



**Research Article**

## **Biodiesel Production from Pongamia oil using Magnetic Composite of Zinc Oxide Nanocatalyst**

**G.Baskar\*, S. Soumiya, R. Aiswarya**

Department of Biotechnology, St. Joseph's College of Engineering, Chennai – 600 119. India.

\*Corresponding author's e-mail: [basg2004@gmail.com](mailto:basg2004@gmail.com)

### **Abstract**

High petroleum prices demand the need for biofuel production. Lower-cost feedstocks are needed since biodiesel from food-grade oils is not economical. Non-edible oils have been found to be promising crude oils for the production of biodiesel. Pongamia oil is a non-edible oil of Indian origin is chosen for the production of biodiesel in the present study. Transesterification of pongamia oil was carried out using iron (II) doped zinc oxide nanocatalyst. The nanocatalyst was synthesized by co-precipitation method. Using Fourier Transform Infrared Spectroscopy fatty acids of pongamia oil were analyzed. The catalytic activities under various reaction conditions were also evaluated. Transesterification of pongamia oil yielded 93% of biodiesel and was achieved within 50 min at 55°C using 12% (w/w) loading of the synthesized catalyst, methanol to oil molar ratio of 10:1 (v/v), stirring rate of 250 rpm and calcination temperature of 700°C. It was also observed that the nanocatalyst can be recycled up to four times. The produced biodiesel were characterized by gas chromatography and Fourier Transform infrared. Reaction kinetics was also studied for biodiesel production.

**Keywords:** Biodiesel, Iron (II) doped zinc oxide; Pongamia oil, Nanocatalyst; Recyclability.

### **Introduction**

Biomass derived renewable fuels have been gaining paramount importance since the conventional source of energy such as fossil fuel resources, are depleting at a faster rate in recent times[1]. The focus of the human society at present is to obtain energy from low carbon sources and development of eco-friendly green Technology. Biodiesel is an alternative renewable liquid fuel which holds promise to the increase in demand of petroleum diesel [2]. Biodiesel can be produced by transesterification of triglycerides (vegetable oils or animal fats) with a short chain alcohol (mainly methanol). The main asset of this fuel is that its properties and performances are similar to traditional diesel fuels. Biodiesel has not been yet commercialized all over the world [3]. The high cost of raw materials used for biodiesel production, which greatly restricts its widespread application. One way of lowering the cost of biodiesel is to exploit low quality feedstocks such as waste cooking oils, non-edible oils etc [4].

Transesterification reaction can progress with catalytic or non-catalytic mechanisms. Catalytic transesterification can be homogeneous, heterogeneous or enzymatic.

Homogeneous catalysis can be classified as basic, acidic or enzymatic. Homogenous alkali catalysts retain the advantage of providing a fast reaction rate under milder reaction conditions. Some of its drawbacks are production of large quantities of wastewater due to the washing of the catalyst and glycerols are some of its drawbacks [5]. The biodiesel synthesis using solid catalysts instead of homogeneous liquid catalyst could potentially head to lowering of production costs due to reuse of the catalyst. It can also carry out both transesterification and esterification simultaneously. Additional benefit associated with solid heterogeneous catalyst is the lesser consumption of catalyst [6].

Pongamia pinnata is one of the most suitable non edible oil plant species in India because of its good N<sub>2</sub> fixing ability. The seed of pongamia contain 30-40% oil. Pongamia oil is considered as alternative source for production of biodiesel [7]. The main factors affecting transesterification reaction are the amount of the catalyst, alcohol, reaction temperature, reaction time, stirring rate, calcination temperature etc. High catalytic efficiency and ease in separation from products makes nanocatalysts a competitive candidate among all other heterogeneous catalysts [8]. Nanocatalysts comprise of large porous catalytic

surface which increases the contact between alcohol and oil. This leads to an increase in the nanocatalytic effectiveness. High specific surface area of nanomaterials in contrast with the bulk catalysts aids in the contact between catalyst and substrates thus improving the yield of products [9]. Nanoparticles of ZnO comprises of good optical, electrical and chemical properties. Doping with various concentrations of  $Fe^{3+}$  improves electron-hole pair separation and also decreases the size. Doped ZnO synthesized through coprecipitation method has many gains over other production techniques such as ease of processing, control of composition, purity and homogeneity of the produced materials [10].

