by me for the award of any other degree.

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## **CERTIFICATE**

This is to certify that the work embodied in the dissertation entitled “**SOLAR ASSISTED BIODIESEL PRODUCTION FROM SESAME OIL**” by TULIKA SRIVASTAVA (2K15/RET/16) in partial fulfilment for the award of degree of Master of Technology in Renewable Energy And Technology, is an authentic record of student’s own work carried out under my guidance and supervision.

It is also certified that the report has not been submitted to any other institute/university for the award of any degree.

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## **ABSTRACT**

The depletion of exhaustible resources such as fossil fuels, alternative energy sources are more focussed now-as-days. Biodiesel, being a source of clean energy can be a good alternative to prevent the depletion of petro-fuels as it is produced from edible and non-edible oils which renewable and non-toxic in nature. Transesterification is the only process for synthesis of biodiesel. This process involves, heating of triglyceride with methanol in the presence of catalyst like KOH. In this project, biodiesel was produced from sesame oil by using solar technology.

The conversion yield was maximized using Response Surface Method and Taguchi's method of optimization. This method was adequate for calculating the effect of control parameters as well as it optimizes the experiment with finite number of runs. Comparison and cost analysis by various methods including parabolic dish type collector, box type solar cooker and mechanical method for making biodiesel is done in this project.

At the end, results were compared for mechanical method as well as solar assisted method of biodiesel production. It was concluded that the quality and conversion yield of biodiesel produced from solar assisted method is better even when there is no requirement of mechanical stirring in this method.

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# CHAPTER – 1

## INTRODUCTION

As population is increasing the demand of energy is also increasing at a rapid rate. Our exhaustible resources such as fossil fuels, are rigorously used to meet the energy demands as well as human comfort. As a result, pollution level as well as depletion of our exhaustible resource are increasing at an alarming rate. Renewable fuel like biodiesel is easy to produce domestically as they are manufactured from vegetable oils, animal fats, or from various seeds like Jatropha, karjana, sesame, cotton seed etc. Because of cleaner burning property, biodiesel is unlike to petroleum & diesel, but it is a cleaner-burning alternative. Therefore its world's demand to opt for alternate fuel source and that too cleaner fuel and biofuel can be a good replacement. Diesel fuel is base of the economy of any country as it is used to generate power in various fields. For instance- in transportation, railroad, agricultural works and construction equipment etc. Fossil fuels contains different types of hydrocarbons such as toluene sulfur and aromatics rings etc. In recent times, due to combustion of petrol and diesel an alarming increase in pollution levels has been observed as result of which various protocols are signed. In addition, prices of these diminishing fuels are triggering.

Non-renewable fuel like petroleum can be replaced safely with biodiesel. Due to its high-lubrication and clean-burning properties, it can be used as an expendable fuel and is suitable for use in existing, unmodified diesel engines. This indicates that little quantity of diesel is requires to blend with biodiesel in diesel powered combustion engine. It is the only fuel which provides this much convenience. Biodiesel is biodegradable as well as safer for our environment unlike petro fuels. Biodiesel is designed in such a compatible manner that it can be blended with petroleum diesel in any ratio, from additive levels to 100 percent biodiesel. Currently, biodiesel is produced via transesterification reactions catalyzed by chemical catalysts, which produces higher fatty acid alkyl esters in shorter reaction time.

## **1.1 Energy Scenario in India:-**

India reckons greatly on conventional energy sources to meet its power requirements, due to abundance of coal it is kept at the forefront and the scenario will continue if we don't halt ourselves right now. In the forthcoming years, to be part of the fastest growing economies, an ambitious push for renewable energy is very essential. For the time being, quick steps become crucial to limit ecological damage at various levels such as production, transmission and consumption <sup>[1]</sup>.

For the growth of economy and welfare of the countries, power is the most critical component. Power sector of India is most broadened in the world. Power generation source ranging from conventional sources such as natural gas, hydro, coal and nuclear to renewable sources such as wind, solar, geothermal, agricultural and domestic waste. The call for electricity in our country is increasing at rapid rate and expectations are much higher to rise further in forthcoming years. So to meet the rising demand for electricity, huge generating capacity is needed.

India ranks third among 40 countries in EY's Renewable Energy Country Attractiveness Index, by the support of the government on promoting renewable energy resources and executing various projects in stipulated time [2].

To shift India towards clean energy will take time as well as money since it will require advanced technology and expertise. For cleaner production, Indian government has taken few steps. By 2016-2017, the goal of our government is to shift to new generation units, known as 'supercritical' that will reduce pollutant emissions by 25-30% but will also increase the cost by 30%. One can shift to even more eco-friendly 'ultrasupercritical' facilities is a challenging target to design, as declared by Integrated Research and Action for Development (IRADE).

Installed Capacity as on	Thermal (MW)				Nuclear (MW)	Renewable (MW)			Total (MW)	% Growth (on yearly basis)
	Coal	Gas	Diesel	Sub-Total Thermal		Hydro	Other Renewable	Sub-Total Renewable		
31-Dec-1947	756	-	98	854	-	508	-	508	1,362	-
31-Dec-1950	1,004	-	149	1,153	-	560	-	560	1,713	8.59%
31-Mar-1956	1,597	-	228	1,825	-	1,061	-	1,061	2,886	13.04%
31-Mar-1961	2,436	-	300	2,736	-	1,917	-	1,917	4,653	12.25%
31-Mar-1966	4,417	137	352	4,903	-	4,124	-	4,124	9,027	18.80%
31-Mar-1974	8,652	165	241	9,058	640	6,966	-	6,966	16,664	10.58%
31-Mar-1979	14,875	168	164	15,207	640	10,833	-	10,833	26,680	12.02%
31-Mar-1985	26,311	542	177	27,030	1,095	14,460	-	14,460	42,585	9.94%
31-Mar-1990	41,236	2,343	165	43,764	1,565	18,307	-	18,307	63,636	9.89%
31-Mar-1997	54,154	6,562	294	61,010	2,225	21,658	902	22,560	85,795	4.94%
31-Mar-2002	62,131	11,163	1,135	74,429	2,720	26,269	1,628	27,897	105,046	4.49%
31-Mar-2007	71,121	13,692	1,202	86,015	3,900	34,654	7,760	42,414	132,329	5.19%
31-Mar-2012	112,022	18,381	1,200	131,603	4,780	38,990	24,503	63,493	199,877	9.00%
31 Mar 2017 <sup>[34]</sup>	192,163	25,329	838	218,330	6,780	44,478	57,260	108,518	326,841	10.31%

Fig. 1.1 India's installed capacity growth <sup>[3]</sup>

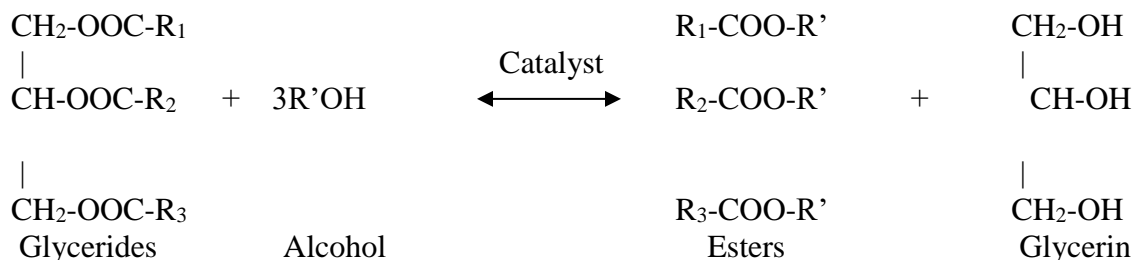
There are many technological solutions for reducing the levels of pollution but are expensive. For instance, to prevent CO<sub>2</sub> waste to go in the atmosphere a new infrastructure is required, this is known as carbon capture.

## 1.2 ABOUT BIODIESEL:-

Predominantly, the biodiesel is the alkyl ester of fatty acids, made by the transesterification of oils or fats, from plants or animals, with short chain alcohols such as methanol and ethanol. Glycerin is a by-product from biodiesel production [4]. It is defined as mono-alkyl esters of longer chain fatty acids contained in vegetable oils or animal fats. Biodiesel enhances the combustion efficiency and emission characteristics. Biodiesel is more preferred as an alternative fuel as it is biodegradable, renewable and non-toxic. It produces less particulate.

### 1.3 Reactions involved in biodiesel:-

In the process of transesterification, different types of oils containing triglycerides react with alcohol, generally methanol or ethanol in order to produce esters and glycerin. To make it possible, a catalyst is added to the reaction. In the initial step, triglycerides are reduced to di-glyceride and from di-glycerides mono-glyceride is produced. And in the last stage, glycerin is obtained from mono-glyceride. In all these reactions esters are produced.



The overall process involves three consecutive steps; these reactions are reversible in nature.

Sesame oil belongs to the category of edible vegetable oil. It is used as flavor enhancer in North Korea, China and Japan and as cooking oil in India. It has medicinal properties as well and very popular in Asia. It consists of various fatty acids. These are linoleic acid (41%), oleic acid (39%), palmitic acid (8%), stearic acid (5%) and others in small amount. It is drought resistant crop therefore was cultivated for more than 5000 years. In developed countries the oil is extracted using oil expeller or by compacting followed by chemical solvent extraction. Due to cold press the oil gets pale yellow. Cold pressed cause to give different flavor to the sesame oil. Under low temperature conditions, cold pressing method using an expeller press can be used to extract oil. In Ayurvedic medicine, sesame oil is good for massaging [5].

### 1.4 Biofuels need :-

Biofuels are organic fuels derived from biomass. The need for biofuels arrived as it is clear from energy scenario that our society is more dependent on fossil fuels. Biofuels are generally utilized for combustion in industries, heat engines and for generating

electricity. Presently, due to absence of engines running purely on biodiesel we can't rely totally on biodiesel as a primary fuel. As compared to diesel fuel, biodiesel has higher cetane number as well as combustion efficiency. Aromatic and Sulphur content are much lower in biodiesel that is symbol of producing non-toxic emissions. Moreover exhaust gases including monoxides, unburnt hydrocarbons, NOx emissions are reduced to great extent. So, environmental degradation problem can be avoided along with petro-fuel crisis.

### **1.5 Fuel Properties:-**

A fuel is virtually any substance which participates in self-reliant exothermic reaction with comparative easiness.

**Viscosity:-** is a physical property of the fluid and is defined as measure of the resistance of the fluid to flow. Under the action of gravity, time is recorded for the flow of a fixed quantity of fuel through standard orifice and that time helps in calculating the viscosity of the fuel.

**Volatility:-** can be defined as the tendency of a liquid to evaporate. The volatility of a fuel should be sufficient enough to give combustible homogeneous air-fuel ratio inside the engine cylinder. This ratio plays a vital role at the time of ignition operating under all conditions, including starting and warming up period of a cold engine. The standard practice for measuring the relative volatility of fuels is by means of ASTM distillation test.

**Ignition Quality:-** is the ability to auto-ignite inside the engine cylinder in presence of air under different operating conditions and is measured by its delay period. Self-ignition temperature of coil is a function of delay period. Good ignition quality means short delay angle.

Cetane number:- is defined by blending two pure hydrocarbon reference: Cetane( $C_{16}H_{34}$ )- a straight chain paraffin having high ignition quality and Isocetane having a very poor ignition quality. Higher the cetane number better is the fuel ignition quality. Antiknock characteristics of a fuel means requirement of longer delay period and that is specified by the higher value of the cetane number.

Cold starting:- CI engines faces problems in cold weather. To overcome this, higher cetane number fuel is proposed.

### **1.6 OBJECTIVES:-**

Presently, although many research and developments are taking place, but still people are facing the electricity deficit problem. Undoubtedly, biodiesel can be an alternative solution to petro-fuels, but the production of biodiesel is not very cost efficient that we can use it commercially in automobiles.

In conventional transesterification process, vegetable oil with methanol is heated under strong basic condition. Heat is provided by external means, in this case it was electrical heater. Our objective is to avoid the means of electricity produced by fossil fuel and instead of it use solar energy, which contains sufficient amount of heat. This solar energy can be utilized by either box type solar cooker as well as by concentrating solar radiations at a point as observed in concentrated solar cooker. Intensity of solar radiation should be greater than  $400\text{W}/\text{m}^2$  [6] . By utilizing this method, production of biodiesel can be more sustainable and cost efficient.

### **1.7 Problem Statement:-**

The concept of using solar energy for increasing the temperature wherever required is good but there is need to analyze the reflectors we are choosing for our experiment. Temperature control is very important factor as surplus temperature can cause evaporation of alcohol and lesser degree can result into bad quality of biodiesel.

Various parameters of solar reflector such as reflector area, inclination angle etc. are also responsible for producing the desired quality of biodiesel and determining the transesterification process of biodiesel. According to the production scale i.e. large or small, the design of reflectors are modified. Oil containing low FFA content should be used in order to avoid tiresome process of producing biodiesel.



## CHAPTER- 2

### LITERATURE REVIEW

To set the target of present research and to justify it properly, the available literatures have been critically reviewed. The literature review is presented in this chapter classifying them into two broad categories as follows:

1) Review pertaining to Biodiesel production techniques

Under this heading, the biodiesel production using the Mechanical stirring machine and solar assisted apparatus have been taken into consideration.

2) Review pertaining to cost involved in various techniques

Under this heading, the research that reported the testing of biodiesel derived from different sources has been covered.

#### **2.1 Traditional method of biodiesel production:-**

The most practiced method for biodiesel production is transesterification. Xuang Wu et al. presented a review on common vegetable oils or animal fats (also known as triglycerides) react with alcohol in the presence of a catalyst, a process known as transesterification. When triglycerides containing a high percentage of free fatty acids or water, the alkali catalyst will react with the free fatty acids to form soaps. The water can hydrolyze the triglycerides into di-glycerides and form more free fatty acids. Both of the above reactions are undesirable and reduce the yield of the biodiesel product. In addition, it binds with the catalyst that would require more catalyst and hence the process will involve a higher cost. Water, present in the oils and fats or formed during the saponification reaction, retards the transesterification reaction through the hydrolysis reaction. In those cases, the acidic materials should be pre-treated to restrict the saponification reaction. The main factors that affects the yield of biodiesel are:- alcohol quantity, reaction time, reaction temperature and catalyst concentration. Generally, methanol is preferred alcohol for producing biodiesel because of its low reactivity<sup>[7]</sup>.

Svitlana Niti\_ema-Yefanova et al., carried out the process of producing ethyl biodiesels from non-edible vegetable oils (NEVO) were determined with the ultimate objective of proposing an on-farm processing technology that should be sustainable for emerging countries Their conversion to fatty acid ethyl esters (FAEE) was conducted via a two-stage procedure under atmospheric pressure: an alkali-catalyzed ethanolysis at ambient temperature for the BA and AI oils (leading to 93 and 87 wt.% FAEE respectively) and an acid catalyzed ethanolysis at the normal boiling of the alcohol for the JC oil (leading to 89 wt.% FAEE). Based on the intermediate addition of glycerol at ambient temperature, the two-stage procedure combines chemical kinetics, chemical equilibrium, and phase equilibrium phenomena. Simple procedures with optimal operating conditions were determined for the alkali-catalyzed ethanolysis of BA and AI oils (leading to 93 and 87 wt.% FAEE respectively), and for the acid catalyzed ethanolysis of JC oil (leading to 89 wt.% FAEE). Both types of ethanolysis are based on a two-stage process using a single catalyst but an intermediate addition of glycerol at temperature to induce liquid-liquid phase splitting and thus, glycerol removal from the reacting phase. The conditions of ethanolysis have to be optimized depending on the nature of the feedstock, and this until theoretical models combining the chemical kinetic and thermodynamic aspects are developed to predict these conditions<sup>[8]</sup>.

Aadel Xaaldi Kalhor et al. , investigated on microalgae as a source of oil production. In recent years, waste water pollutions have caused many ecological problems, and therefore, waste water phycore mediation has attracted the international attention. This paper studied the cultivation of *Chlorella vulgaris* in a crude oil polluted environment for biodiesel production. Intended concentrations were 10 and 20 g per liter (crude oil/water) at two times. The results showed that the growth of *C. vulgaris* was improved in wastewater and the maximum amount of dry mass and oil was produced at the highest concentration of crude oil (0.41 g and 0.15 g/l, respectively). In addition, dry mass and oil yield of the microalga were significantly enhanced by increasing the experiment duration. The growth of the microalga was surprisingly improved in wastewater and a notable amount of dry mass and oil were produced at a high concentration of crude oil. Biodiesel yield was enhanced with increasing the duration

of the experiment ( $P < 0.05$ ), and it was decreased by increasing the concentration of crude oil. The potential function of *C. vulgaris* for biodiesel production in the crude oil polluted environment makes the species attractive for further extensive studies<sup>[9]</sup>.

Idris Atadashi Musa, observed the nature of alcohol and alcohol to oil molar ratio plays an important role on the method of biodiesel production. As a result, this paper examined different alcohols commonly used for the production of biodiesel fuel with more emphasis on methanol and ethanol. Further the different alcohol to oil molar ratios used for the production of biodiesel have been extensively discussed and reported. Also the effects of alcohol to molar ratios on biodiesel refining process and its physicochemical properties were investigated. It was found that quite a number of biodiesel production facilities employ methanol due to its low cost and short chain molar size (for the avoidance of steric hindrance effects). Although ethanol is more expensive than methanol but biodiesel production involving ethanol is completely bio-base, hence renewable. Most of the researchers recommend a 6:1 molar ratio for methanol and a 9:1 molar ratio for ethanol. It was found that the presence of alcohol affects the quality of biodiesel fuel by lowering its viscosity and density values, and flash point<sup>[10]</sup>.

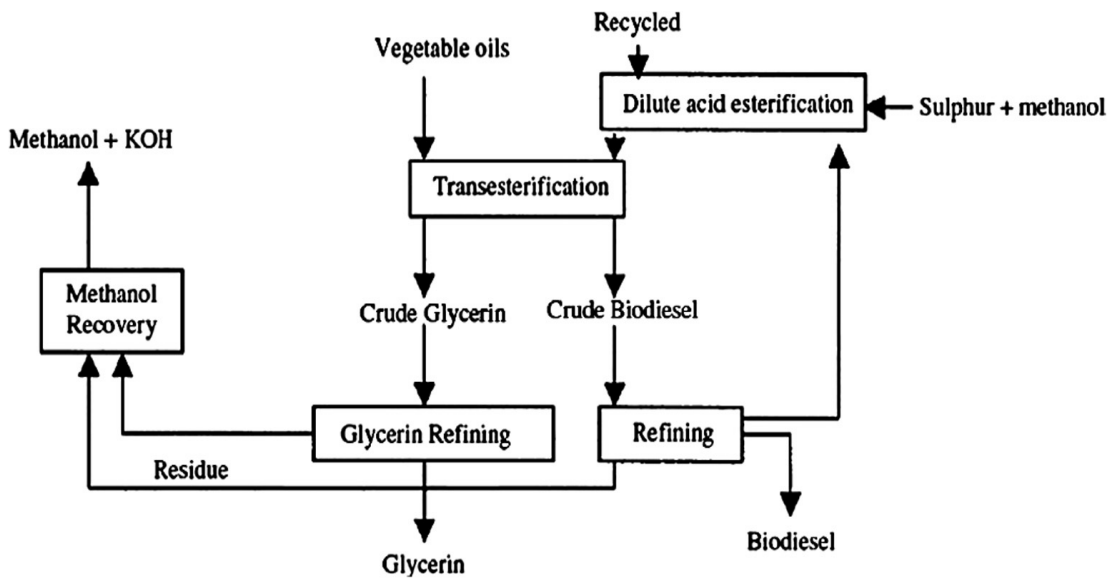


Fig. 2.1 Biodiesel Formation<sup>[11]</sup>

G. Baskar et al., studied the biodiesel was produced from castor oil using ferromagnetic zinc oxide nanocomposite as heterogeneous catalyst for transesterification reaction. Single phase of nanocatalyst were confirmed by X-Ray Diffraction analysis. The spherical shape of the aggregated nanocatalyst was observed in Scanning Electron Microscopy. Magnetic properties were analyzed using vibrating sample magnetometer. Atomic Force Microscopic analysis revealed the larger surface area and roughness of nanocatalyst. The biodiesel yield of 91% (w/w) was obtained in 50 min at 55<sup>0</sup>C with 14 wt % catalyst loading and 12:1 methanol/oil ratio and was confirmed by Gas chromatograph with Mass Spectrometer. The result showed that the iron (II) doped ZnO nanocatalyst is a promising catalyst for the production of biodiesel via heterogeneous catalytic transesterification under milder reaction conditions. Iron (II) doped Zinc oxide catalysts prepared by the co-precipitation method demonstrated an efficient catalytic activity for the transesterification of castor oil using methanol. All the parameters such as reaction time, catalyst loading, temperature, time and methanol to oil molar ratio gave a significant effect. The maximum biodiesel yield obtained from was 91% at optimum conditions: 50 min of reaction time, temperature of 55<sup>0</sup>C, 14 wt% of catalyst loading and methanol to oil molar ratio of 12:1. The first order tends to fit the kinetic model where the activation energy was found to be 1527.53 J/mol <sup>[12]</sup>.