The present study was aimed at the conversion of pongamia oils to biodiesel via transesterification reaction with iron (II) doped ZnO as heterogeneous catalyst. The variables affecting the methyl ester conversion such as catalyst amount, methanol oil molar ratio, reaction temperature, reaction time, stirring rate and calcination temperature were investigated. The conversion of the resulted biodiesel was analyzed by Gas chromatography and Fourier Transform Infrared Spectroscopy. The kinetics

of the conversion of pongamia oil into biodiesel was also examined.

## Materials and methods

### Materials used

The Pongamia oil was used to carry out the transesterification reaction. Reagents required for the synthesis of catalyst and production of biodiesel are Ferrous Sulphate, Zinc Sulphate, Sodium Hydroxide and Methanol was procured from Chemspure, Chennai, Reachem chemicals, Chennai and Merck, India respectively.

### Feedstock used

Pongamia oil was purchased from local market in Chennai for the production of biodiesel by transesterification reaction.

### Characterization of pongamia oil using FT-IR

Raw oil comprises of a mixture of low and high molecular weight components which is spread over different reaction time in chromatograph with greater molecular mass being detected at higher retention time [11]. The numbers of peaks corresponds to the saturated and unsaturated fatty acids of oil. Depending on the elements and the type of bonds molecular bonds vibrate at various frequencies. The FT-IR spectrum of the pongamia oil is shown in Fig. 1.