J. Kakati et al., investigated biodiesel production from Amari tree seed oil (ATSO). Oil content in the seed was 42.85%. Linoleic (32.938%) and oleic acids (23.007%) were the major fatty acid constituents in ATSO with 84.617% unsaturation. The free fatty acid (FFA) in ATSO was 16%, hence, a two stage acid base transesterification was done to produce biodiesel from ATSO. Effect of reaction time, temperature, methanol and catalyst concentrations on yields from the 1st stage acid pre-treatment and 2nd stage base transesterification was evaluated. Maximum 96% yield (vol.%) from the 1st stage occurred with 0.80% (v/v) H<sub>2</sub>SO<sub>4</sub> at an oil methanol ratio of 4:1 after 3.5 h of acid esterification. From the 2nd stage, highest biodiesel yield of 88.5% (vol.%) was obtained during base catalysed transesterification by adding 1% (w/v) NaOH and 30% (v/v) methanol with the acid pre-treated oil after 2.5 h. Characterization of ATSO fatty acid methyl ester (FAME) was done and properties were compared with Amora and

Pithraj tree seed biodiesels from same Meliaceae family. Properties were found similar and most of them conformed to the standards except water and sulphur contents with slight deviations.

In this study, biodiesel was produced from ATSO using a two stage acid–base transesterification process. Experiments were done to evaluate the effect of reaction time, amounts of methanol, H<sub>2</sub>SO<sub>4</sub> and NaOH catalysts and reaction temperature on optimal yield of methyl ester. ATSO FAME properties were determined and compared with properties of conventional diesel and two other tree seed based biodiesels (Amoora and Pithraj) of same Meliaceae tree family. The following conclusions were drawn.

1. Amari fruit seed contained 42.85% oil with 16% FFA.
2. During 1st stage acid esterification, maximum 96% yield (vol.%) was obtained with 0.80% (v/v) H<sub>2</sub>SO<sub>4</sub> at 4:1 (v/v) oil methanol ratio after a reaction duration of 3.5 h.
3. Maximum 88.5% biodiesel yield (vol.%) was obtained during the 2nd stage base catalyzed transesterification. This was achieved by carrying out reaction of the 1st stage product with 1% (w/v) NaOH and 30% (v/v) methanol at 60<sup>0</sup>C continuously for 2.5 h.
4. Almost all properties of ATSO FAME were compatible with ASTM D6751 and EN14214 standards except water and Sulphur contents with slight deviations. The ester content (95%) was slightly lower than the minimum 96.5% prescribed in the EN14103 standard.

Hence, it is concluded that Amari tree seed could be a potential renewable source for biodiesel production in India <sup>[13]</sup>.

Grisel Corro et al. (2017), investigated production of biodiesel from waste frying oil (WFO) by a two-step process. In the first step, the free fatty acids (FFAs) present in the oil were esterified with methanol by a photocatalytic process using Cr/SiO<sub>2</sub> semiconductor composite and solar irradiation as light source. The catalytic performances of Cr/SiO<sub>2</sub> calcined at different temperatures (500–1000 °C) were investigated in FFAs photo-esterification process. The samples were characterized by

X-ray diffraction (XRD), X-ray photoelectron spectroscopy, diffuse reflectance spectroscopy (DRS), and nitrogen physisorption. Characterization results revealed the presence of  $\text{Cr}^{6+}$  in the samples calcined at temperatures between 500 and 600 °C. However, a mixture of  $\text{Cr}^{6+}$  and  $\text{Cr}^{3+}$  was detected in the samples calcined at higher temperatures (700–1000 °C). The catalysts calcined between 700 and 1000 °C showed a very high activity for FFAs photo-esterification. A mechanism is proposed to explain this high photoactivity, assuming a synergetic electronic interaction between  $\text{Cr}^{6+}$  and  $\text{Cr}^{3+}$  during photo-irradiation, which generates  $\text{H}^+$ ,  $\text{CH}_3\text{O}\cdot$  and  $\text{R}-\text{COOH}\cdot$  radicals in high concentration at the photocatalyst surface. The second step consisted in the triglycerides transesterification with methanol through thermal activation using solar irradiation as heating source and catalyzed by NaOH. Produced biodiesel by this process, fulfills all the international requirements for its utilization as a fuel.[14]

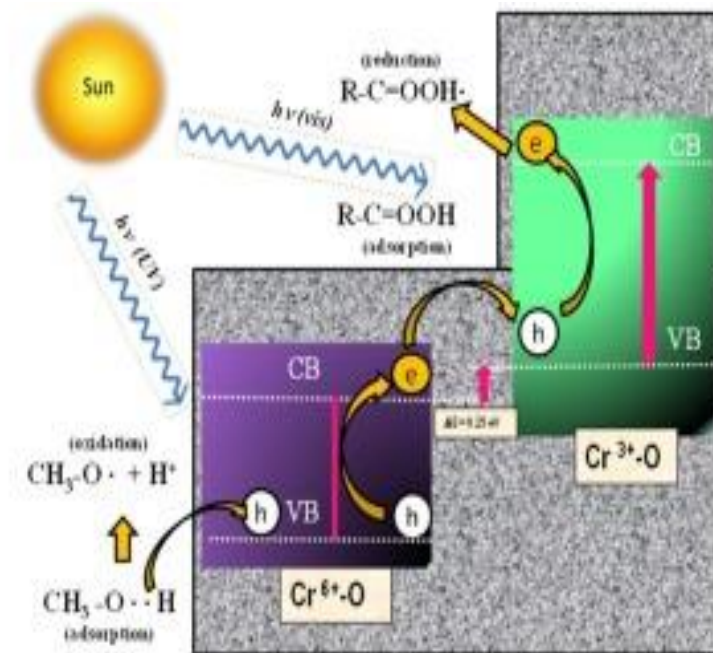


Fig. 2.2 Transesterification process carried in presence of solar radiation [15]

## 2.2 Ultrasonic method of biodiesel production:-

Gajendra Kumar et al. discussed the new technology of carrying out faster transesterification process for the producing biodiesel using an ultrasonic-assisted

continuous tank reactor in the presence of fatty acids used with potassium hydroxide used as a catalyst. This research provided a new biodiesel production process, the optimal results were obtained at: reaction temperature 25°C oil to methanol molar ratio was 1:5, catalyst concentration 0.75 wt% of oil, solvent concentration 7.5%, ultrasonic amplitude 60% and ultrasonic cycles 0.7 s, transesterification was completed within 1.09 min (residence time).

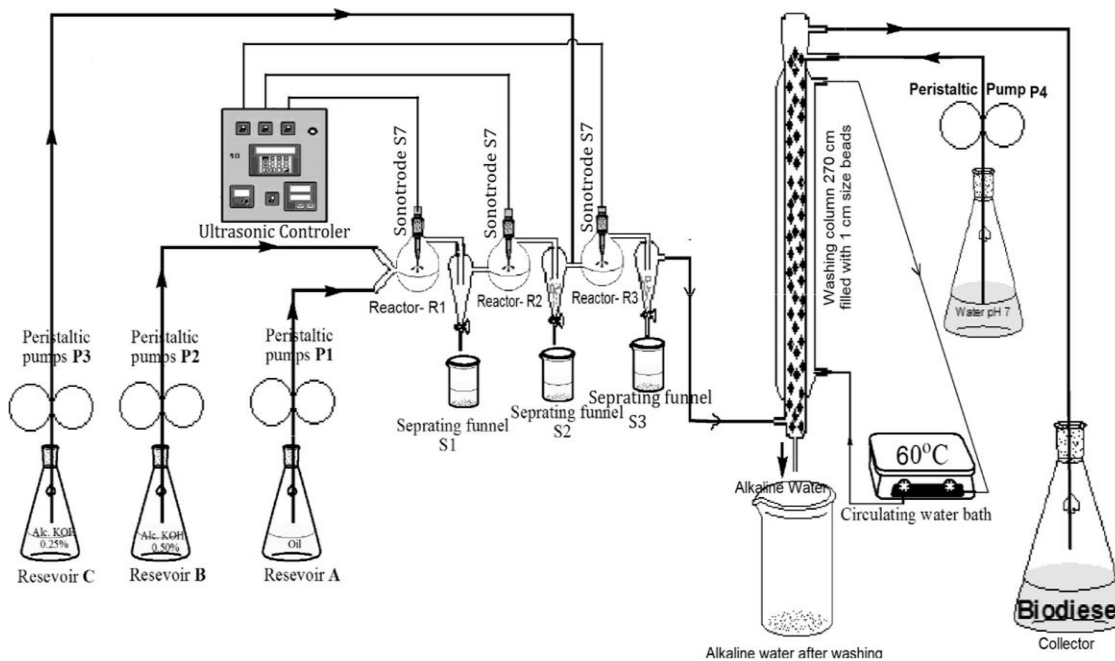


Fig. 2.3 Ultrasonic setup for producing biodiesel [16]

The maximum yield was found at the amplitude 60% and cycle 0.7 s ultrasonic; helped to escalate the rate of reaction because ultrasonic energy produced by the sonotrode were responsible for the ultrasonic cavitations and microbubble formation which excites the reactant molecule and greatly improve the interfacial contact between the methanol and vegetable oil.[17]

Dharmendra Kumar et al. (2014) worked for biodiesel production in which reduced reaction time and production cost was observed along with energy saving. In this

project, ultrasonic assisted transesterification of *Jatropha curcas* oil was carried out in the presence of methanol and potassium hydroxide (KOH) as catalyst. The molar ratio of oil to alcohol taken was 1:5, catalyst concentration 0.75 wt% of oil, ultrasonic amplitude 50% and pulse 0.3 cycle, 7 min reaction time under normal atmospheric conditions. The conclusion of this experiment came out that due to ultrasonic mixing the rate of transesterification reaction was increased as compared to the mechanical mixing. Experimental results proved that ultrasonic mixing method requires shorter reaction time for biodiesel formation as well as less energy consumption than the conventional mechanical stirring method. As a result ultrasonic irradiation effect can be a faster alternative to the usual process together with high product yield. This brings about considerable time saving as well as cost <sup>[18]</sup>.

Wilson Wei Sheng Ho et al. (2016) reviewed advancements in ultrasound-assisted biodiesel transesterification derived from various feedstocks utilizing acid, base and enzyme catalysts. With an intention to use ultrasound energy in conjunction with heterogeneous catalysts. Each of the technology was reviewed, the interactions between reaction parameters and process efficiency as well as productivity were analyzed and discussed. Ultrasonic cavitation have proven successful in accelerating the mixing rate of reactants with either homogeneous or heterogeneous catalysts leading to noticeable reductions in reaction time with comparable biodiesel conversions as conventional transesterifications. The various research studies carried out so far focuses on parametric and optimization investigations. Also catalyst leaching and reusability as well as biodiesel quality are on focus too. This offers an opportunity for future work in this field to advance the current level of knowledge and understanding. The current ultrasound-assisted biodiesel production technology status remains largely at bench-scale with a few notable pilot-scale demonstrations. To overcome the gap between this and large-scale commercialization, several areas worthy of future investigation include continuous ultrasonic reactor systems especially using heterogeneous catalysts, pilot-scale tests as well as techno-economic feasibility studies<sup>[19]</sup>.



Adewale Peter et al. (2015) have mentioned few techniques of preparing biodiesel that involves ultrasonic method and agitating the reaction by using microwave radiations. Ultrasound involves process of propagating an oscillating sound pressure wave with a frequency greater than the upper limit of the human hearing range. Application of ultrasound in the chemical processing enhances both mass transfer and chemical reactions, that will lead to shorter reaction times, and cheaper reagents. They concluded that methanolysis of triglycerides using ultrasonic irradiation was an efficient, time saving and economical method of producing biodiesel fuel. Microwave irradiation was employed for different applications that included organic synthesis where chemical reactions were accelerated because of selective absorption of microwave energy by polar molecules, nonpolar molecules being inert to the microwave dielectric loss <sup>[20]</sup>.

Miaomiao Wan et al.(2017) produced biodiesel from soyabean oil deodorizer distillate intensified by dual-frequency counter-current pulsed ultrasound. Results showed that the biodiesel conversions enhanced by single-frequency were lower as compared to those enhanced by dual-frequency. For dual-frequency, the biodiesel conversion of SMM was higher than those of SQM. The biodiesel conversion of the combination of 20/28 kHz is the highest. methanol to triglyceride molar ratio 8:1, catalyst content 1.8%, the water content in feedstock should be less than 0.4%, the acid value of feedstock should be less than 2 mgKOH-g<sup>-1</sup>, the biodiesel conversion could reach 96.3%. The effects of 20/28 kHz simultaneous mode (SMM) on biodiesel production were studied and 8:1 was the optimal methanol to triglyceride molar ratio, 1.8% was the optimal catalyst content, the water content in feedstock should be less than 0.4%, the acid value of feedstock should be less than 2 mgKOH-g<sup>-1</sup>, the biodiesel conversion could reach 96.3%.[21]

Hameed Motabdi et al. (2010), investigated ultrasonic-assisted transesterification of palm oil in the presence of alkaline earth metal oxide catalysts (CaO, SrO and BaO). Intensity of ultrasonic waves of 20kHz, cavitation was carried out to study the effect of reaction time (10–60 min), alcohol to palm oil molar ratio (3:1–15:1), catalysts loading

(0.5–3%) and varying of ultrasonic amplitudes (25–100%). The activities of the catalysts were according to their basic strength. 50% amplitude of ultrasonic irradiation was considered the most suitable value and physical changes on the catalysts after the ultrasonic-assisted reaction were successfully illuminated. Catalysts dissolution was found to be responsible for drop of the activity of the reused catalysts, especially with BaO catalyst. Ultrasonic cavitation might lead to a localized increase in temperature at the phase boundary, so control of reaction temperature in ultrasonic-assisted reactor is very critical issue. The reaction temperature in magnetic stirrer was set at 65<sup>0</sup>C. To be comparable, the temperature of the ultrasonic and non-ultrasonic reactions had to be the same. Thus, the water bath temperature was varied in order to reach a steady state temperature of 65<sup>0</sup>C in ultrasonic-assisted reactor.. In short, there was a significant decrease in the water bath temperature as the ultrasonic output power was increased<sup>[22]</sup>.

Mohd. RazealyAnuar et al. (2016), presented a paper on ultrasound-assisted biodiesel production from waste cooking oil catalyzed by hydrotalcite (HT) catalyst prepared using combustion method was studied. Two important parameters in the HT synthesis i.e., calcination temperature (550–850 °C) and fuel type (saccharose, glucose and fructose) were particularly investigated. The dependence of HT's characteristics on the synthesis parameters and correlations with their catalytic performance under ultrasound condition were successfully elucidated. The HT catalyst prepared using saccharose and calcined at 650 °C was the best catalyst to be used in the transesterification reaction. It showed high biodiesel yield (about 76.45%) in just 60 min in the presence of low ultrasound amplitude (~11 kHz). The enhancement effect of ultrasound was successfully demonstrated. The reaction only needed short reaction time (about 1 h) to give a biodiesel yield of up to 76.45% compared to conventional stirring method that needed about 5 h to achieve the same yield <sup>[23]</sup>.

Preeti B. Subhedar et al. (2016), investigated on synthesis of biodiesel from waste cooking oil via ultrasound assisted intensification using methyl acetate and immobilized lipase as a catalyst.

The reaction was investigated by conventional approach too, i.e. based on stirring in order to compare the beneficial effects obtained by the use of ultrasound. Effect of operating conditions such as reactant molar ratio (oil and methyl acetate), temperature and enzyme loading on the yield of biodiesel has been investigated. Optimum conditions for the conventional method was established as reactant molar ratio of 1:12 (oil:methyl acetate), enzyme loading of 6% (w/v), temperature of 40<sup>0</sup>C and reaction time of 24 h and under these conditions, 90.1% biodiesel yield was obtained. The optimum conditions for the ultrasound assisted approach were oil to methyl acetate molar ratio of 1:9, enzyme loading of 3% (w/v), and reaction time of 3 h and the biodiesel yield obtained under these conditions was 96.1%. Use of ultrasound resulted in significant reduction in the reaction time with higher yields and lower requirement of the enzyme loading. The obtained results have clearly established that ultrasound assisted inter-esterification was a fast and efficient approach for biodiesel production giving significant benefits, which can help in reducing the costs of production. Reusability studies for the enzyme were also performed but it was observed that reuse of the catalyst under the optimum experimental condition resulted in reduced enzyme activity and biodiesel yield<sup>[24]</sup>.

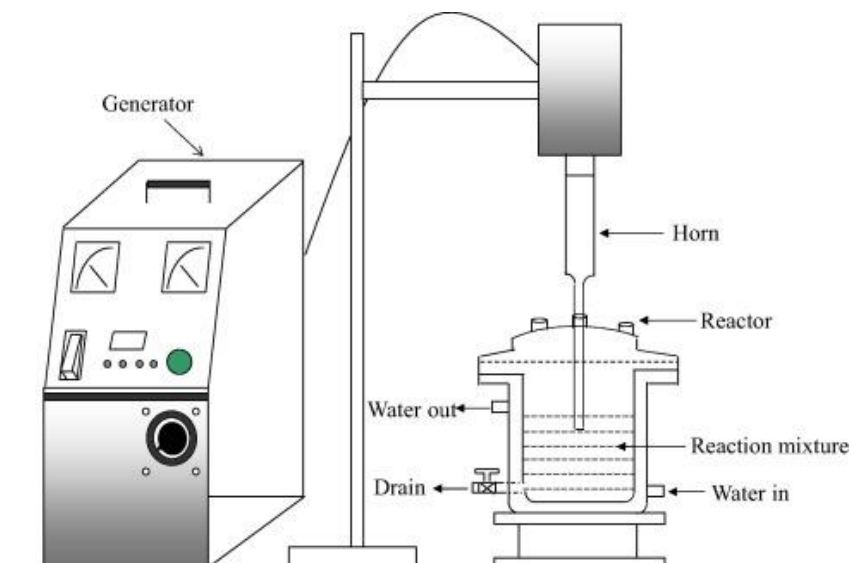


Fig. 2.4 Ultrasonic assisted intensified biodiesel production using enzymatic interesterification<sup>[25]</sup>

Mohammad Mahdi A. Shirazi et al. (2013) reviewed that while making biodiesel, slow separation of glycerol occurs; it could lead to longer operating times and require bigger equipment and can result into increased production cost. Therefore, acceleration of glycerol/biodiesel decantation can play vital role in the overall biodiesel refinery process. In this work, NaCl-assisted gravitational settling was considered as an economizing strategy. The results obtained indicated that the addition of NaCl salt decreased the glycerol settling time significantly up to more than five times. However, NaCl inclusion rates of more than 3 g to the mixture (i.e. 5 and 10 g) resulted in significantly less methyl ester purity due to the occurrence of miniemulsion phenomenon. Overall, addition of 1 g NaCl/100 ml glycerol–biodiesel mixture was found as optimal by accelerating the decantation process by 100% while maintaining the methyl ester purity as high <sup>[26]</sup>.

Javad Karimi-Sabet et al. (2017) in his review presented that Microalgae can be a candidate for production of biodiesel as they possess a hard cell wall that prevents intracellular lipids leaving out from the cells. Direct transesterification has the potential for destruction of microalgae hard cell wall and conversion of extracted lipids to biodiesel that consequently reduces the total energy consumption. Temperature, Time, Methanol-to-dry algae, Hexane-to-dry algae, and Moisture content. Thirty-two experiments were designed to perform in a batch reactor, and biodiesel efficiency was found to be in between 0.44% and 99.32% <sup>[27]</sup>.

Ortega Roman et al. (2015) presented paper on transesterification with alternative energy sources, i.e. ultrasound method from Sativa oil. Ultrasonic probe (20 kHz, 70% duty cycle, 50% amplitude). Design of experiments included the combination of sonication and agitation cycles, w/wo heating (50<sup>0</sup>C). To gain knowledge about the implications of the proposed methodology, conventional transesterification was optimized, resulting in higher needs on catalyst concentration and reaction time, compared to the proposed reaction. Sonicated and non-sonicated transesterification provides similar FAME yields, although sonication reduces reaction inputs and energy

consumption. Moreover, the combination of sonication and agitation steps significantly increases FAME yield while reducing glyceride concentration. Although there are no significant differences in terms of FAME content and properties, all of them meeting EN 14214, when sonication plus agitation cycles are supplemented with heating, the inclusion of heating steps shows lower glyceride concentration but higher energy consumption. Therefore, it may be concluded that BD produced by sonication combining agitation cycles requires lower amount of energy for its synthesis and better fitting of EN 14214<sup>[28]</sup>.

I Choedkiatsakul et al. (2014), investigates the production of biodiesel from palm oil using a combined mechanical stirred and ultrasonic reactor. The incorporation of mechanical stirring into the ultrasonic reactor explored the further improvement the transesterification of palm oil. Initial reaction rate values were 54.1, 142.9 and 164.2 mmol/L min for the mechanical-stirred (MS), ultrasonic (US) and MS–US reactors, respectively. Suitable methanol to oil molar ratio and the catalyst loading values were found to be 6 and 1 of oil, respectively. The effect of ultrasonic operating parameters; i.e. frequency, location, and number of transducer, has been investigated. Based on the conversion yield at the reactor outlet after 1 h, the number of transducers showed a relevant role in the reaction rate. Frequency and transducer location would appear to have no significant effect. The properties of the obtained biodiesel (density, viscosity, pour point, and flash point) satisfy the ASTM standard. The combined MS–US reactors improved the reaction rate affording the methyl esters in higher yield. A dual frequency US reactor integrated with a horizontal mechanical stirrer has been designed to produce biodiesel from palm oil and methanol in the presence of NaOH as a catalyst. Two transducer frequencies, 20 and 50 kHz, were employed in four possible locations along the length of the reactor. The results showed that this reactor provided high methyl ester yield in only 5 min reaction time, whereas the MS reactor required 60 min. The optimal operating conditions were; a methanol to oil molar ratio of 6 and a NaOH catalyst loading value of 1% wt of oil as operated in the circulation of reaction mixture with feed flow rate 55 mL/min. The influence of transducer number on methyl ester yield was more important than differences in transducer frequency and location along the

length of the reactor. The identical distance of each transducer along the length of the reactor was able to provide uniform cavitation intensity and resulted in the same effect at the different locations [29].

Le Tu Thanh et al., performed transesterification reaction of waste cooking oils (WCO) with methanol in the presence of a potassium hydroxide catalyst was performed in a continuous ultrasonic reactor of low-frequency 20 kHz with input capacity of 1 kW, in a two-step process. For the first step, the transesterification was carried out with the molar ratio of methanol to WCO of 2.5:1, and the amount of catalyst 0.7 wt.%. The yield of fatty acid methyl esters (FAME) was about 81%. A yield of FAME of around 99% was attained in the second step with the molar ratio of methanol to initial WCO of 1.5:1, and the amount of catalyst 0.3 wt.%. The FAME yield was extremely high even at the short residence time of the reactants in the ultrasonic reactor (less than 1 min for the two steps) at ambient temperature, and the total amount of time required to produce biodiesel was 15 h. The quality of the final biodiesel product meets the standards JIS K2390 and EN 14214 for biodiesel fuel.

The continuous ultrasonic reactor with a two-step process is a beneficial technique for the production of biodiesel from WCO. The use of WCO reduces the product cost of the raw material. The optimal conditions for the transesterification are the total molar ratio 4:1, KOH 1.0 wt.%, and the residence time in the reactor of 0.93 min for the entire process. Under these conditions, the recovery of biodiesel from WCO is 93.8 wt.% [30].

Kenji Okitsu et al. (2010), investigated biodiesel production from canola oil with methanol was performed in the presence of a base-catalyst by a circulation process at room temperature. In this process, the transesterification was accelerated by ultrasonic irradiation of low frequency (20 kHz) with an input capacity of 1 kW. The influences of various parameters on the transesterification reaction, including the amount of catalyst, the molar ratio of methanol to oil and the reaction time, were investigated. The objective of this work was to produce biodiesel satisfying the biodiesel–fuel standards of low energy consumption and material savings. The optimal conditions were: methanol/oil molar ratio of 5:1 and 0.7 wt.% catalyst in oil. Under these conditions, the

conversion of triglycerides to fatty acid methyl esters was greater than 99% within the reaction time of 50 min. Crude biodiesel was purified by washing with tap water and drying at 70°C under reduced pressure.

This study demonstrates that the ultrasound-assisted transesterification reaction of canola oil and methanol with potassium hydroxide is very efficient. The maximum yield was more than 99% with the methanol/oil molar ratio of 5:1, 0.7 wt.% potassium hydroxide catalyst and 50 min reaction time. The cost of the process can be significantly reduced using cheap oils such as waste frying oils <sup>[31]</sup>.

Leonardo S.G. Teixeira et al., compared transesterification of fatty acids by means of ultrasonic energy has been used for biodiesel production from different vegetable oils. However, application of ultrasonic irradiation for biodiesel production from beef tallow has received little attention. In this work, the transesterification of beef tallow with methanol was performed in the presence of potassium hydroxide as a catalyst using ultrasound irradiation (400W, 24 kHz). The reaction time, conversion and biodiesel quality were compared with that seen in conventional transesterification. The results indicated that the reaction conversion and biodiesel quality were similar; however, the use of ultrasonic irradiation decreased the reaction time, showing that this method may be a promising alternative to the conventional method.

Unlike vegetable oils, beef tallow's transesterification tends to form a gel-like material in some experimental runs and the high concentration of saturated fatty acid components in beef tallow is one of the suspects for this phenomenon. Unlike vegetable oils, beef tallow's transesterification tends to form a gel-like material in some experimental runs and the high concentration of saturated fatty acid components in beef tallow is one of the suspects for this phenomenon<sup>[32]</sup>.

### **2.3 Microwave method of biodiesel production:-**

D C Pandare et al. (2017), his paper stated the Use of microwave irradiation for enzymatic production of biodiesel. Microwave enzymatic catalysis process showed uniform heat transfer and remarkable increase in mass transfer to the reaction mixture. Therefore, the process diminished the time boundaries of reaction completion up to

240 minutes as well as intensified the conversion up to 94%. Use of microwave technique has also reduced the evaporation losses as compared to the reaction operated in conventional mode [33].

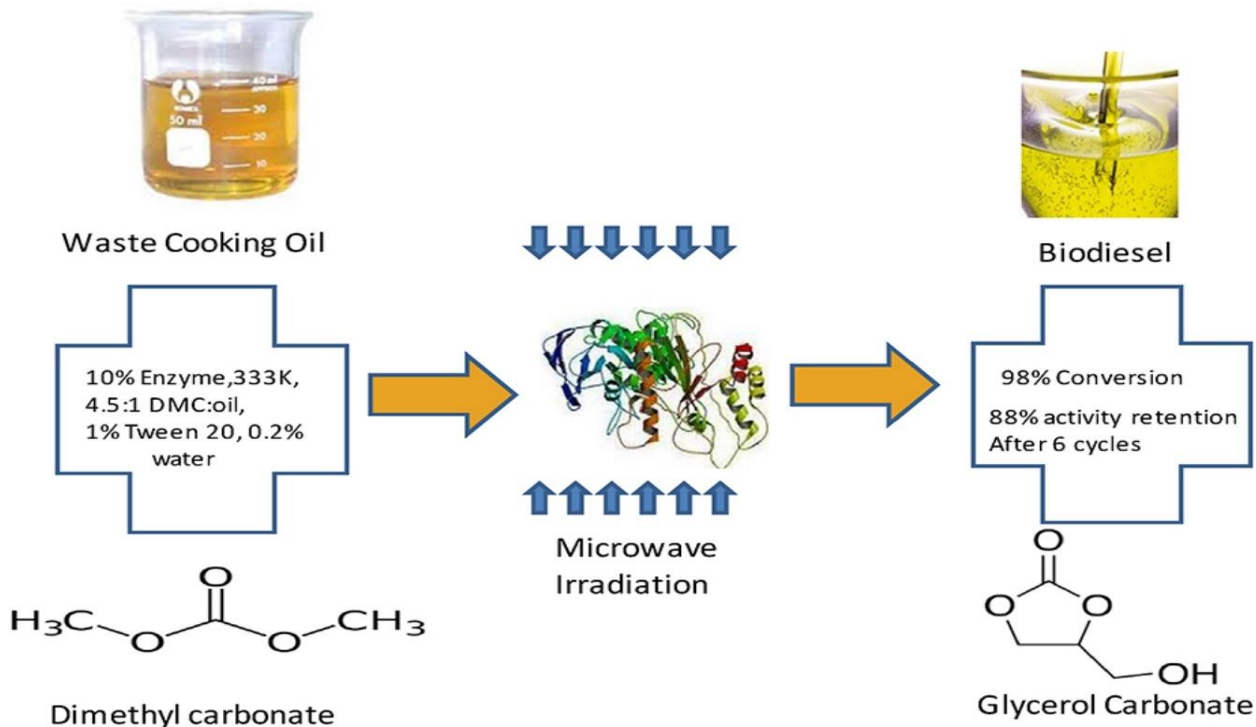


Fig. 2.5 Schematic diagram of reactions for microwave assisted biodiesel production [34]

Luiz A. Jermolovicius et al., mentioned following things in his paper: Transesterification microwave enhancement is done by changing kinetics parameters; Microwave heated process higher productivity was quantified and explained; Complete kinetic equation justify transesterification Independence from ethanol; Kinetic equation under microwave irradiation induces operational optimization. Three kinds of biodiesel production processes are compared and it is shown that the microwave heated acid process has higher productivity than the conventional heating alkaline or acid processes. The reaction time to reach the maximum yield in microwave-irradiated process is reduced to 50% of the time required by conventional heating processes. An optimization of process variables was done to identify the optimum microwave process



irradiation. Searching an explanation for this non thermal microwave effect, a statistical design to establish the complete chemical kinetic equation for soya oil transesterification with ethanol catalyzed by methane sulfonic acid was realized. The kinetic results show that this process has a complex mechanism that can be seen as a pseudo -1.6 global order reaction with low activation energy. Precisely, the order is 0.004 with respect to ethanol and -1.6 to soya oil. An analysis of this negative order was done and it brings a little more information to understand how to get best returns from biodiesel operations<sup>[35]</sup>.

P.S. Barela et al. (2017) proposed a microwave-assisted digestion (MW-AD) using diluted nitric acid and hydrogen peroxide as auxiliary reagent was proposed for the first time for biodiesel for further trace elements determination by sector field inductively coupled plasma mass spectrometry (SF-ICP-MS). Biodiesel samples were digested under high-pressure closed vessels and the concentration of nitric acid (1, 2, 3.5, 7, 9.3 and 14 mol L<sup>-1</sup>) and volume of hydrogen peroxide (0, 1, 1.5, 2 and 3 mL) were evaluated in order to obtain high efficiency of digestion. The efficiency of biodiesel digestion was determined by residual carbon content and residual acidity in final solutions. Up to 700 mg of biodiesel were completely digested using 7 mol L<sup>-1</sup> nitric acid and 2 mL of hydrogen peroxide in high-pressure quartz vessels assisted by microwave radiation at 900W during 60 min (included 20 min for cooling step). Residual carbon content and residual acidity in digests obtained after MW-AD using optimized conditions were lower than 9 and 4%, respectively. After sample digestion, Ba, Cr, Cu, Mn, Mo, Ni, Pb, Sr, V and Zn were determined by SF-ICP-MS and analytes recoveries were in the range of 94 to 109%. Accuracy was evaluated using certified reference material (used oil) and agreement higher than 94% was achieved. Although biodiesel digestion could be performed using concentrated nitric acid, the use of diluted acid was preferable in view of the low reagent consumption and low blanks values, achieving better limits of detection (LODs) that is important aspect to green chemistry recommendations for trace elements analysis. The high sample mass (up to 700 mg) can be digested without exceeding the maximum operating pressure (80 bar) of the microwave system, combining good performance for biodiesel digestion, safety and

relatively high sample throughput (up to eight samples could be simultaneously digested). Therefore, possible interferences in the determination step caused by high acid concentration or high residual carbon content in resultant solutions could be avoided. Moreover, the use of diluted nitric acid instead of concentrated acid reduces the blank values significantly and it was possible to achieve better LODs. These facts are important aspects concerning trace elements determination in biodiesel<sup>[36]</sup>.

Jo Han Ng et al. investigated the utilization of a microwave-assisted pyrolysis technique to convert crude glycerol from biodiesel waste into usable bioenergy source. Operating conditions ranged from a temperature of 300–800°C at carrier gas flow rates of 100–2000 mL/min, with the effects of carbonaceous catalyst on the selectivity of reaction. The third factor of carbonaceous catalyst shows a predisposition towards hydrogen gas selectivity, leading to a lower overall gaseous product mass when factoring in products from all phases. An analysis of the energy content revealed that overall energy profit increases with decreasing temperature and increasing residence time. This concurs with solid energy content increasing in the same conditions, while it increases for liquid and gaseous products with decreasing temperature and flow rate, respectively. The conversion of waste into portable and energy profit positive products through pyrolysis makes crude glycerol a potential candidate for bioenergy production of bio-oil and syngas.

Results from this experimental study have shown the potential of crude glycerol from biodiesel production waste as a technically feasible pyrolysis choice for energy production. For this microwave-assisted pyrolysis of crude glycerol, the proportions of pyrolysis product phases are dependent on carrier gas flow rates and reaction temperatures. It is apparent that the factor of gas carrier flow rate is the qualifying criteria, before reacting temperature can affect the pyrolysis process<sup>[37]</sup>.

Honglei Zhang et al. discussed about the traditional heating and microwave assisted method for biodiesel production using cation ion-exchange resin particles (CERP)/PES catalytic membrane were comparatively studied to achieve economic and effective method for utilization of free fatty acids (FFAs) from waste cooking oil (WCO). The

optimal esterification conditions of the two methods were investigated and the experimental results showed that microwave irradiation exhibited a remarkable enhanced effect for esterification compared with that of traditional heating method. The FFAs conversion of microwave assisted esterification reached 97.4% under the optimal conditions of reaction temperature 60<sup>0</sup>C, methanol/acidified oil mass ratio 2.0:1, catalytic membrane (annealed at 120<sup>0</sup>C) loading 3 g, microwave power 360W and reaction time 90 min. The study results showed that it is a fast, easy and green way to produce biodiesel applying microwave irradiation.

Compared with traditional heating method, microwave assisted esterification needs less reaction time, lower reaction temperature, less energy and lower methanol additive. Thus, manufacturing biodiesel using microwave represents a fast, easy and effective route with advantages of a short reaction time, a low methanol/acidified oil mass ratio, an ease of operation, reduced energy consumption and all with lower production cost<sup>[38]</sup>.

Wei Liu et al. studied that biodiesel production from waste cooking oil with high free fatty acids usually requires esterification step to produce fatty acid methyl/ethyl ester. In the present work, the heterogeneous catalyst amino-phosphonic acid resin D418 has been successfully utilized in the energy-efficient microwave-assisted esterification reaction of fatty acid ethyl ester (FAEE) from free fatty acid (FFA) stearic acid with short-chain alcohol ethanol. Under the reaction conditions of 9 wt% D418 and 11: 1 M ratio of ethanol to stearic acid at 353 K and atmospheric pressure, more than 90% conversion of the esterification was achieved in 7 h by microwave heating, while it took about 12 h by conventional heating. Moreover, the kinetics of this esterification reaction has been studied, and the relevant values of activation energy and pre-exponential factor were obtained.

From the studies on esterification of stearic acid with ethanol over D418 under microwave irradiation reported in this paper, we can draw conclusion bases on the above studies. D418 exhibited remarkable catalytic performance for ethyl stearate formation, and the conversion ratio of stearic acid could reach more than 90% under microwave irradiation. Compared with traditional heating esterification, microwave

assisted esterification needs less reaction time, lower temperature, less energy and lower ethanol additive. Moreover, the kinetic of the microwave assisted esterification of stearic acid with ethanol catalyzed by D418 was experimentally investigated <sup>[39]</sup>

Amit Kumar Sharma et al. (2016), explores the integrated approach for the sustainable production of biodiesel from *Chlorella vulgaris* microalgae. The microalgae were cultivated in 10 m<sup>2</sup> open raceway pond at semi-continuous mode with optimum volumetric and areal production of 28.105 kg/L/y and 71.51 t/h/y, respectively. Alum was used as flocculent for harvesting the microalgae and optimized at different pH. Lipid was extracted using chloroform: methanol (2:1) and having 12.39% of FFA. Effect of various reaction conditions such as effect of catalyst, methanol:lipid ratio, reaction temperature and time on biodiesel yields were studied under microwave irradiation; and 84.01% of biodiesel yield was obtained under optimized reaction conditions. A comparison was also made between the biodiesel productions under conventional heating and microwave irradiation.

The present study describes the integrated approach of upstream and downstream processes for microwave assisted sustainable biodiesel production from microalgae *Chlorella vulgaris*. The results revealed that *Chlorella vulgaris* was successfully grown in open raceway pond at semi-continuous mode with biomass productivity of 19.61 g/m<sup>2</sup>/day under outdoor conditions. 84.01% biodiesel yield was achieved via two-step microwave assisted acid-base transesterification. Moreover, *Chlorella vulgaris* is able to produce 13.62 ton/hectare/year biodiesel annually<sup>[40]</sup>.