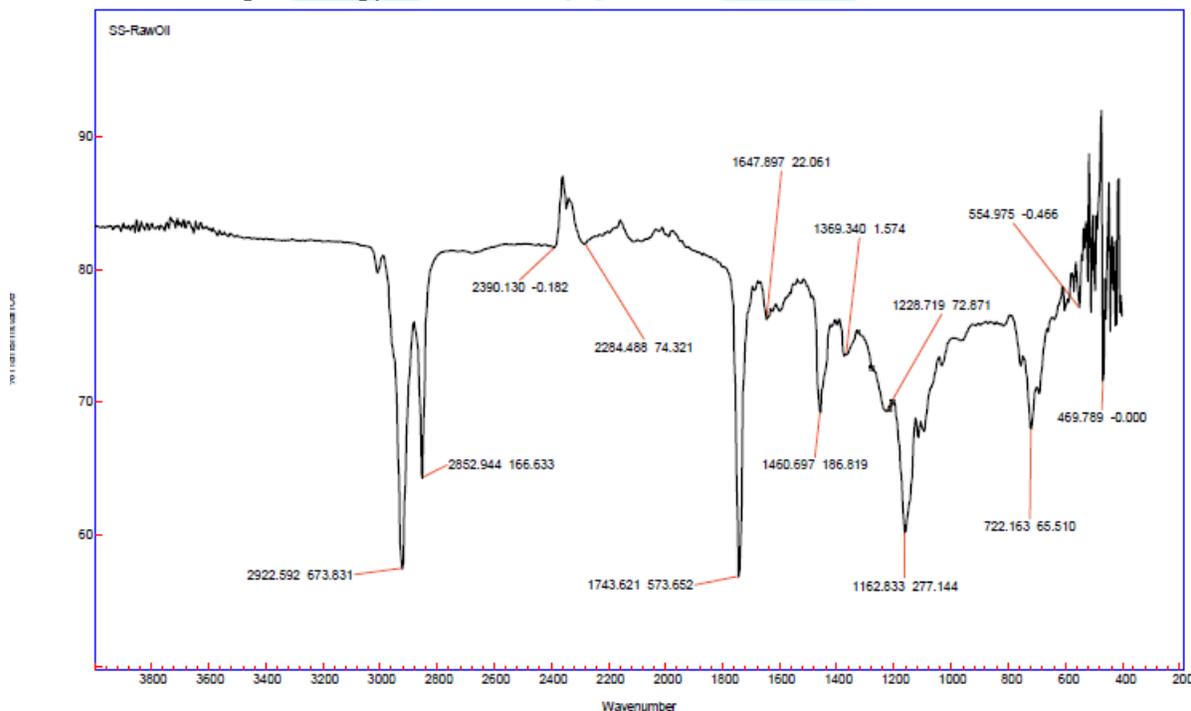


Fig. 1. FT-IR spectrum for pongamia oil

### Synthesis of magnetic composite of zinc oxide nanocatalyst

Iron (II) doped ZnO nanoparticle was synthesized by co-precipitation method. The

requisite amounts of Zinc Sulphate and Ferrous Sulphate was dissolved in distilled water depending on the percentage of Fe doped (Solution-A). Solution-A was then put into an

ultrasonicator at a 57 kHz operating frequency for 2 h. Then, solution A was subsequently stirred with a magnetic stirrer at room temperature. Simultaneously, 0.5M NaOH was prepared in deionized water (Solution-B). Solution-B was added into solution A until the final pH of the solution reached 12 and then the solution was further stirred continuously for 30 min.

The solution was kept at room temperature for 18 h. Then the solution was centrifuged and washed several times with ethanol and distilled water to remove residual and unwanted impurities. The solid product thus obtained was dried in an oven at 200°C for 1h, yielding brown, iron (II) doped ZnO nanoparticles and it was further calcinated at 700°C [12].

### **Transesterification of castor oil**

The transesterification of pongamia oil with methanol was carried out in a 500 ml flask as a reactor equipped with stirrer, temperature measurement and heat plate arrangement. The catalyst was dispersed in methanol under stirring for 5 minutes at room temperature. Quantity of catalyst to be measured depends upon the oil g/g oil. The pongamia oil was then added into the mixture. The reactor was subsequently heated under vigorous stirring at a fixed speed. The requisite temperature was maintained and the set up were left undisturbed depending upon the reaction time. After the desired duration, the mixture was transferred to the separating funnel for the separation of the desired product. The set up were left undisturbed for 2 hr for the separation of liquid phase. The liquid phase was separated between layers, where the upper layer was collected as biodiesel feedstock and the lower layer was separated as glycerol.

The solid catalyst was collected carefully and regenerated. The regenerated catalyst was successively used to study the reusability. The obtained biodiesel was washed and measured. The transesterification reactions were carried out by varying catalyst concentration, O/M ratio, reaction time, temperature, stirring rate and calcination temperature of about 2-14%, 1:5-1:12, 10-60 minute, 40-60°C, 100-300 rpm and 300-800°C respectively. The collected biodiesel products were analyzed using a Gas Chromatography–Mass Spectrometry (GC-MS) to identify the composition and presence of resulted biodiesel.

### **Kinetic studies**

The experiment was carried out at different temperatures 40, 45, 50, 55, and 60°C respectively in order to test for the kinetics of the reaction. The kinetics of the transesterification reaction is represented by equation 1.

$$d[P]/d[t] = k[P] \dots\dots(1)$$

The  $\ln[P]$  versus  $\ln[dP]/dt$  at different interval of time and temperature were plotted. Rate constant were determined from intercept and slope. Arrhenius relationship and the activation energy were studied for the transesterification reaction. The influence of temperature on the specific reaction rate was studied using Arrhenius equation. The activation energy was calculated from the slope of  $1/T$  versus  $(\ln k)$  [13].

## **Results and discussion**

### **Effect of nanocatalyst loading**

Catalyst loading is one of the most important factors that affect the biodiesel yield during the transesterification reaction; therefore it is essential to optimize the catalyst amount for efficient biodiesel production. The biodiesel yield obtained over varying of iron (II) doped zinc oxide nanocatalyst at identified reaction conditions is depicted in Fig. 2. Biodiesel conversion was investigated with 10:1 molar ratio of methanol: oil at a temperature of 55 °C for 50 min, stirring rate of 250 rpm and calcination temperature of 700°C. When the catalyst concentration was increased from 2 to 12%(w/w) the conversion was increased from 27% to 92%. However when the catalyst concentration was increased beyond 12 wt%, there is slight reduction in the conversion. This is due to the mixing problems of the reaction mixture involving reactants, products and solid catalyst [14].