## **CHAPTER-3**

### **METHODOLOGY**

#### **3.1 What is Solar assisted biodiesel production?**

We know that solar radiations carries temperature of about 5700 K <sup>[41]</sup>. This thermal energy of solar radiation is utilized for chemical heating required in transesterification process. Before proceeding to the experiment, we need to discuss about the traditional method of producing biodiesel i.e. Mechanical method. Biodiesel is a chemical composition of fatty acid methyl ester, non-toxic and biodegradable in nature obtained from renewable sources. It is derived from edible or non-edible oils, animal fats or from vegetable seeds. In this project, sesame oil is used as raw material to harness biodiesel originally derived from sesame seeds. Transesterification reaction is an essential reaction carried out in the process of synthesizing biodiesel. The biodiesel production process involves heating a triglyceride with alcohol in the presence of catalyst. Under this project, biodiesel is produced from sesame oil using potassium hydroxide as catalyst. The results were optimized by using Taguchi's method of optimization. After performing conventional method of biodiesel production, solar assisted transesterification was used in which thermal energy of solar irradiation was utilized for chemical heating process in biodiesel synthesis.

#### **3.2 Methodology:-**

##### **3.2.1 Concentrated Solar cooker:-**

Concentrated solar cooker works on the principle of convex lens. Reflective surfaces of collectors concentrates sunlight at its focal point where light is absorbed and converted into heat. Concentrators increase the power flux of sunlight hundred times. The chief types of concentrating collectors include: compound parabolic, parabolic trough, fixed reflector moving receiver, fixed receiver moving reflector, Fresnel lens, and central receiver [42]. A concentrating module uses optical elements such as Fresnel lens to increase the amount of sunlight incident onto it. Concentrating modules/arrays track the sun and use concentrating devices to reflect direct sunlight onto the container

in order to provide sufficient heat for various purposes or to produce electricity directly. Concentrating solar collectors in Concentrated Solar Power (CSP) concentrate sunlight at its focus onto a receiver where it heats a heat transfer fluid that exchanges its absorbed heat to water to produce steam to power a steam turbine-generator (STG) to produce electricity. It can cook food for larger number of people as well as faster than regular box- type. Bigger concentrator cookers have the capacity to cook food for at least 15 persons at a time. Paraboloid concentrating type of solar cooker and panel type solar cooker are few of the models of solar food cooking technology used worldwide.

A solar energy collector is equipped with at least one reflector panel, number of solar receivers (in my apparatus 36 reflector plates are installed), and a supporting structure that supports the reflector panels in a manner that defines the reflector trough having a trough base. The reflective walls of paraboloid solar collector is installed in such a way that concentration of reflected sunlight rays at focal point is utilized for transesterification process.



Fig. 3.1 Concentrated Solar Cooker of DTU Solar Green Energy Lab

**Table 3.1 Specifications of concentrated solar cooker**

Diameter of outer circle	1.42m
Rim angle	121.5 <sup>0</sup>
Concentration ratio of dish	33
Focal length of dish	0.28m
Aperture area of dish	1.54 m <sup>2</sup>

Solar cooker is an instrument which utilizes solar radiation and helps in saving conventional fuels. Also it does not end with heating up the kitchen along with its simplicity. It is energy efficient and also helps in fighting global warming. In India, these technologies are utilized at various places to cook food for large number of people.

### **3.2.2 Determination of FFA content in the sesame oil:-**

Determining the FFA content is essential because it helps in deciding the type of transesterification process will be used. If FFA content is found to be less than 2.5 weight% then transesterification process will be alkali based else it will be acid based. Titration is performed to determine the FFA content of the feedstock oil. Under this, phenolphthalein indicator solution was prepared in which mixing of 0.05g phenolphthalein was done with 50ml ethanol and 50ml of distilled water. After this, stirring of the solution was done for few seconds and hence phenolphthalein indicator solution is prepared. Another solution is prepared which contains 1g of NaOH is mixed with 100ml of distilled water and then we dilute it by adding 900ml of distilled water. This solution is poured into burette.

In another beaker, mix 1ml of sesame oil with 10ml of methanol. Mix two or three drops of phenolphthalein indicator in the oil and alcohol solution. After this, 0.1%

NaOH solution is added into oil-alcohol-phenolphthalein solution using burette till the solution in the beaker appears pink. As soon as pink color appears in beaker, titration is assumed to be completed. The FFA content is calculated according to the amount of 0.1% NaOH solution used in titration. The table below shows the information of the FFA content:-

**Table 3.2 Data of FFA content obtained by titration of ten samples of sesame oil**

<b>Titration of 0.1% NaOH (in ml)</b>	2	2.3	2.1	2.4	2.1	2.3	2	2.2	2.3	2
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**Table 3.3 Details of FFA content according to ml of 0.1% NaOH used in titration**

<b>Millilitres of 0.1% NaOH</b>	<b>FFA content in weight%</b>
0	0
0.5	0.357822
1	0.715644
1.5	1.073466
2	1.431289
2.5	1.789111
3	2.146933
3.5	2.504755
4	2.862377
4.5	3.220400
5	3.578222

Taking an average value of the above observations, it comes out to be 2.17ml titration of 0.1% NaOH which shows that the FFA content is around to be 1.43%. So transesterification will be alkali base catalyzed for biodiesel production from sesame oil.

### **3.2.3 Preparation of sample:-**

a.) Heat the oil upto 100<sup>0</sup>C so that if there is any moisture content in the raw oil, it gets flashed off into vapors and our oil remains moisture free. This heating was carried out in the concentrated solar cooker. If it is carried out during summer morning around 10a.m it will take about 1hour to reach the 100<sup>0</sup>C and around 12:00p.m it took half an



hour to attain the temperature. The average solar radiation intensity over New Delhi during April was approximately  $6.88 \text{ kW/m}^2/\text{day}$ <sup>[43]</sup>.

b.) In second step, prepare catalyst solution till our oil temperature reaches the temperature of about  $40^\circ\text{C}$ . Depending upon the molar ratio, choose the amount of KOH and methanol to prepare the catalyst. For instance, if one wants to opt for 6:1 molar ratio, following steps will be performed:-

1. Take 50 grams of oil by weighing it on digital weight balance. After this perform step 3.2.3.a .

2. Take 0.5 grams of potassium hydroxide(KOH) and 11 grams of methanol and stir it well so that KOH gets completely dissolved in the methanol. This was carried out manually by using glass rod and it took about 5 minutes to dissolve it completely.

If one wish to produce it on large scale then use mechanical stirrer.

3. As soon as mixing is done and oil temperature reaches  $40^\circ\text{C}$ , pour the catalyst solution in the beaker containing oil. Don't mix the solution when the oil is at high temperature as methanol will evaporate.

4. This solution was kept in a black painted container as black is good absorber of heat coming out through solar radiation. The beaker is tightly closed and kept at the focal point of concentrated solar cooker.

5. Since concentrated solar cooker has the capability to attain the temperature of  $300^\circ\text{C}$  and we need temperature of  $60^\circ\text{C}$ - $70^\circ\text{C}$ , so experiments were carried out during 9:00 a.m- 12:00 p.m else appropriate results were difficult to obtain.

- 6.) It will take approximately 2-2:15 hours to form biodiesel. Unlike mechanical method, in this no stirring is done. Biodiesel is formed due to thermal agitation from the solar radiations which is available free of cost. In between if one opens the container to note down the temperatures and check the formation of biodiesel, glycerin deposition will be seen at the base of the container.

- 7.) Take different readings by varying the time in order to get the appropriate yield.

After this procedure, pour the content into separating flask, so that glycerin gets settled down at the base of the flask. Allow glycerin to settle down properly for a day. Then after separate the oil from glycerin.

8.) For water wash process, take water 35% of the weight of the oil and heat it till it reaches  $40^{\circ}\text{C}$ . Pour that water in the flask containing biodiesel so that catalyst can be separated by dissolving it in water. And remove water after 1 hour. Repeat this step 4-5 times until we start getting clear water.

9.) Heat the oil depending on its weight so that water get off into vapors and hence the clear biodiesel is obtained.

10.) To get more efficient results use concentrated solar cooker installed with trackers and sensor system. This is to eliminate the work load to stand every time whenever sun changes its position focus point also gets changed.

11.) Its properties were measured with Anton Paar viscometer. This instrument provide us the result within two minutes. The properties such as kinematic viscosity, density and specific gravity are calculated with the help of this device. For calorific value measurement bomb calorimeter was used.



**Fig. 3.2 Synthesis of Biodiesel followed by water wash**

### 3.3 Box- type solar cooker :-

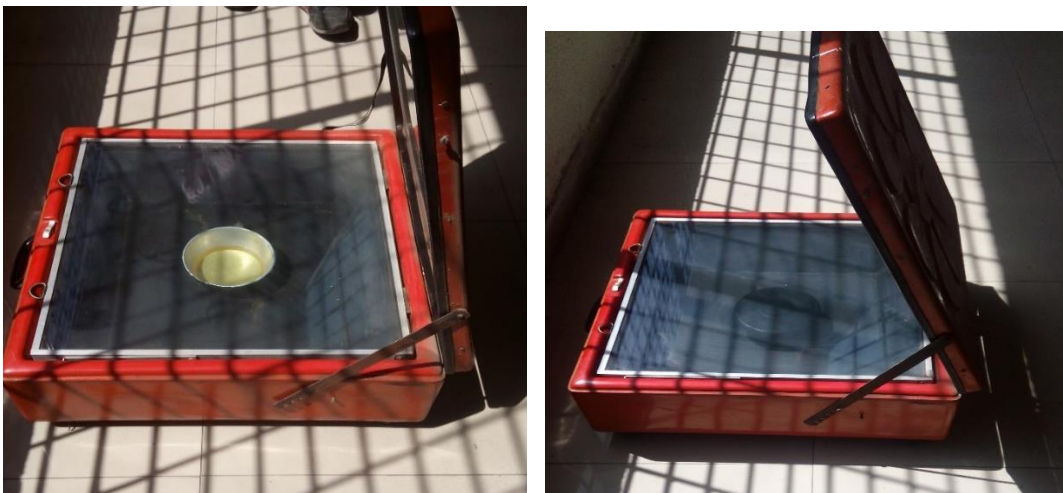
Among the available models of the solar cookers in our country box type solar cooker gets more attention because of its simplicity and easy to handle nature. In India, solar cooking has reached high level, 0.6 million Indians are using box- type solar cooker. The high energy photons of solar radiations are absorbed by transparent glass cover and the incident solar radiations convert it to thermal heat as the short wavelength gets converted into longer wavelength by the process of photo-thermal effect. Thermodynamically, the temperature of the exposed surface may achieve the temperature of sun at ideal equilibrium conditions, but at higher temperatures the convective and radiative losses becomes more dominant. In order to prevent the heat losses, insulation on sides and bottom is provided along with the double glazing over the black painted absorber. This type of solar cooker consists of metallic trapezoidal container which is black painted and is covered with glass window. The cooking tray or trapezoidal tray is kept insulated with glass wool filled between the spaces. The incoming solar radiations fall on the glass lid and passes to strike the blackened cooking pots and the cooking tray. The glass cover transmits the radiation of short wavelengths into longer wavelengths. The temperature of box increases until the equilibrium is attained between the heat received through glazing and heat lost by glazed surface. A plane reflecting mirror of same size as that of aperture area is used for collection of solar radiation on the aperture.



**Fig. 3.3 Box type solar cooker of DTU solar and green energy lab**

### 3.3.1 Comparison between concentrated solar cooker and box type solar cooker:-

As we have seen earlier that heating of oil from first step to the last was done by concentrated solar cooker. But in this, heating process was done by box type solar cooker. It can achieve the temperature of about  $150^{\circ}\text{C}$ . but as compared to concentrated solar cooker it took more time but results were more good. As the container get uniform heat as a result of greenhouse effect. Although shifting was required as sun rays incident on the cooker changes its position due to rotation of the earth. But properties obtained were upto the mark. In this also heating leads to thermal agitation due to which biodiesel formation takes place. Though it is a time consuming process unlike conventional mechanical method but very cost effective as well as non-laborious process. It took about 2.5-3 hours when we used to start our experiments near 11:30a.m – 3:00 p.m. if we keep it in a slight shady portion as shown in the figure, it was easier for us to maintain the temperature condition ( $60^{\circ}$ - $70^{\circ}\text{C}$ ) as required without any shifting required from time to time as it was done in concentrated solar cooker.



**Fig. 3.4 Biodiesel made in Box type solar cooker**

### **3.4 Comparison of the above methods with the mechanical method:-**

Although mechanical method is quicker than the solar assisted method but it requires more expense of electricity. Heating will be done by usual heater or induction heater which will be costlier than solar energy which is free of cost.

Moreover, in this method mechanical mechanism is required to stir the prepared solution because in this temperature provided is not sufficient for self agitation so mechanical stirring is also required for agitation. On small scale we use magnetic stirrer to stir the solution whenever required. So our solution gets prepared faster but it will be suitable for industrial use only. But our solar method is economic and less laborious. One can do every preparation and keep the container inside the cooker and can leave the setup for 2-3 hours in the sun and can come back for separation process.

This method is useful where we want to produce biodiesel at industrial level or on large scale. One can produce biodiesel at home and sell it in market for business purpose. 20-30% blend of diesel with biodiesel is done to for engine operations, which will be economical as compared to engine running on pure diesel. Moreover obnoxious emissions will be reduced to great extent. This will be understood in a better way when economic analysis will be shown in next topic.



**Fig. 3.5 Biodiesel made by Magnetic Stirrer**

## CHAPTER-4

### COST ANALYSIS

#### **4.1 Economic analysis of solar assisted biodiesel production versus mechanical method:-**

- a.) cost of oil- Rs. 100 per litre
- b.) cost of KOH- Rs. 500 per kilogram
- c.) cost of Methanol- Rs. 250 per litre

#### 3.5.1 Equipments involved in mechanical stirring method:-

Stirrer Capacity:- 1MLH

Stirrer Rating:- 600Watt

Stirrer cost:- Rs. 4200

Warranty:- 1 year

Induction heater rating:- 2KW

Induction heater cost:- Rs. 3000

Cost of electricity – Rs. 8/unit

#### 3.5.2 Equipments involved in solar assisted method:-

Cost of concentrated solar cooker:- Rs. 4500

Cost of box type solar cooker:- Rs. 2500

Warranty – 10-15 years

Total fixed cost will include cost of materials such as heater, stirrer, different types of solar cookers used. Similarly, operating cost will involve electricity bill caused by equipments operated during experiment time. Safety/storage cost will involve cost of oil, methanol, potassium hydroxide which requires storage.

In case of mechanical method,

Total fixed cost= induction heater cost+ stirrer cost

$$= 4,200 + 3,000$$

$$= \text{Rs.}7,200$$

Operating cost= Rs.4.267

Calculation:-

In New Delhi cost of electricity is Rs.8/unit

For 2KW heater, if we use it for 10 minutes in total process depending upon the quantity we are using,

$$2000 * (10/60) * 8 = \text{Rs.} 2.667$$

For 600W stirrer, if we use it for minimum of 20 minutes,

$$0.6 * (20/60) * 8 = \text{Rs.} 1.6$$

Total operating cost=2.667+1.6= Rs. 4.267

Storage cost= 100+500+250

$$= \text{Rs.} 850$$

## CHAPTER- 5

### RESULTS AND DISCUSSION

#### 5.1 Optimization by RESPONSE SURFACE METHOD:-

To achieve the maximum optimum conditions of calorific value and yield, minimum condition of kinematic viscosity, were determined by the use of statistical package Design-Expert, version 7.0.0(Stat- Ease, Inc., Minneapolis, MN, USA).To enquire about the interaction and quadratic effects of two independent variables i.e., molar ratio and catalyst concentration on yield, calorific value and kinematic viscosity the Central composite was implemented.

The maximum and minimum values of molar ratio was 6.31 and 4.19 respectively and that too of catalyst concentration was 1% and 0.5% respectively. The optimum outputs were produced during transesterification of sesame oil by various methods. The thorough optimized study consist of 15 combinations, which includes five replicates for the center point.

#### 5.2 Statistical analysis :-

The main factors influencing the transesterification includes reaction temperature, alcohol/oil molar ratio and catalyst concentration. Response surface method was opted to give optimized result to give a high yield of biodiesel. This method consist of three sequential process that generally starts at one reasonable operating condition, and further requires three stages to attain a set of “better” conditions at rapid and efficient rate. In the initial stage several experiments were conducted to the optimal value and best properties. In the second stage several runs were performed along the direction as indicated in the first stage until an optimal value was attained. In the last step a mathematical model was deduced and a profile was generated in response surface method showing optimal result.



**Table 5.1 Box type solar cooker analysis by Response Surface Method**

Runs	Input 1 Molar Ratio	Input 2 Catalyst Conc.(%)	Calorific Value Kcal/gm	Kinematic Viscosity mm <sup>2</sup> /sec	Yield %
1	4.5:1	0.5	36.987	7.253	84.78
2	5.25:1	0.75	37.5	6.4	90.95
3	5.25:1	0.75	37.8	6.8	90.95
4	6.31:1	0.75	42.481	4.999	96.72
5	5.25:1	1.1	38.999	5.999	91.39
6	5.25:1	0.75	37.5	6.34	90.95
7	6:1	1	40.088	5.11	94.86
8	6:1	0.5	39.984	5.3	93.67
9	4.5:1	1	36.999	7	87.47
10	5.25:1	0.75	37.5	6.5	90.95
11	5.25:1	0.75	37.56	6.342	90.95
12	4.19:1	0.75	35.888	7.1	85.38
13	5.25:1	0.4	35.999	6.434	88.47

The above table include molar ratio and catalyst concentration as the input variables and the value of the rest three columns were determined experimentally. The central point of the methanol/oil molar ratio was set at 5.25:1. Since higher catalyst concentration can lead to soap formation, catalyst amount (catalyst/oil) of 1.0% (w) was chosen as an upper level of catalyst concentration. The three-dimensional surface profiles were plotted showed that a higher yield could be obtained at a combination of 6:1 and 1% when the reaction temperature was around 80<sup>0</sup>C.

Design-Expert® Software

kinematic Visco.



X1 = A: Molar Ratio  
X2 = B: Catalyst Conc.

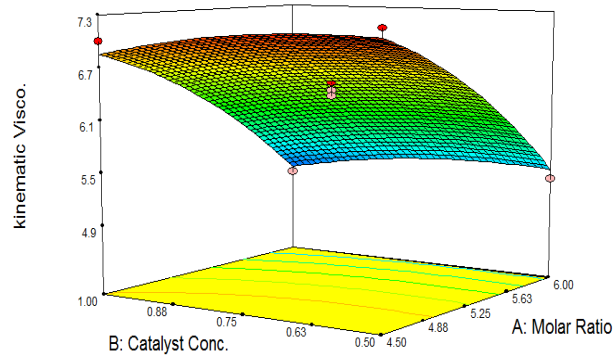


Fig. 5.1 Optimization of Kinematic Viscosity of sesame oil

Design-Expert® Software

Yield



X1 = A: Molar Ratio  
X2 = B: Catalyst Conc.

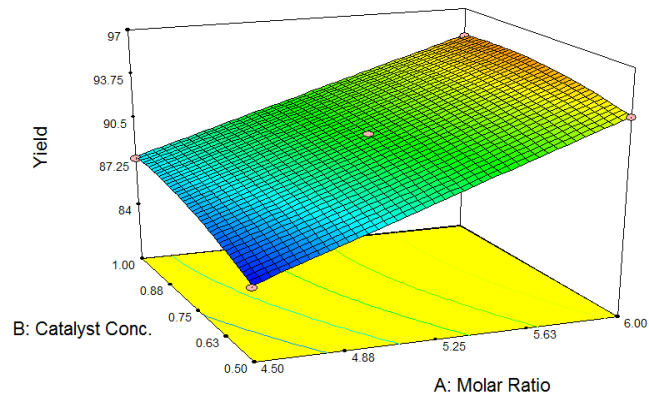


Fig. 5.2 Optimization of Yield of Sesame oil

Design-Expert® Software

Calorific value



X1 = A: Molar Ratio  
X2 = B: Catalyst Conc.

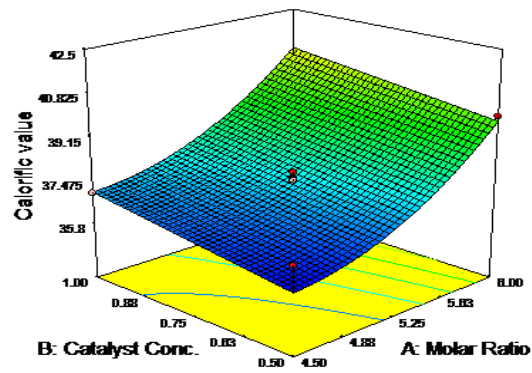


Fig. 5.3 Optimization of Calorific Value of Sesame oil

The values in the figures are transformed into real values. All the figures concludes that combination of 6:1 molar ratio and 1% catalyst concentration proved to be optimized result. Though the reaction temperature in the box type solar cooker was maintained around 70<sup>0</sup>C and time for which the sample was kept for around 3hours to 3.5 hours. The yield decreased and kinematic viscosity increased when the catalyst concentration and molar ratio was decreased below 0.5% and 4.5:1 respectively. The RESPONSE SURFACE METHOD exhibits the optimal value of the methanol/oil molar ratio for the yield, in which too high or too low values of the methanol/oil ratio have negative effects.

**Table 5.2 Concentrated type Solar cooker analysis by Response Surface Method**

Runs	FACTOR 1 Molar Ratio	FACTOR 2 Catalyst Conc. (%)	Yield (%)	Kinematic Viscosity (mm <sup>2</sup> /sec)	Calorific Value (kcal/gm)
1	5.25:1	0.4	90.98	8.634	38.54
2	4.5:1	1	90.2	8.991	34.13
3	4.5:1	0.5	90.6	9.411	33.47
4	4.19:1	0.75	90.88	9.785	32.69
5	5.25:1	0.75	92.5	7.757	39.67
6	6.31:1	0.75	95.72	4.778	43.79
7	5.25:1	0.75	92.57	8.001	39.64
8	5.25:1	0.75	92.49	7.8	39.57
9	5.25:1	0.75	92.5	7.779	39.6
10	6:1	0.5	93.59	5.416	42.87
11	6:1	1	93.88	5.212	43.36
12	5.25:1	1.1	92.912	7.316	40.84
13	5.25:1	0.75	92.64	7.921	39.6

Design-Expert® Software

Yield  
95.72  
90.2

X1 = A: Molar Ratio  
X2 = B: Catalyst Conc.

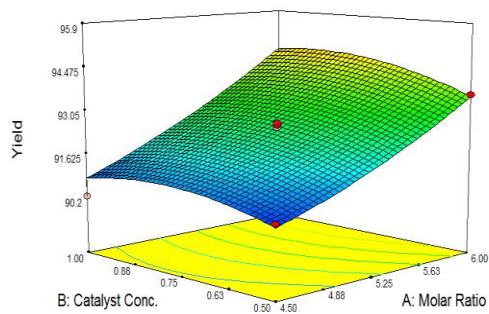


Fig. 5.4 Optimization of Yield

Design-Expert® Software

Kinematic Visc.  
9.785  
4.778

X1 = A: Molar Ratio  
X2 = B: Catalyst Conc.

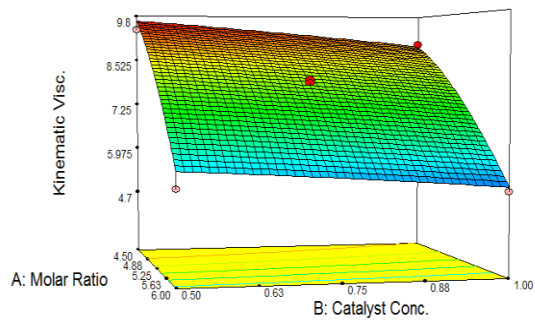


Fig. 5.5 Optimization of Kinematic Viscosity

Design-Expert® Software

Calorific Value  
43.79  
32.69

X1 = A: Molar Ratio  
X2 = B: Catalyst Conc.

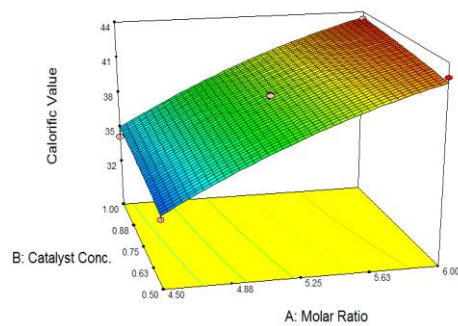


Fig. 5.6 Optimization of Calorific Value

The values in the figures are transformed into real values. All the figures concludes that combination of 6:1 molar ratio and 1% catalyst concentration proved to be optimized result. Though the reaction temperature in the concentrated type solar cooker was maintained around 85-90<sup>0</sup>C and time for which the sample was kept for around 2hours. The yield decreased and kinematic viscosity increased when the catalyst concentration and molar ratio was decreased below 0.5% and 4.5:1 respectively. The RESPONSE SURFACE METHOD exhibits the optimal value of the methanol/oil molar ratio for the yield, in which too high or too low values of the methanol/oil ratio have negative effects.

### **5.3 Mechanical stirrer analysis by Response Surface Method**

Runs	Molar Ratio	Catalyst Conc.(%)	Yield (%)	Kinematic Viscosity(mm <sup>2</sup> /sec)	Calorific Value(kcal/gm)
1	5.25:1	0.75	95.997	5.345	42.4
2	5.25:1	1.1	96.471	5.111	42.871
3	4.5:1	0.5	94.129	6	40.481
4	5.25:1	0.75	95.997	5.313	42.289
5	5.25:1	0.75	95.997	5.3	42.341
6	6.31:1	0.75	97.694	4	45.671
7	5.25:1	0.4	95.412	5.879	41.991
8	6.0:1	1.0	97.213	4.031	45.253
9	4.19:1	0.75	91.364	6.216	40.236
10	4.5:1	1.0	94.522	5.748	40.991
11	6.0:1	0.5	96.934	4.741	45.011
12	5.25:1	0.75	95.997	5.299	42.333
13	5.25:1	0.75	95.997	5.321	42.4

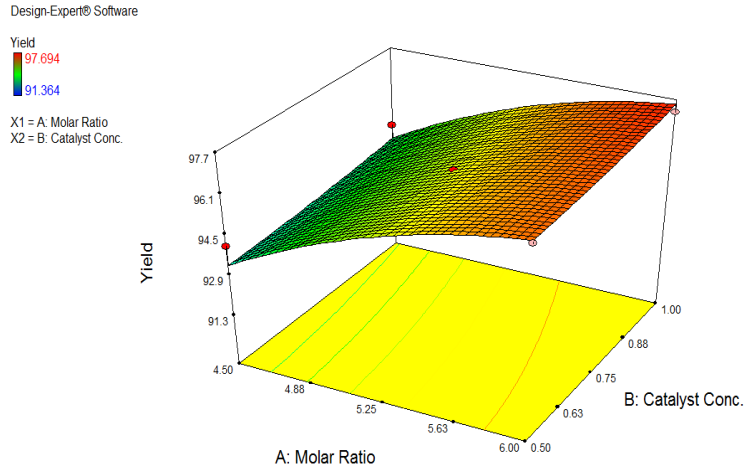


Fig. 5.7 Optimization of Yield

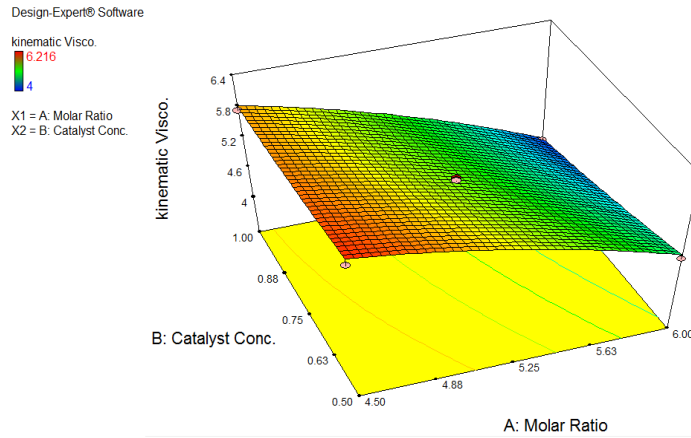


Fig. 5.8 Optimization Of Kinematic Viscosity

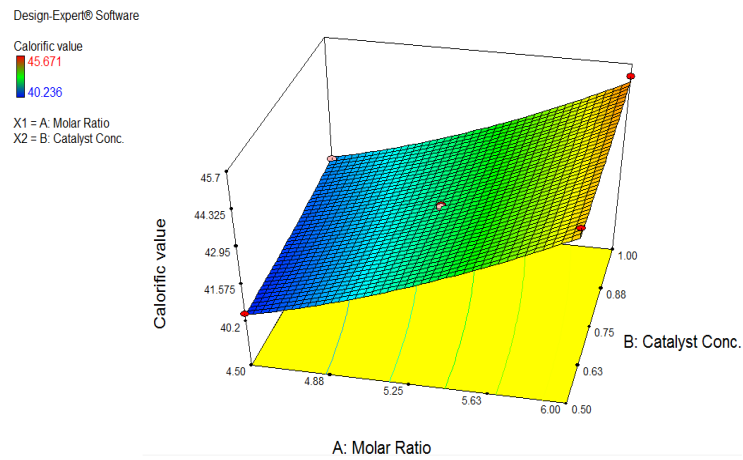


Fig. 5.9 Optimization of Calorific Value

The values in the figures are transformed into real values. All the figures concludes that combination of 6:1 molar ratio and 1% catalyst concentration proved to be optimized result. Though the reaction temperature in the concentrated type solar cooker was maintained around 60-70<sup>0</sup>C and time for which the sample was kept for around 30-45minutes. The yield decreased and kinematic viscosity increased when the catalyst concentration and molar ratio was decreased below 0.5% and 4.5:1 respectively. The RESPONSE SURFACE METHOD exhibits the optimal value of the methanol/oil molar ratio for the yield, in which too high or too low values of the methanol/oil ratio have negative effects.

### 5.3 Experiment design using Taguchi method:-

Optimization of process parameters was done by Taguchi’s approach which used three parameter: molar ratio, reaction temperature (<sup>0</sup>C) and catalyst concentration (%) were taken into consideration. The table below shows the factors and their levels-

#### i) Concentrated solar cooker:-

**Table 5.4 Process parameters and their levels**

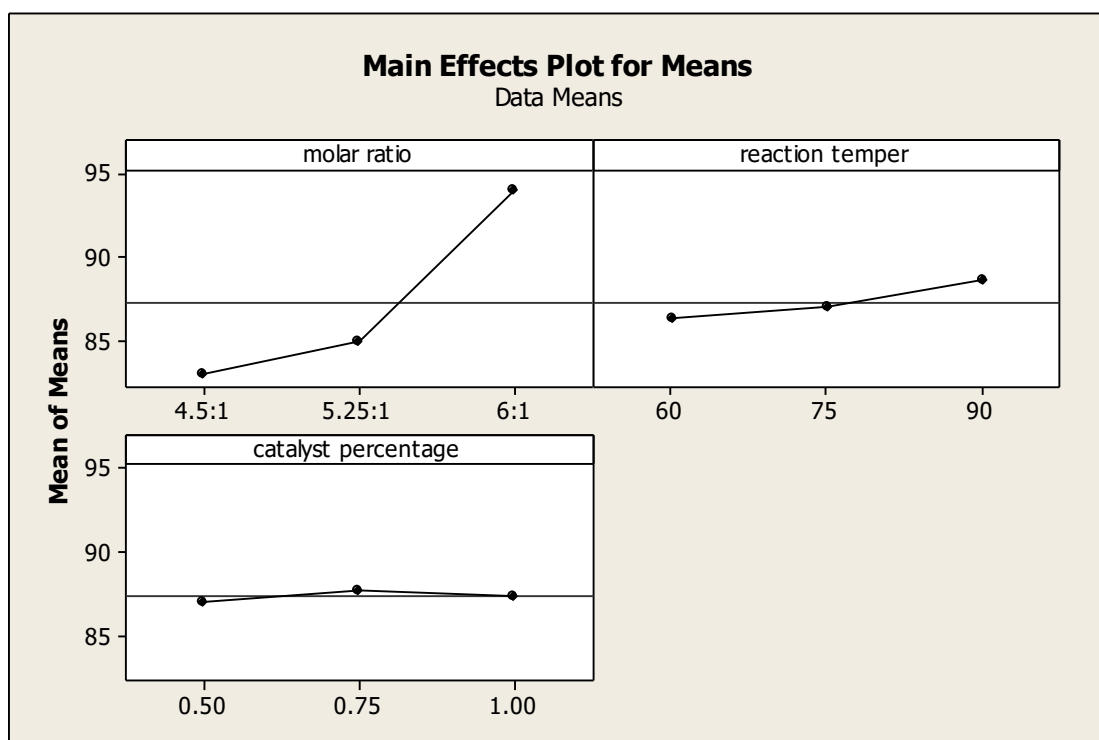
Parameters/Factors	LEVEL 1	LEVEL2	LEVEL3
Molar Ratio	4.5:1	5.25:1	6:01
Reaction Temperature( <sup>0</sup> C)	60	75	90
Catalyst Concentration (%)	0.5	0.75	1

On the basis of this 3- level 3-factor design, we adapt L9 orthogonal array by creating Taguchi design in Minitab 17 software. The following table shows the possible nine iterations of experiments as described by L9 orthogonal array along with the corresponding conversions yield of biodiesel found after performing nine experiments-

**Table 5.5 Analysis of yield:-**

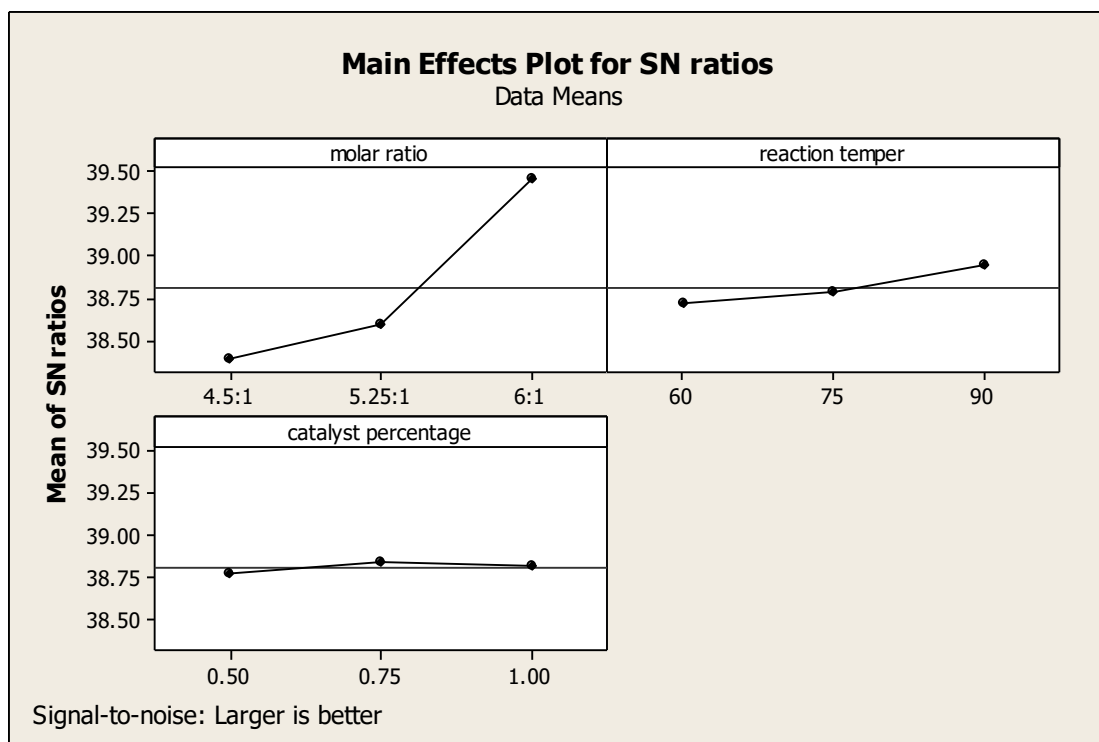
Exp No.	Molar ratio	Reaction temp.	Catalyst %	Yield(%)	SNRA1	MEAN1
1	4.5:1	60	0.5	82	38.27628	82
2	4.5:1	75	0.75	83	38.38156	83

3	4.5:1	90	1	84	38.48559	84	
4	5.25:1	60	0.75	84	38.48559	84	
5	5.25:1	75	1	85	38.58838	85	
6	5.25:1	90	0.5	86	38.68997	86	
7	6:1	60	1	93	39.36966	93	
8	6:1	75	0.5	93	39.36966	93	
9	6:1	90	0.75	96	39.64542	96	BEST



**Fig. 5.10 Graphical Variation of Yield**





**Fig. 5.11 Signal to Noise Ratio Plot with respect to factors**

The essential factor affecting yield is molar ratio. From the above graph, it can be observed that conversion yield increases with molar ratio of oil to alcohol ranging from 4.5:1 to 6:1. From the above graph it is understood that yield increased as temperature was increased. However we can't increase temperature beyond 90°C as yield will decrease.

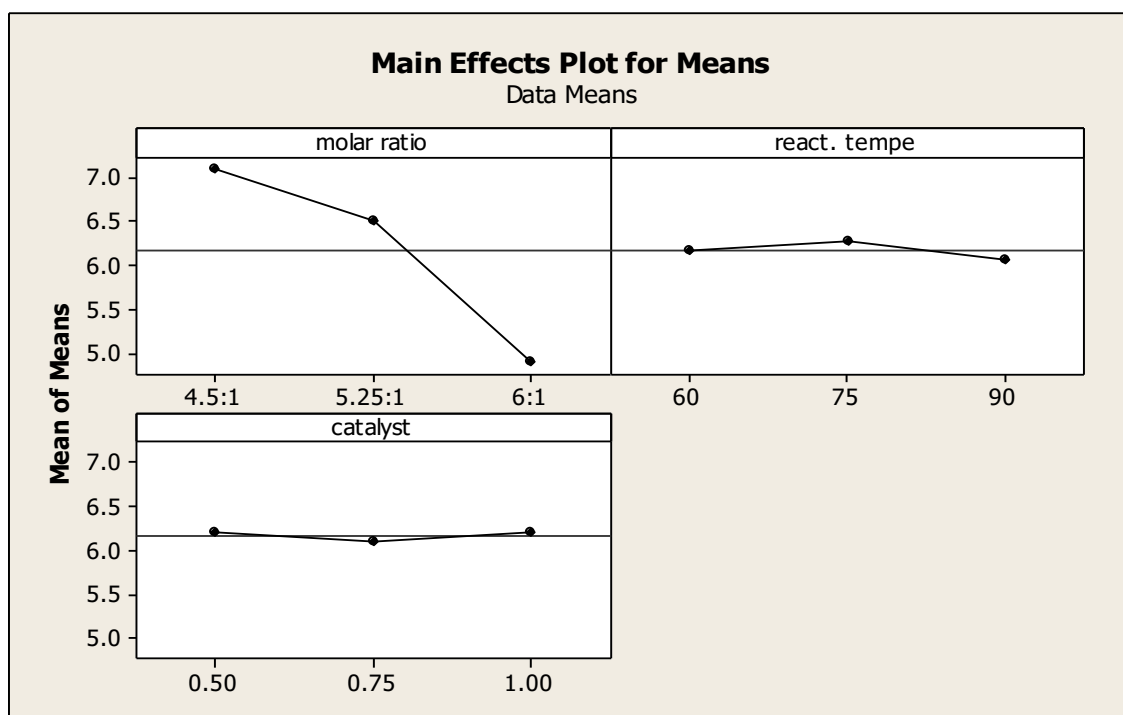
Since KOH is used as catalyst in transesterification reaction and concentration taken was 0.5%, 0.75% and 1% by weight. The yield increased from 0.5% to 1%. Less than 0.5% concentration will not be enough to enhance yield.