The results showed that biodiesel yield increased as the catalyst loading was increased due to an increase in the number of active sites. Maximum biodiesel yield (92%) was obtained at 12% catalyst loading. This suggested that at 12% catalyst contact between the reactants and solid catalyst was maximum, which directly influenced the forward transesterification reaction speed, thus providing higher biodiesel yield. Therefore, the optimum catalyst amount of 12% was chosen in this study.

### Effect of methanol to oil molar ratio

The effect of methanol to oil molar ratio on biodiesel yield from transesterification reaction of pongamia oil in the presence of iron (II) doped zinc oxide nanocatalyst is illustrated in Fig. 3. The reaction was carried out by varying molar ratios of methanol from 5:1 to 12:1 under the conditions of 12 wt% catalyst, stirring rate of 250 rpm, calcination temperature of 700°C, reaction temperature of 55 °C in 50 min. It was observed that biodiesel yield increased as the amount of methanol was increased, and maximum biodiesel yield of 93% was obtained at methanol to oil molar ratio of 10:1. However, it was noted that beyond the optimum molar ratio, there was no significant hike in the biodiesel yield and remained almost constant.

The maximum biodiesel yield at optimum methanol to oil molar ratio (10:1) was due to the methoxy species formation on the active sites present on the surface of solid heterogeneous catalyst, thus pushing the transesterification reaction in the forward direction to produce maximum biodiesel [15]. Moreover, the results also showed that this optimal methanol concentration provided suitable contact between the reactants and, thereby provided maximum biodiesel yield. In the present work, the optimum methanol to oil molar ratio was found to be 10:1 for the transesterification of pongamia oil.

### Effect of temperature

Due to endothermic nature, transesterification reaction is strongly influenced by the reaction temperature [16]. As the reaction temperature increases collisions among the reactant molecules also increase by gaining kinetic energy, thereby increases the miscibility and mass transfer between the phases [17]. In the present work, the effect of reaction temperature on biodiesel yield at given reaction conditions such as reaction time of 50 min, catalyst loading of 12%, methanol to oil ratio of 10:1, stirring rate of 250 rpm, calcination temperature of 700°C is depicted in Fig. 4. The results revealed that biodiesel yield increased as the temperature was increased and maximum biodiesel yield of 92.7% was obtained at 55°C. This is based on the fact that at high temperature the carbonyl group of triglyceride molecules become more activated thereby favoring the methanol nucleophilic attack on the triglyceride. It forces the transesterification reaction in the forward

direction to produce higher biodiesel yield. The conversion dropped on further increase in temperature 82% at 60°C. The yield tends to decrease after certain temperature due to vapourization of methanol [18].

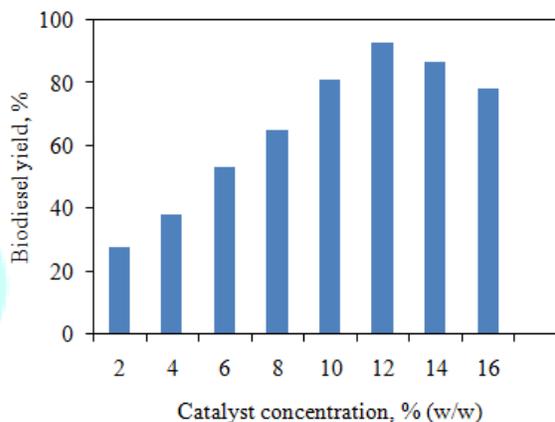


Fig. 2. Effect of catalyst loading on biodiesel yield

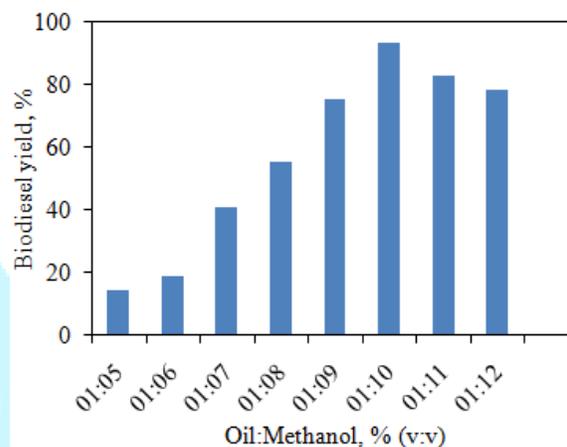


Fig. 3. Effect of oil to methanol ratio on biodiesel yield

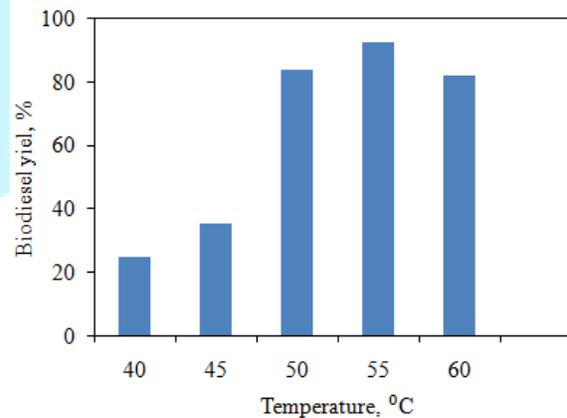


Fig. 4. Effect of temperature on biodiesel yield

### Effect of reaction time

Transesterification reactions were carried out at various periods of time and temperature

between 10 to 60 min and 40-60°C in order to identify the linear relationship between reaction time and temperature. Fig. 5 shows the effect of this factor on the biodiesel yield. The effect of reaction time on conversion was studied at optimized 10:1 molar ratio of methanol to oil, stirring rate of 250 rpm, calcination temperature of 700°C, varying reaction temperature 40-60°C with 12% catalysts loading. The results reveal that the conversion increases with reaction time. The maximum conversion of 92% was obtained in 50 min at 55°C. Biodiesel yield was low at the beginning of the reaction and increases to achieve the maximum yield at 50 min. Then the yield decreases marginally with increasing reaction time. This is in accordance with literature which report that longer reaction time will lead to a reduction in the yield due to the hydrolysis (backward reaction of transesterification), which tends to produce more fatty acids leading to soap formation [19].

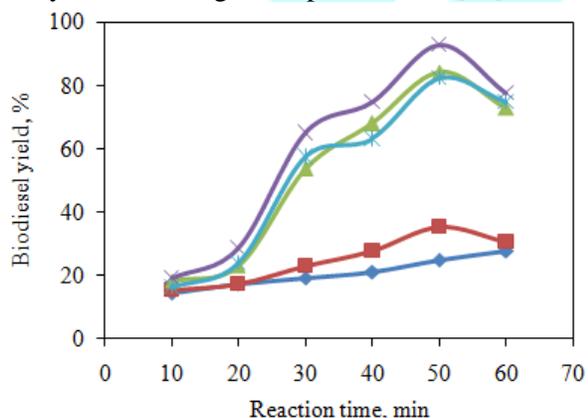


Fig. 5. Effect of reaction time over the temperature on biodiesel yield (♦40C, ■45C, ▲50C, ×55C, \*60C)

#### Effect of stirring rate

The stirring rate is also important reaction variable that affect the biodiesel yield during the transesterification reaction of triglycerides. In the case of heterogeneous catalyst, the reaction mixture displays a three-phase system (oil-methanol-catalyst). Due to the presence of three-phase system in the reaction mixture it will retard the reaction rate due to strong mass transfer limitations [20].

Therefore, it is vital to investigate the influence of stirring rate on biodiesel yield in the transesterification reaction of pongamia oil. The effect of stirring rate on the biodiesel yield, while other parameters were kept at their optimal values, is depicted in Fig. 6. The experiment was

run for 50 min at 55°C with 12% catalyst, 700°C calcination temperature and 10:1 methanol oil molar ratio. The stirring speed was varied from 100 to 300 rpm. The results showed that biodiesel yield increased as the stirring speed was increased and maximum biodiesel yield of 92.7% was achieved at 250 rpm. However, beyond this optimum stirring speed, no significant increase in the biodiesel yield was examined. In the present work, the agitation speed of 250 rpm was used to get maximum biodiesel yield.