**Table 5.6 Analysis of kinematic viscosity**

Exp No.	Molar ratio	React. temp.	Catalyst	Viscosity	SNRA1	MEAN1
1	4.5:1	60	0.5	7.2	17.1466	7.2
2	4.5:1	75	0.75	7.1	17.0252	7.1
3	4.5:1	90	1	7	-16.902	7
4	5.25:1	60	0.75	6.4	16.1236	6.4

5	5.25:1	75	1	6.7	-	16.5215	6.7
6	5.25:1	90	0.5	6.4	-	16.1236	6.4
7	6:1	60	1	4.9	-	13.8039	4.9
8	6:1	75	0.5	5	-	13.9794	5
9	6:1	90	0.75	4.8	-	13.6248	4.8

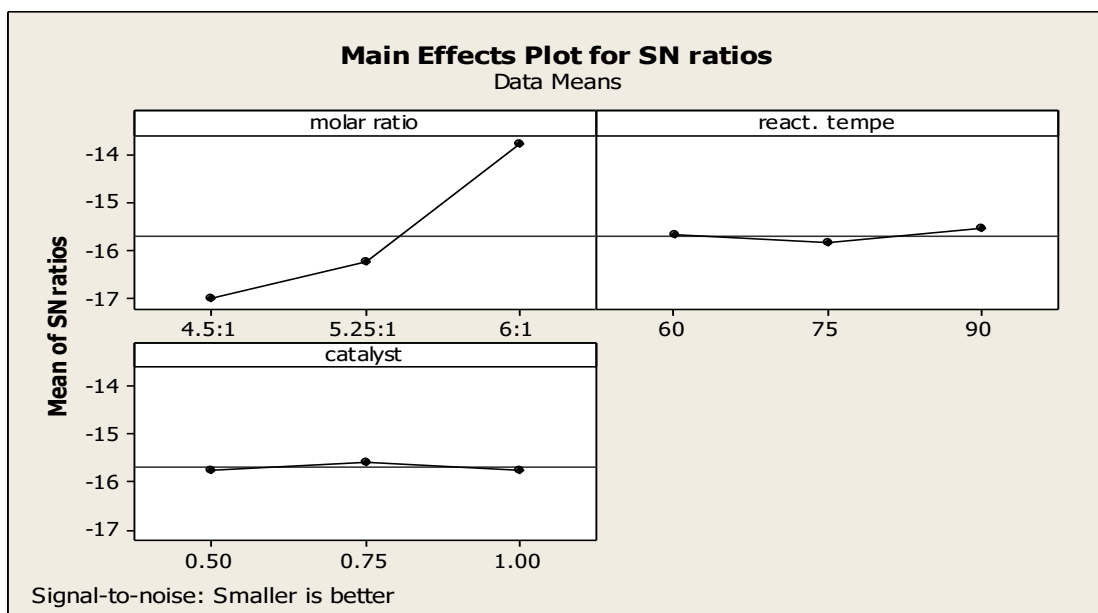
Best



**Fig. 5.12 Graphical Variation of Kinematic Viscosity**

The essential factor affecting kinematic viscosity is catalyst concentration and temperature along with molar ratio. From the above graph, it can be observed that kinematic viscosity decreased with molar ratio of oil to alcohol ranging from 4.5:1 to 6:1. From the above graph it is understood that best results obtained was at between 75- 90<sup>0</sup>C However we can't increase temperature beyond 90<sup>0</sup>C as kinematic viscosity will start increasing.

Since KOH is used as catalyst in transesterification reaction and concentration taken was 0.5%, 0.75% and 1% by weight. The kinematic viscosity decreased from 0.5% to 1%. Less than 0.5% concentration will not be enough to good results for viscosity.

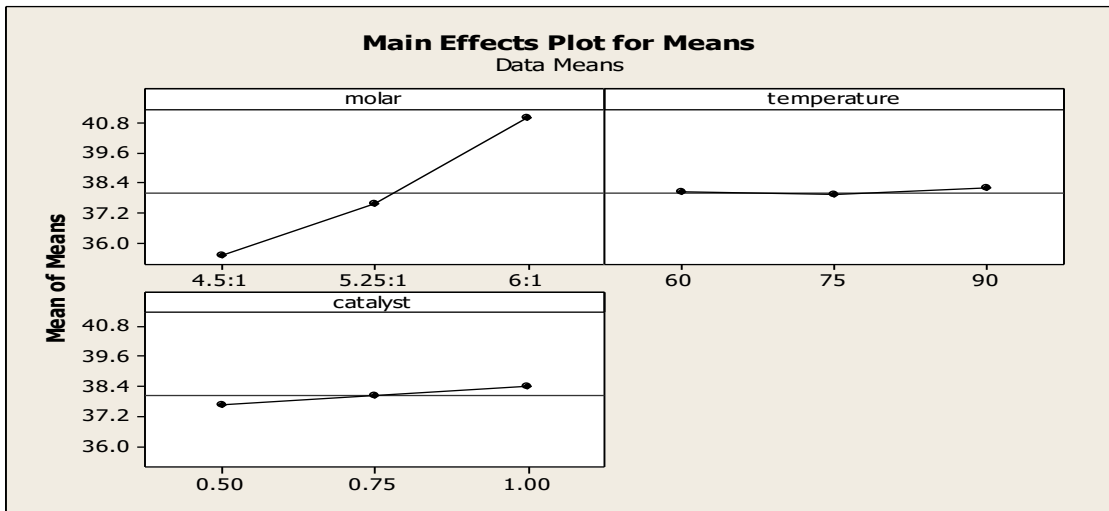


**Fig. 5.13 Signal to Noise Ratio Plot with respect to factors**

**Table 5.7 Analysis for calorific value**

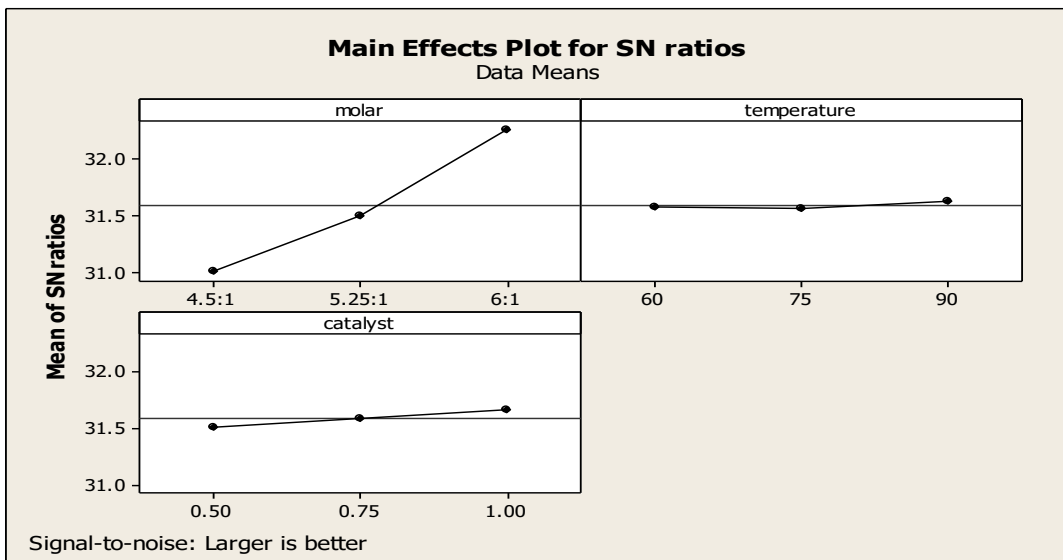
Exp No.	Molar ratio	Temperature	Catalyst	Calorific value	SNRA1	MEAN1
1	4.5:1	60	0.5	35	30.88136	35
2	4.5:1	75	0.75	36	31.12605	36
3	4.5:1	90	1	35.5	31.00457	35.5
4	5.25:1	60	0.75	37	31.36403	37
5	5.25:1	75	1	37.7	31.52683	37.7
6	5.25:1	90	0.5	38	31.59567	38
7	6:1	60	1	42	32.46499	42
8	6:1	75	0.5	40	32.0412	40
9	6:1	90	0.75	41	32.25568	41

Best



**Fig. 5.14 Graphical Variation of Calorific Value**

The essential factor affecting calorific value is molar ratio and catalyst concentration. From the above graph, it can be observed that calorific value increases with molar ratio of oil to alcohol ranging from 4.5:1 to 6:1. From the above graph it is understood that calorific value slightly increased as temperature was increased. However we can't increase temperature beyond 90°C as calorific value will decrease. Since KOH is used as catalyst in transesterification reaction and concentration taken was 0.5%, 0.75% and 1% by weight. The caloric value increased from 0.5% to 1%. Less than 0.5% concentration will not be enough to enhance calorific value.



**Fig. 5.15 Signal to Noise Ratio Plot with respect to factors**

ii) **For Box type solar cooker:-**

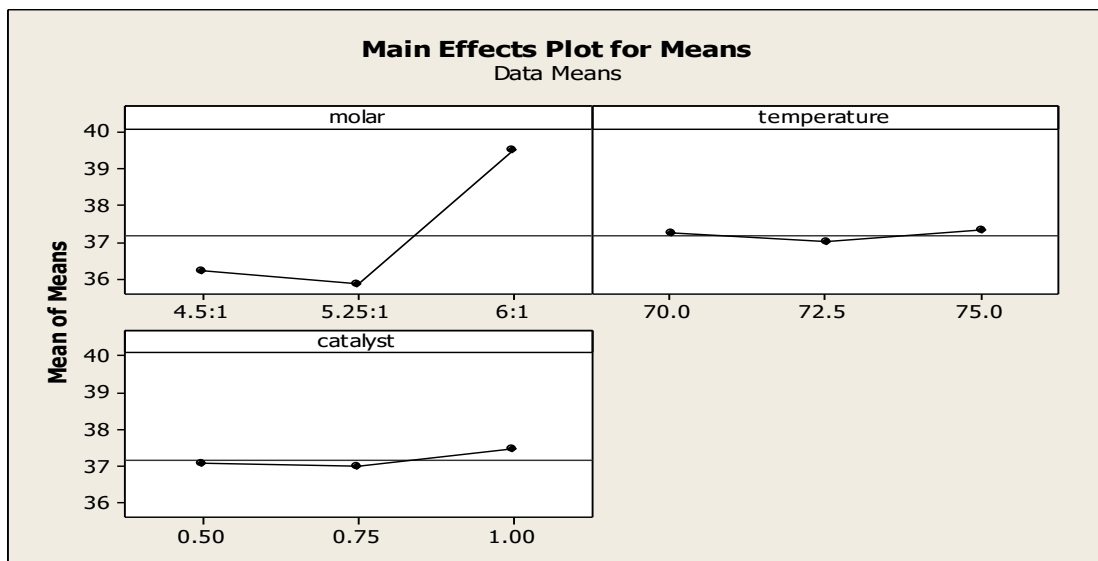
**Table 5.8 Process parameters and their levels**

Parameters/Factors	Level 1	Level 2	Level 3
Molar Ratio	4.5:1	5.25:1	6.0:1
Reaction Temperature	70	72.5	75
Catalyst Concentration (%)	0.5	0.75	1

The following table shows the possible nine iterations of experiments as described by L9 orthogonal array along with the corresponding conversions yield of biodiesel found after performing nine experiments-

**Table 5.9 Analysis of Calorific value**

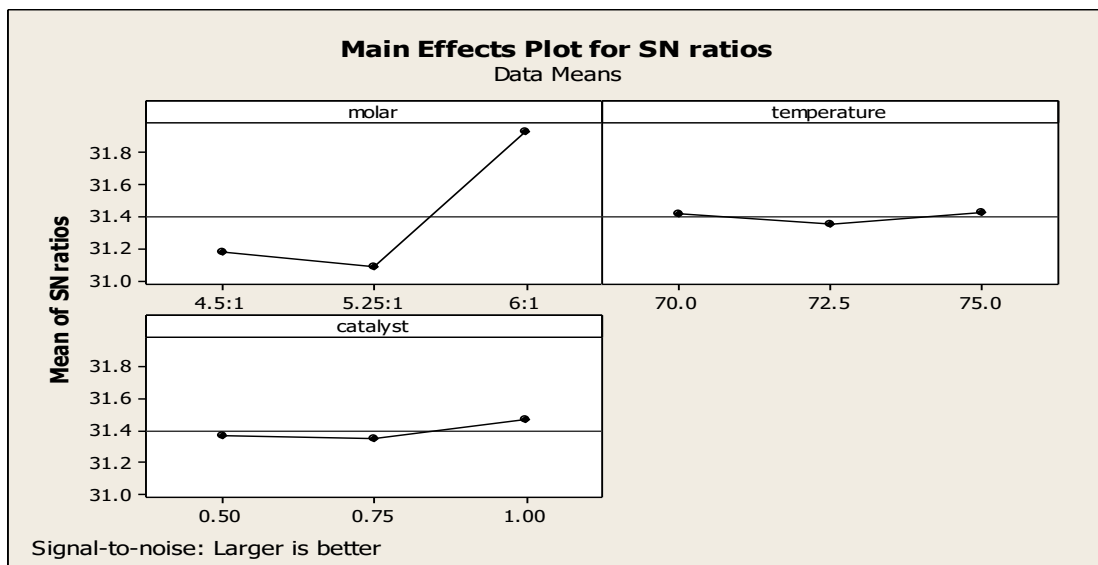
Exp No.	Molar	temperature	catalyst	Calorific value	SNRA1	MEAN1	
1	4.5:1	70	0.5	36.7	31.29332	36.7	
2	4.5:1	72.5	0.75	35	30.88136	35	
3	4.5:1	75	1	36.9	31.34053	36.9	
4	5.25:1	70	0.75	36	31.12605	36	
5	5.25:1	72.5	1	36.5	31.24586	36.5	
6	5.25:1	75	0.5	35	30.88136	35	
7	6:1	70	1	39	31.82129	39	
8	6:1	72.5	0.5	39.5	31.93194	39.5	
9	6:1	75	0.75	40	32.0412	40	best



**Fig. 5.16 Graphical Variation of Calorific Value**

The essential factor affecting calorific value is molar ratio and catalyst concentration. From the above graph, it can be observed that calorific value increases at a rapid rate with molar ratio of oil to alcohol ranging from 4.5:1 to 6:1. From the above graph it is understood that calorific value slightly increased as temperature was increased. However we can't increase temperature beyond 80°C as calorific value will decrease in box type solar cooker.

Since KOH is used as catalyst in transesterification reaction and concentration taken was 0.5%, 0.75% and 1% by weight. The calorific value increased from 0.5% to 1%. Less than 0.5% concentration will not be enough to enhance calorific value.

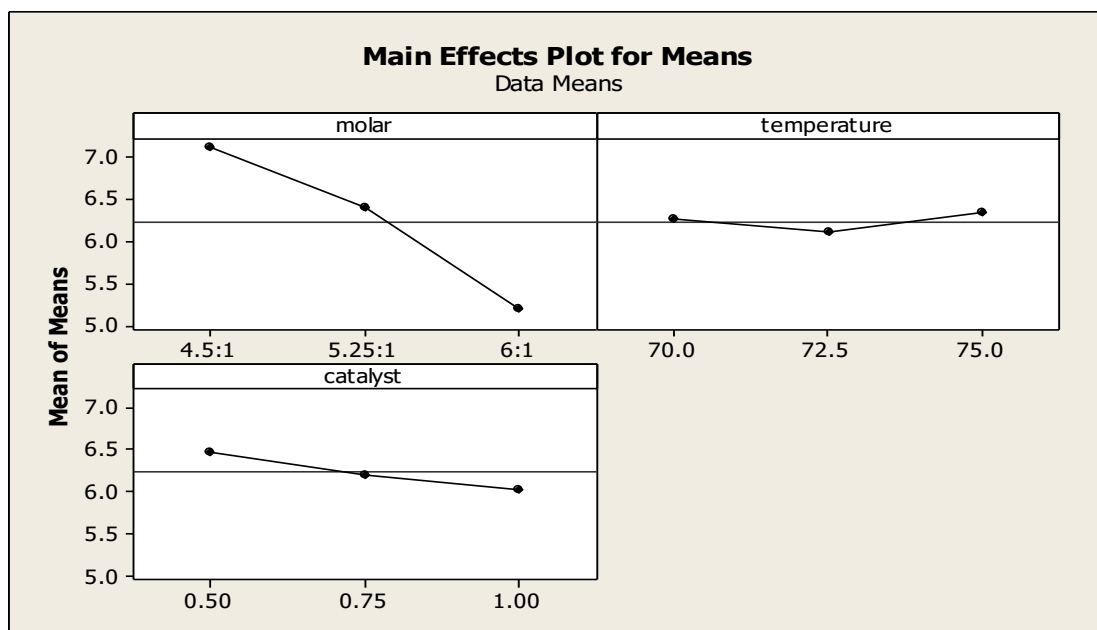


**Fig. 5.17 Signal to Noise Ratio Plot with respect to factors**

**Table 5.10 Analysis of viscosity**

Exp No.	molar ratio	react. tempe	catalyst	viscosity	SNRA1	MEAN1
1	4.5:1	70	0.5	7.2	-17.1466	7.2
2	4.5:1	72.5	0.75	7.1	-17.0252	7.1
3	4.5:1	75	1	7	-16.902	7
4	5.25:1	70	0.75	6.4	-16.1236	6.4
5	5.25:1	72.5	1	5.9	-15.417	5.9
6	5.25:1	75	0.5	6.9	-16.777	6.9
7	6:1	70	1	5.2	-14.3201	5.2
8	6:1	72.5	0.5	5.3	-14.4855	5.3
9	6:1	75	0.75	5.1	-14.1514	5.1

best

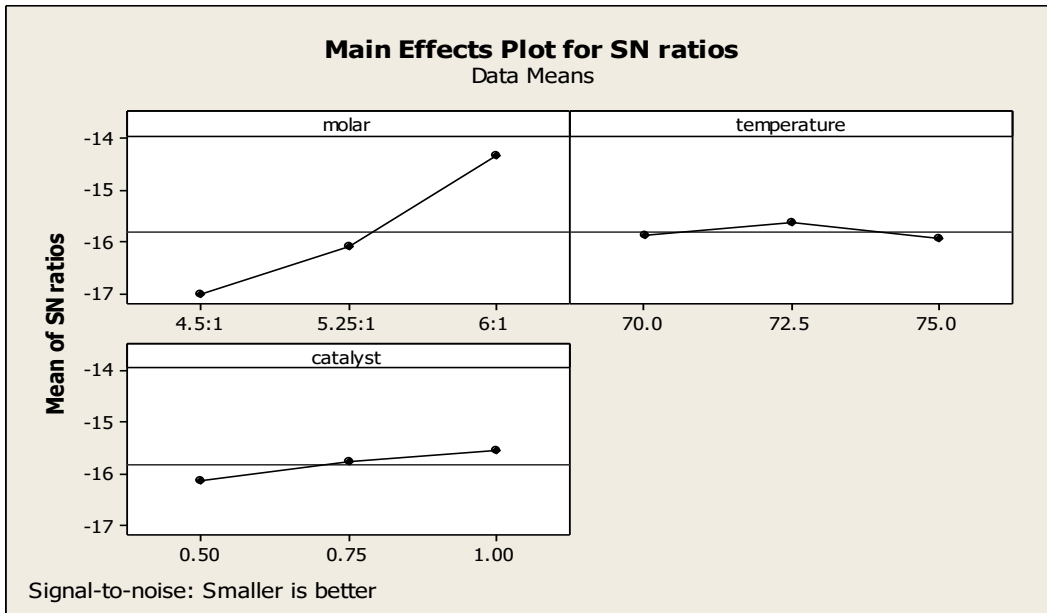


**Fig. 5.18 Graphical Variation of Kinematic Viscosity**

The essential factor affecting kinematic viscosity is catalyst concentration and temperature along with molar ratio. From the above graph, it can be observed that kinematic viscosity decreased with molar ratio of oil to alcohol ranging from 4.5:1 to 6:1. From the above graph it is understood that best results obtained was at between 70-80°C However we can't increase temperature beyond 80°C as kinematic viscosity will start increasing.