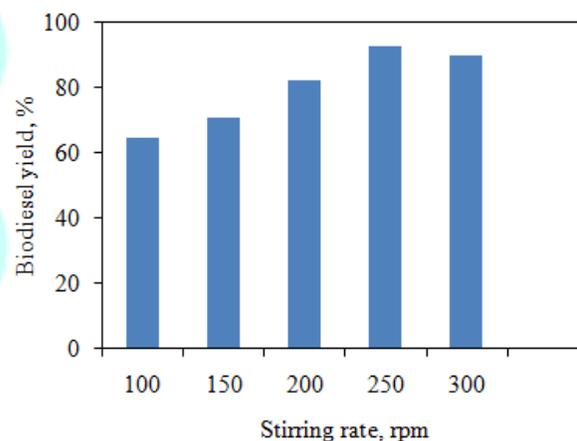


Fig. 6. Effect of stirring rate on biodiesel yield

#### Effect of calcination temperature

Calcination temperature plays an important role on the structural and catalytic properties of the catalysts. Therefore, the effect of calcination temperature of iron (II) doped zinc oxide nanocatalyst catalysts on biodiesel conversion was investigated and was presented in Fig. 7.

The effect of calcination temperature on biodiesel yield was conducted at optimized 10:1 molar ratio of methanol to oil, reaction temperature of 55°C, 12% catalyst loading, stirring rate of 250 rpm and reaction time of 55min. Calcination temperature was varied from 300 to 800°C. At 300°C, lower biodiesel conversion was observed. This is mainly due to the fact that at low calcination temperature ZnO requires a thermal activation to remove the adsorbed CO<sub>2</sub> and moisture [21]. Conversion of biodiesel was increased from 51 to 93% as the calcination temperature increased from 300 to 700°C. Hence, the biodiesel conversion increased with increasing calcination temperature. This is due to the increase in amount of ZnO with increasing calcination temperature which provides important active sites for the transesterification process. However,

the biodiesel conversion started to drop drastically at 800°C.

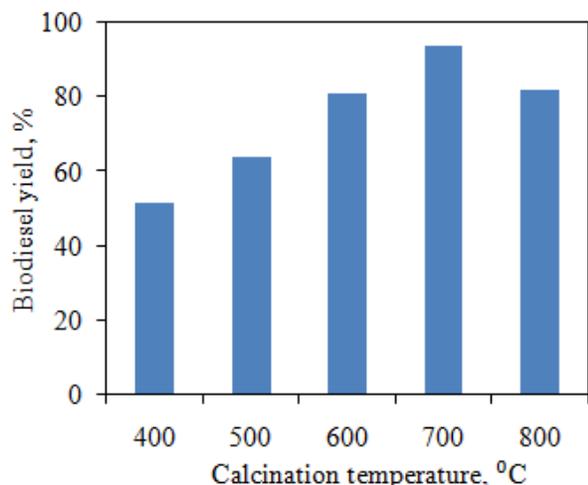


Fig. 7. Effect of Calcination temperature on biodiesel yield

#### Effect of reusability of nanocatalyst

Reusability tests were carried out for the iron (II) doped ZnO nanocatalyst. The catalyst recycling is an crucial step as it minimizes the cost of the process. In order to test the reusability, the iron (II) doped ZnO nanocatalyst was recycled and used for four times. The reusability studies were carried out at reaction temperature of 55°C, stirring speed of 250 rpm, 1:10 molar ratio of oil to alcohol, calcination temperature of 700°C and 12% w/w catalyst loading. The results revealed that the catalyst was reused four times and the yield of biodiesel reduced from 93.6% to 88%, a slight drop in the activity was detected after four recycles indicating the efficiency of the catalyst (Fig. 8). The biodiesel conversion after four cycles tends to decrease at faster rate due to the deactivation of the active sites [22].