Since KOH is used as catalyst in transesterification reaction and concentration taken was 0.5%, 0.75% and 1% by weight. The kinematic viscosity decreased from 0.5% to 1%. Less than 0.5% concentration will not be enough to good results for viscosity.



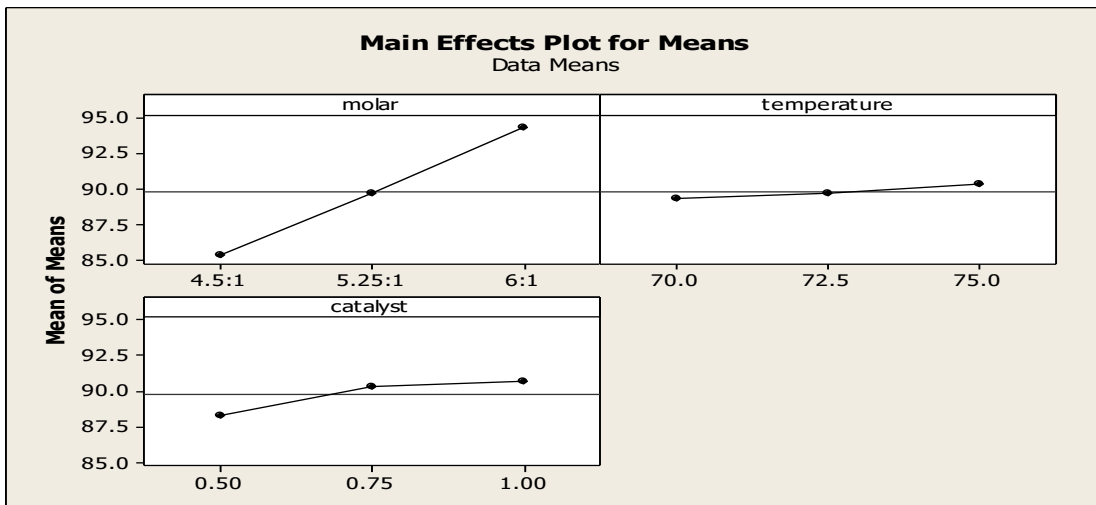


**Fig. 5.19 Signal to Noise Ratio Plot with respect to factors**

**Table 5.11 Analysis for Yield**

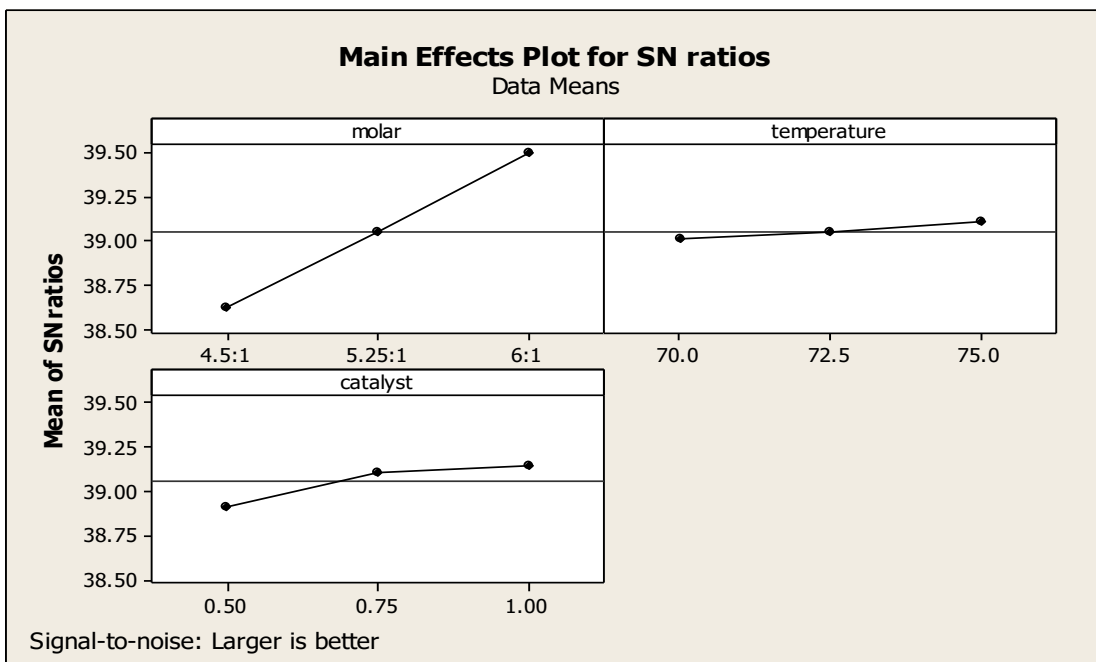
Exp No.	molar	temperature	catalyst	yield	SNRA	MEAN
1	4.5:1	70	0.5	84	38.48559	84
2	4.5:1	72.5	0.75	85	38.58838	85
3	4.5:1	75	1	87	38.79039	87
4	5.25:1	70	0.75	90	39.08485	90
5	5.25:1	72.5	1	91	39.18083	91
6	5.25:1	75	0.5	88	38.88965	88
7	6:1	70	1	94	39.46256	94
8	6:1	72.5	0.5	93	39.36966	93
9	6:1	75	0.75	96	39.64542	96

best



**Fig. 5.20 Graphical Variation of Yield**

From the above graph it is understood that yield increased slightly as temperature was increased. However we can't increase temperature beyond 80°C as yield will decrease. Since KOH is used as catalyst in transesterification reaction and concentration taken was 0.5%, 0.75% and 1% by weight. The yield increased from 0.5% to 1%. Less than 0.5% concentration will not be enough to enhance yield.



**Fig. 5.21 Signal to Noise Ratio Plot with respect to factors**

iii) For Mechanical stirrer method:-

**Table 5.12 Process parameters and their levels**

Parameters/Factors	Level 1	Level 2	Level 3
Molar Ratio	4.5:1	5.25:1	6:01
Reaction Temperature	70	72.5	75
Catalyst Concentration (%)	0.5	0.75	1

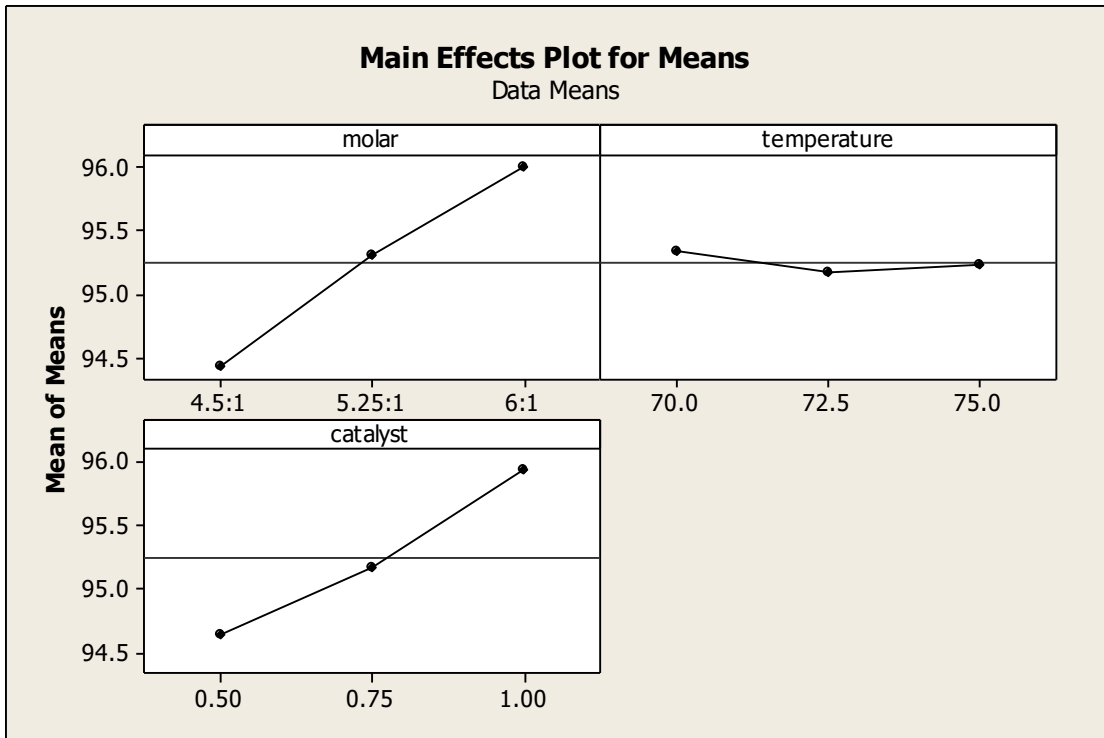
The following table shows the possible nine iterations of experiments as described by L9 orthogonal array along with the corresponding conversions yield of biodiesel found after performing nine experiments-

**Table 5.13 Analysis of yield**

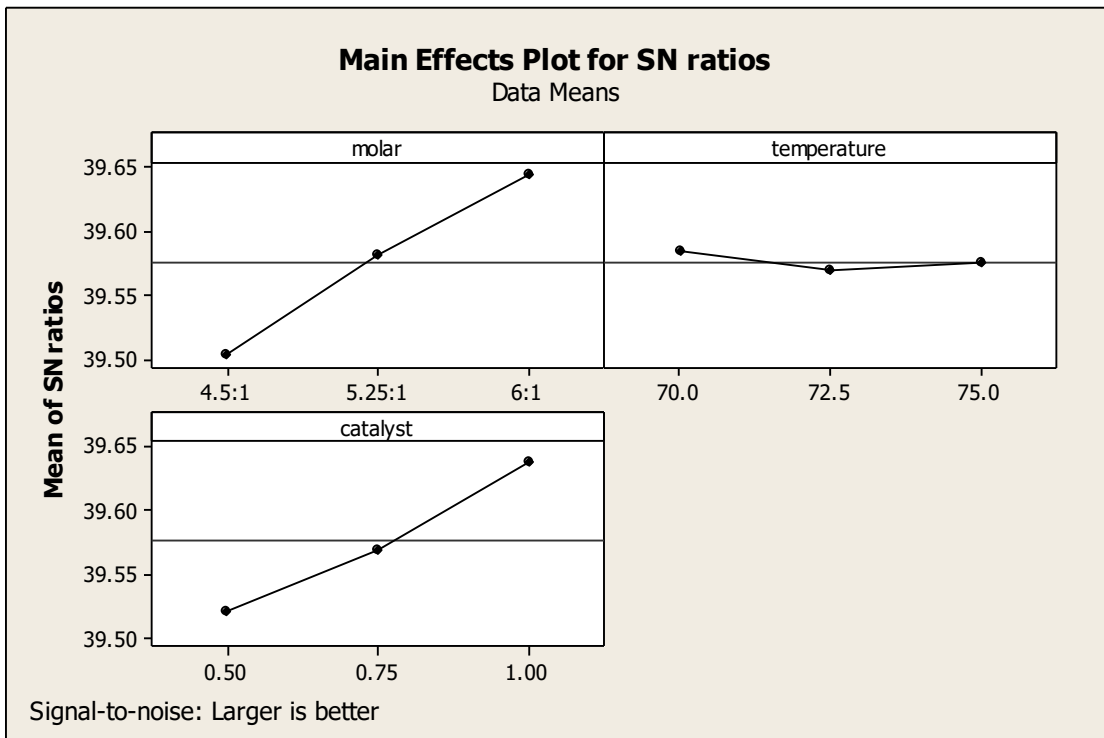
Exp No.	molar	temperature	catalyst	yield	SNRA	MEAN
1	4.5:1	70	0.5	94	39.46256	94
2	4.5:1	72.5	0.75	94.5	39.50864	94.5
3	4.5:1	75	1	94.8	39.53617	94.8
4	5.25:1	70	0.75	95	39.55447	95
5	5.25:1	72.5	1	96	39.64542	96
6	5.25:1	75	0.5	94.9	39.54532	94.9
7	6:1	70	1	97	39.73543	97
8	6:1	72.5	0.5	95	39.55447	95
9	6:1	75	0.75	96	39.64542	96

best

From the above graph it is understood that yield sharply increased as temperature was increased. However we can't increase temperature beyond 70°C as yield will start decreasing. Since KOH is used as catalyst in transesterification reaction and concentration taken was 0.5%, 0.75% and 1% by weight. The yield increased from 0.5% to 1%. Less than 0.5% concentration will not be enough to enhance yield.



**Fig. 5.22 Graphical Variation of Yield**

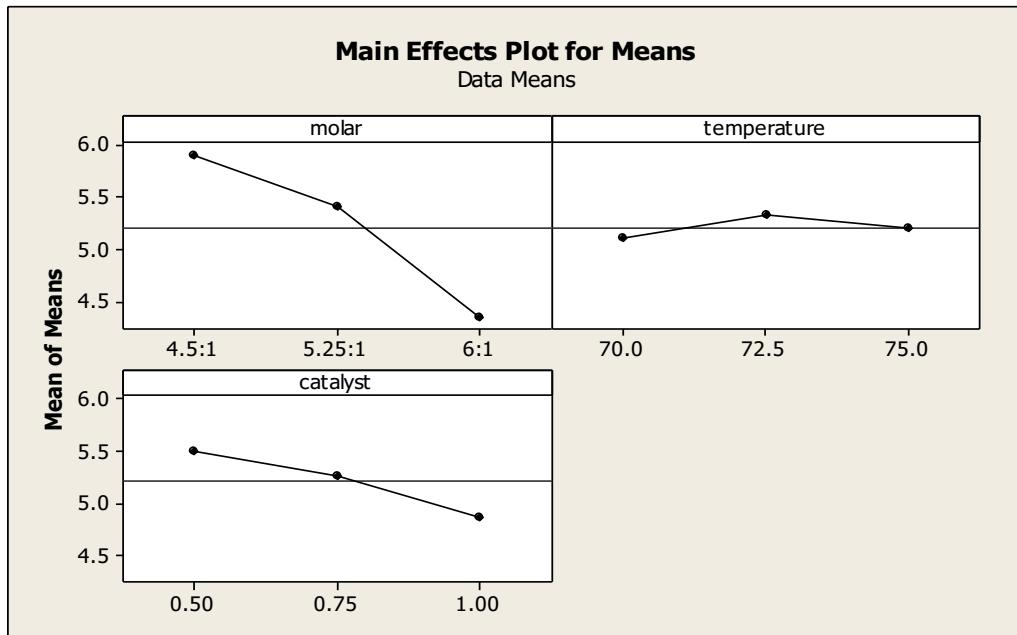


**Fig. 5.23 Signal to Noise Ratio Plot with respect to factors**

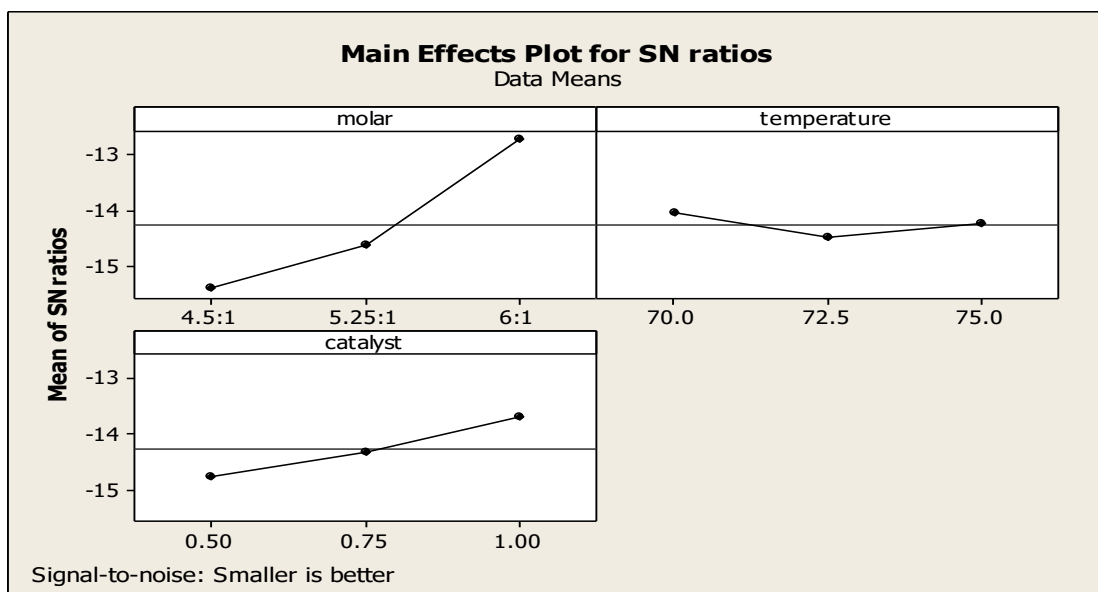
**Table 5.14 Analysis of viscosity**

Exp No.	molar ratio	react. tempe	catalyst	viscosity	SNRA1	MEAN1
1	4.5:1	70	0.5	6	-15.563	6
2	4.5:1	72.5	0.75	6.2	15.8478	6.2
3	4.5:1	75	1	5.48	14.7756	5.48
4	5.25:1	70	0.75	5.3	14.4855	5.3
5	5.25:1	72.5	1	5.1	14.1514	5.1
6	5.25:1	75	0.5	5.8	15.2686	5.8
7	6:1	70	1	4.03	12.1061	4.03
8	6:1	72.5	0.5	4.7	-13.442	4.7
9	6:1	75	0.75	4.3	12.6694	4.3

best



**Fig. 5.24 Graphical Variation of Viscosity**



**Fig. 5.25 Signal to Noise Ratio Plot with respect to factors**

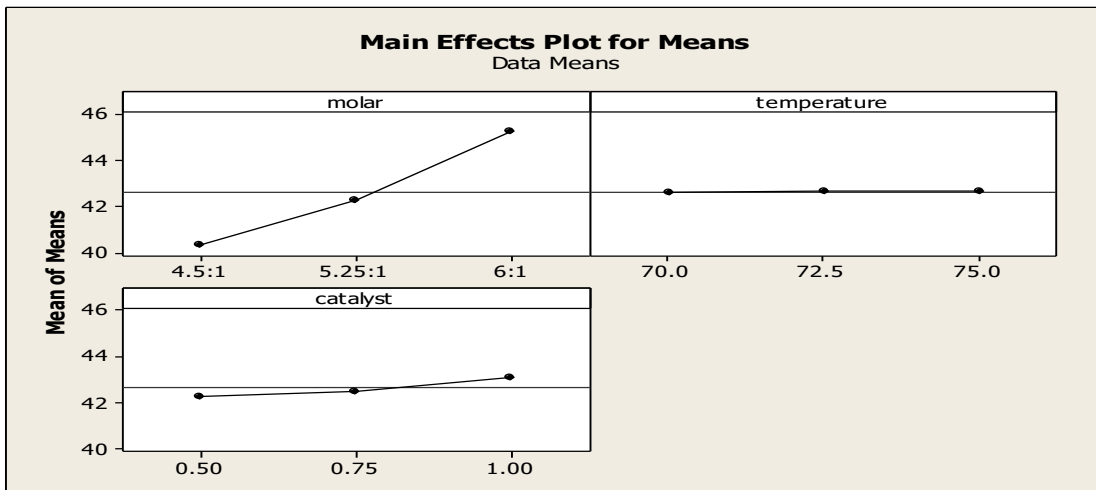
The essential factor affecting kinematic viscosity is catalyst concentration and temperature along with molar ratio. From the above graph, it can be observed that kinematic viscosity sharply decreased with molar ratio of oil to alcohol ranging from 4.5:1 to 6:1. From the above graph it is understood that best results obtained was at between 70 -75°C. However we can't increase temperature beyond 75°C as kinematic viscosity will start increasing.

Since KOH is used as catalyst in transesterification reaction and concentration taken was 0.5%, 0.75% and 1% by weight. The kinematic viscosity decreased from 0.5% to 1%. Less than 0.5% concentration will not be enough to good results for viscosity.

**Table 5.15 Analysis of calorific value**

Exp No.	molar	temperature	catalyst	Calorific value	SNRA1	MEAN1
1	4.5:1	70	0.5	40	32.0412	40
2	4.5:1	72.5	0.75	40.2	32.08452	40.2
3	4.5:1	75	1	40.9	32.23447	40.9
4	5.25:1	70	0.75	42.2	32.50625	42.2
5	5.25:1	72.5	1	42.8	32.62888	42.8
6	5.25:1	75	0.5	41.9	32.44428	41.9
7	6:1	70	1	45.6	33.1793	45.6
8	6:1	72.5	0.5	45	33.06425	45
9	6:1	75	0.75	45.2	33.10277	45.2

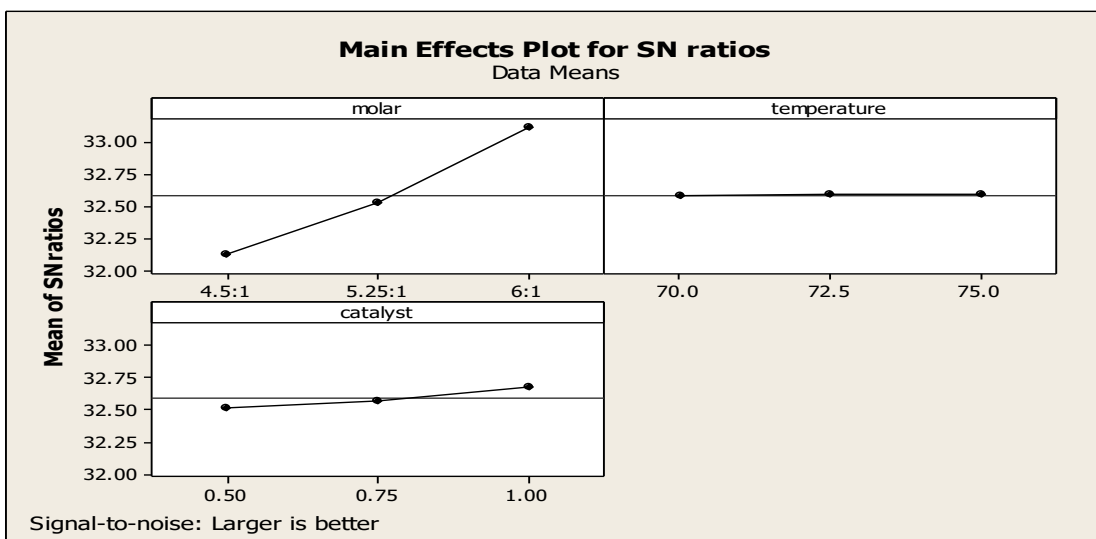
best



**Fig. 5.26 Graphical Variation of Calorific Value**

The essential factor affecting calorific value is molar ratio and catalyst concentration. From the above graph, it can be observed that calorific value increases with molar ratio of oil to alcohol ranging from 4.5:1 to 6:1. From the above graph it is understood that calorific value slightly increased as temperature was increased. However we can't increase temperature beyond 75°C as calorific value will decrease.

Since KOH is used as catalyst in transesterification reaction and concentration taken was 0.5%, 0.75% and 1% by weight. The caloric value increased from 0.5% to 1%. Less than 0.5% concentration will not be enough to enhance calorific value.



**Fig. 5.27 Signal to Noise Ratio Plot with respect to factors**

#### 5.4 Comparison of yield by the methods used for synthesizing biodiesel:-

The graph below best expresses the yield obtained by various methods used for harnessing biodiesel for a 50gram sample of sesame oil:-

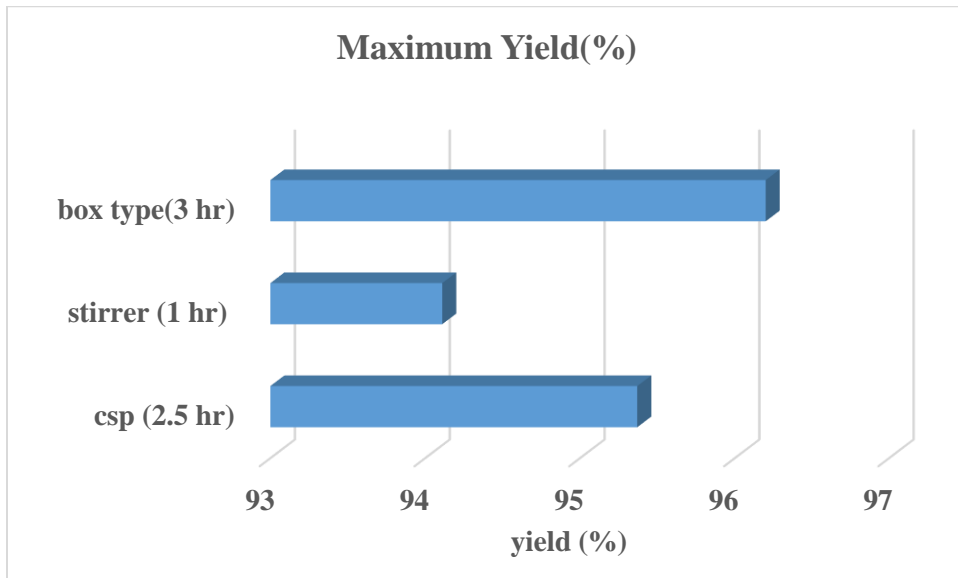


Fig. 5.28 Comparison of yield by different methods

It can be clearly seen from the graph that box type solar cooker gave the best result for yield.



## **CHAPTER-5**

### **CONCLUSION AND FUTURE SCOPE**

In this dissertation, experimental analysis of biodiesel produced from solar technology has been done. The conclusion came out to be that use of solar heating technology during transesterification process is very good option when compared to traditional mechanical method which utilizes electricity for heating purpose. Moreover, solar technology is more cost effective to make it more efficient tracker and temperature sensors are must so we can maintain 80-90<sup>0</sup>C easily without physical presence of a person involved. During use of solar technology, use of mechanical stirrer was eliminated. Continuous stirring is not needed while carrying out transesterification process. Moreover, the conversion yield of biodiesel production obtained was greater.

There is need to focus on improving the process of transesterification. Few points are very essential while carrying out the process and should be taken care of, they are:- measurement of solar radiation data, proper calibration of wind speed was not possible and for temperature readings everytime one has to open the container for thermometer insertion. For carrying out the transesterification, intensity of solar radiation should be more than 400W/m<sup>2</sup>.

## CHAPTER -7

### REFERENCES

1. [http://mediaindia.eu/energy/conventional-energy-sources-dominating-the-power-mix/]
2. [https://www.ibef.org/industry/power-sector-india.aspx]
3. [https://en.wikipedia.org/wiki/Electricity\_sector\_in\_India]
4. [https://en.wikipedia.org/wiki/Sesame\\_oil](https://en.wikipedia.org/wiki/Sesame_oil)
5. Bajpai D, Tyagi VK. Biodiesel: source, production, composition and properties and its benefits, *Journal of oleo science* 55(10) May (2006) 487-502.
6. M. Chegaar, A. Hamzaoui<sup>1</sup>, A. Namoda, P. Petit, M. Aillerie and A. Herguth, Effect of illumination intensity on solar cells parameters, *Energy Procedia* 36 (2013 ) 722 – 729.
7. Dennis Y.C. Leung \*, Xuan Wu, M.K.H. Leung, A review on biodiesel production using catalyzed transesterification, *Applied Energy* 87 (2010) 1083–1095.
8. Svitlana Nitiema-Yefanova , Lucie Coniglio, Raphaël Schneider b, Roger H.C. Nebie, Yvonne L. Bonzi-Coulibaly, Ethyl biodiesel production from non-edible oils of *Balanites aegyptiaca*, *Azadirachta indica*, and *Jatropha curcas* seeds e Laboratory scale Development, *Renewable Energy* 96 (2016) 881e890.
9. Peter Adewale, Marie-Josée Dumont , Michael Ngadi, Recent trends of biodiesel production from animal fat wastes and associated production techniques, *Renewable and Sustainable Energy Reviews* 45(2015)574–588.
10. Idris Atadashi Musa, The effects of alcohol to oil molar ratios and the type of alcohol on biodiesel production using transesterification process: A Review, *Egyptian Journal of Petroleum* (2016) 25, 21–31.
11. Idris Atadashi Musa, The effects of alcohol to oil molar ratios and the type of alcohol on biodiesel production using transesterification process: A Review, *Egyptian Journal of Petroleum* (2016) 25, 21–31.
12. G. Baskar, S. Soumiya, Production of biodiesel from castor oil using iron (II) doped zinc oxide nanocatalyst, *Renewable Energy* 98 (2016) 101e107.

13. J. Kakati , T.K. Gogoi , K. Pakshirajan , Production of biodiesel from Amari (Amoora Wallichii King) tree seeds using optimum process parameters and its characterization, *Energy Conversion and Management* 135 (2017) 281–290.
14. Grisel Corroa, Nallely Sáncheza, Umapada Palb, Surinam Cebadaa, José Luis García Fierroc, Solar-irradiation driven biodiesel production using Cr/SiO<sub>2</sub> photocatalyst exploiting cooperative interaction between Cr<sup>6+</sup> and Cr<sup>3+</sup> moieties, *Applied Catalysis B: Environmental* S0926-3373(16)30768-8.
15. Grisel Corroa, Nallely Sáncheza, Umapada Palb, Surinam Cebadaa, José Luis García Fierroc, Solar-irradiation driven biodiesel production using Cr/SiO<sub>2</sub> photocatalyst exploiting cooperative interaction between Cr<sup>6+</sup> and Cr<sup>3+</sup> moieties, *Applied Catalysis B: Environmental* S0926-3373(16)30768-8.
16. Gajendra Kumar, Vidhi Singha, Dharmendra Kumar, Ultrasonic-assisted continuous methanolysis of *Jatropha curcas* oil in the appearance of biodiesel used as an intermediate solvent, *Ultrasonics - Sonochemistry* 39 (2017) 384–391.
17. Gajendra Kumar, Vidhi Singha, Dharmendra Kumar, Ultrasonic-assisted continuous methanolysis of *Jatropha curcas* oil in the appearance of biodiesel used as an intermediate solvent, *Ultrasonics - Sonochemistry* 39 (2017) 384–391.
18. Gajendra Kumar, Dharmendra Kumar, Rajeev Johari, Time reducing process for biofuel production from non edible oil assisted by ultrasonication, *Ultrasonics Sonochemistry* 21 (2014) 1618–1623.
19. Wilson Wei Sheng Ho, Hoon Kiat Ng, Suyin Gan, Advances in ultrasound-assisted transesterification for biodiesel production, *Applied Thermal Engineering* 100 (2016) 553–563.
20. Peter Adewale, Marie-Josée Dumont, Michael Ngadi, Recent trends of biodiesel production from animal fat wastes and associated production techniques, *Renewable and Sustainable Energy Reviews* 45(2015)574–588.

21. Xiulian Yin, Xuejuan Zhang a, Miaomiao Wana, Xiuli Duan, Qinghong You, Jinfeng Zhang, Songlin Li, Intensification of biodiesel production using dual-frequency counter-current pulsed ultrasound, *Ultrasonics Sonochemistry* 37 (2017) 136–143.
22. Hamed Mootabadi, Babak Salamatinia, Subhash Bhatia, Ahmad Zuhairi Abdullah, Ultrasonic-assisted biodiesel production process from palm oil using alkaline earth metal oxides as the heterogeneous catalysts, *Fuel* 89 (2010) 1818–1825.
23. Mohd Razealy Anuar, Ahmad Zuhairi Abdullah, Ultrasound-assisted biodiesel production from waste cooking oil using hydrotalcite prepared by combustion method as catalyst, *Applied Catalysis A: General* 514 (2016) 214–223.
24. Preeti B. Subhedar, Parag R. Gogate, Ultrasound assisted intensification of biodiesel production using enzymatic inter-esterification, *Ultrasonics Sonochemistry* 29 (2016) 67–75.
25. Preeti B. Subhedar, Parag R. Gogate, Ultrasound assisted intensification of biodiesel production using enzymatic inter-esterification, *Ultrasonics Sonochemistry* 29 (2016) 67–75.
26. Mohammad Mahdi A. Shirazi, Ali Kargari, Meisam Tabatabaei, Boyuk Mostafaeid, Mandana Akia , Mohammad Barkhi, Mohammad Javad A. Shirazi, Acceleration of biodiesel–glycerol decantation through NaCl-assisted gravitational settling: A strategy to economize biodiesel production, *Bioresource Technology* 134 (2013) 401–406.
27. Hamed Mohamadzadeh Shirazi, Javad Karimi-Sabet , Cyrus Ghotbi, Biodiesel production from *Spirulina* microalgae feedstock using direct transesterification near supercritical methanol condition, *Bioresource Technology* 239 (2017) 378–386.
28. J. Sáez-Bastante , C. Ortega-Román , S. Pinzi , F.R. Lara-Raya , D.E. Leiva-Candia, M.P. Dorado, Ultrasound-assisted biodiesel production from *Camelina sativa* oil, *Bioresource Technology* 185 (2015) 116–124.

29. I. Choedkiatsakul, K. Ngaosuwan , G. Cravotto , S. Assabumrungrat, Biodiesel production from palm oil using combined mechanical stirred and ultrasonic reactor, *Ultrasonics Sonochemistry* 21 (2014) 1585–1591.
30. Le Tu Thanh, Kenji Okitsu, Yasuhiro Sadanaga, Norimichi Takenaka, Yasuaki Maeda, Hiroshi Bandow, A two-step continuous ultrasound assisted production of biodiesel fuel from waste cooking oils: A practical and economical approach to produce high quality biodiesel fuel, *Bioresource Technology* 101 (2010) 5394–5401.
31. Le Tu Thanh, Kenji Okitsu, Yasuhiro Sadanaga, Norimichi Takenaka, Yasuaki Maeda, Hiroshi Bandow, Ultrasound-assisted production of biodiesel fuel from vegetable oils in a small scale circulation process, *Bioresource Technology* 101 (2010) 639–645.
32. Leonardo S.G. Teixeira, Júlio C.R. Assis, Daniel R. Mendonça, Iran T.V. Santos, Paulo R.B. Guimarães, Luiz A.M. Pontes, Josanaide S.R. Teixeira, Comparison between conventional and ultrasonic preparation of beef tallow biodiesel, *Fuel Processing Technology* 90 (2009) 1164–1166.
33. D.C. Panadare, V.K. Rathod, Microwave assisted enzymatic synthesis of biodiesel with waste cooking oil and dimethyl carbonate, *Journal of Molecular Catalysis B: Enzymatic* S1381-1177(17)30049-8.
34. D.C. Panadare, V.K. Rathod, Microwave assisted enzymatic synthesis of biodiesel with waste cooking oil and dimethyl carbonate, *Journal of Molecular Catalysis B: Enzymatic* S1381-1177(17)30049-8.
35. Luiz A. Jermolovicius, Luana C.M. Cantagesso, Renata B. do Nascimento, Edmilson R. de Castro, Eduardo V.dos S. Pouzada, José T. Senise, Microwave fast-tracking biodiesel production, *Chemical Engineering and Processing: S0255-2701(16)30209-4*.
36. P.S. Barela, N.A. Silva, J.S.F. Pereira, J.C. Marques, L.F. Rodrigues, D.P. Moraes, [Microwave-assisted digestion using diluted nitric acid for further trace elements determination in biodiesel by SF-ICP-MS, *Fuel* 204 (2017) 85–90.
37. Jo-Han Ng, Swee Kim Leong, Su Shiung Lam, Farid Nasir Ani, Cheng Tung Chong, Microwave-assisted and carbonaceous catalytic pyrolysis of crude

- glycerol from biodiesel waste for energy production, *Energy Conversion and Management* 143 (2017) 399–409.
38. Honglei Zhang, Jincheng Ding, Zengdian Zhao, Microwave assisted esterification of acidified oil from waste cooking oil by CERP/PES catalytic membrane for biodiesel production, *Bioresource Technology* 123 (2012) 72–77.
39. Wei Liu, Ping Yin, Xiguang Liu, Wen Chen, Hou Chen, Chunping Liu, Rongjun Qu, Qiang Xu, Microwave assisted esterification of free fatty acid over a heterogeneous catalyst for biodiesel production, *Energy Conversion and Management* 76 (2013) 1009–1014.
40. Amit Kumar Sharma, Pradeepta Kumar Sahoo, Shailey Singhal, Girdhar Joshi, Exploration of upstream and downstream process for microwave assisted sustainable biodiesel production from microalgae *Chlorella vulgaris*, *Bioresource Technology* 216 (2016) 793–800.
41. [https://www.google.co.in/search?q=sun+temperature&rlz=1C1CHBF\\_enIN743IN743&oq=sun+tempe&aqs=chrome.1.69i57j0l5.5573j0j4&sourceid=chrome&ie=UTF-8](https://www.google.co.in/search?q=sun+temperature&rlz=1C1CHBF_enIN743IN743&oq=sun+tempe&aqs=chrome.1.69i57j0l5.5573j0j4&sourceid=chrome&ie=UTF-8).
42. [https://www.google.co.in/url?sa=t&rct=j&q=&esrc=s&source=web&cd=10&cad=rja&uact=8&sqi=2&ved=0ahUKEwiT9LS69t\\_UAhUIpY8KHadTBoYQFgiCATAJ&url=http%3A%2F%2Fwww.eai.in%2Fclub%2Fusers%2Fkrupali%2Fblogs%2F908&usg=AFQjCNGIu6dfBgA6tbO3d05n5PjwgwoZ9g](https://www.google.co.in/url?sa=t&rct=j&q=&esrc=s&source=web&cd=10&cad=rja&uact=8&sqi=2&ved=0ahUKEwiT9LS69t_UAhUIpY8KHadTBoYQFgiCATAJ&url=http%3A%2F%2Fwww.eai.in%2Fclub%2Fusers%2Fkrupali%2Fblogs%2F908&usg=AFQjCNGIu6dfBgA6tbO3d05n5PjwgwoZ9g).
43. <http://www.synergyenviron.com/tools/solar-irradiance/New+Delhi%252CDelhi%252CInd>