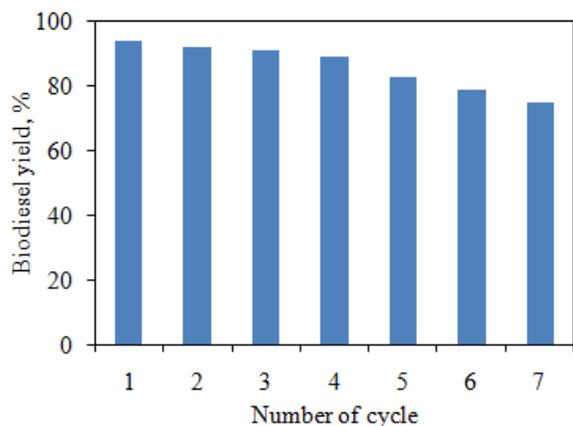


Fig. 8. Effect of reusability on biodiesel yield

#### Kinetic studies

The kinetics of the transesterification reaction is represented by equation 1. Plot of  $\ln[p]$  versus  $\ln(dp/dt)$  at different intervals of time and temperatures with optimum conditions was found to be linear for first-order kinetic model with higher regression coefficient ( $R^2$ ). The yield was also analyzed with respect to the reaction time at temperature ranging from 40 to 60°C. From the data, the biodiesel yield was found to be increased with increase in reaction time. The determination of reaction rate constant explains about the time required to get maximum biodiesel from the pongamia oil. Rate constant was found to increase with rise in temperature. This is due to the increase in the reactivity of the solvent to enhance the rate of extraction. Hence, the reaction rates were found to be dependent strongly on temperature [23].

The Arrhenius relationship and the activation energy were examined for the transesterification process. Activation energy ( $E_a$ ) was calculated based on the Arrhenius equation 2.

$$\ln k = -E_a/RT + C \quad (2)$$

where  $E_a$  is the activation energy,  $R$  is the gas constant ( $J \text{ mol}^{-1} \text{ K}^{-1}$ ),  $T$  is the absolute temperature, and  $C$  is a constant. Hence, the reaction rate is dependent on the temperature and time. The activation energy was calculated from the slope of  $1/T$  versus  $(\ln k)$  and it was found to be 940.31 J/mol.

#### Characterization of the biodiesel by Fourier Transform Infrared Spectroscopy

FT-IR paves way for a feasible solution of all biodiesel components without derivation. It helps in quantifying the various conversions of transesterification reactions. Infrared spectrum of biodiesel reported the presence of pronounced functional groups, which indicated the presence of alkanes and poly-aromatic groups. The existence of these bonds in biodiesel could be determined by identifying characteristic frequencies as absorption bands in the infrared spectrum. Different bonds were present in biodiesel (C-O, C=O, O-H, N-H etc.) with different vibrational frequencies. In the region from 1800-1700  $\text{cm}^{-1}$ , it can be observed peaks that can be attributed to the stretching of C=O, typical of esters [24]. Spectrum on Fig. 9 shows that the bands of C-O (ester function) appear at 1245  $\text{cm}^{-1}$  and the results are in agreement with literature [25].

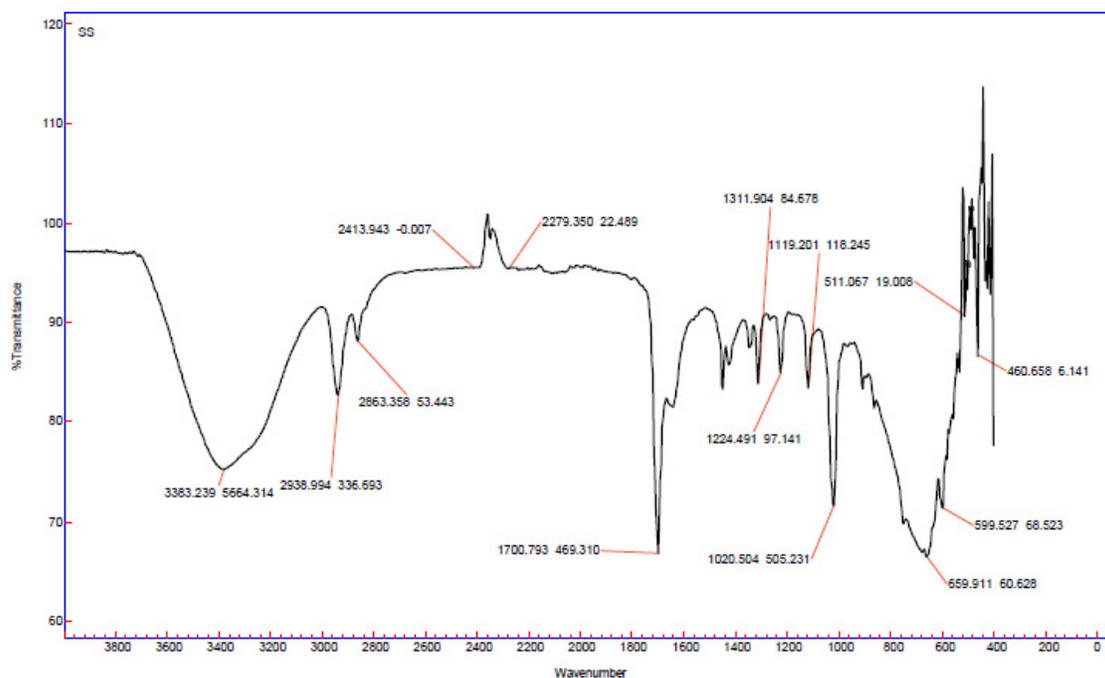


Fig. 9. Characterization of biodiesel by FT-IR

#### Characterization of the biodiesel by Gas Chromatography-Mass Spectroscopy

Characterization of the biodiesel was done by simple chromatographic method. A gas chromatographic method was used in determination of glycerol, mono, di and triglycerides present in the oil and methyl esters. The column used in the analysis are essential where the inertness of the column are required to obtain good peaks and detection limits. The absolute saturated fatty acid methyl esters (C-14-18) are estimated by this method. Internal temperatures along with the hold-up time are important in identification of methyl esters. The retention time along with the mass range are used, as mass spectra are unique to individual compounds. GC-MS was carried out by Agilent 7890B gas chromatograph. The oven temperature was programmed at 35°C with hold for 2 min, it was then ramp at 20°C per minute to 300°C and hold for 5 min. The helium carrier gas was set to 0.5 ml/minute flow rate at constant flow mode.

The injection volume was 1 ml with a split ratio of 20:1. The temperatures of the injector and detector were operated at 300°C. Figure indicates that the different peak represent the presence of methyl esters where the maximum peak represent the existence of Hexadecenoic acid, methyl ester with the retention time at 12.84 min (Fig. 10). The components were found

to be matched with the data bank mass spectra of NIST library provided by the instruments software.

#### Biodiesel properties

The quality of the biodiesel obtained from the transesterification reaction of castor oil and methanol at 55°C with 12% catalyst and 10:1 methanol/oil ratio was evaluated by ASTM methods and compared with standard values, as shown in Table 1.

Table 1. Comparison of properties of ASTM standard with biodiesel produced

Property	ASTM D6751	Present work
Density gm/cc	0.860-0.90	0.829
Kinematic Viscosity	1.9-6.0	1.948

#### Conclusions

The catalytic activity of the solid iron (II) doped ZnO catalyst was evaluated for the production of biodiesel from pongamia oil. The Biodiesel yield reached 93% at the reaction time of 55 min, catalyst of 12%, calcination temperature of 700°C molar ratio of methanol to oil of 10:1, reaction temperature of 55°C and stirring rate of 250 rpm. In addition, the recovery and reutilization of the iron (II) doped ZnO in consecutive catalytic processes suggested that the catalyst can be reused until four times with

slight loss of activity under the optimized condition. The obtained biodiesel was confirmed by GC-MS and FT-IR. The transesterification follows first order reaction kinetics and activation energy was determined as 940.31 J/mol. In addition, the fuel properties of

biodiesel were compared with ASTM Standard D6751 for density and kinematic viscosity and found out to fulfill the requirements. Application of iron (II) doped ZnO nanocatalyst can potentially promote a reduction of the costs related to biodiesel production.

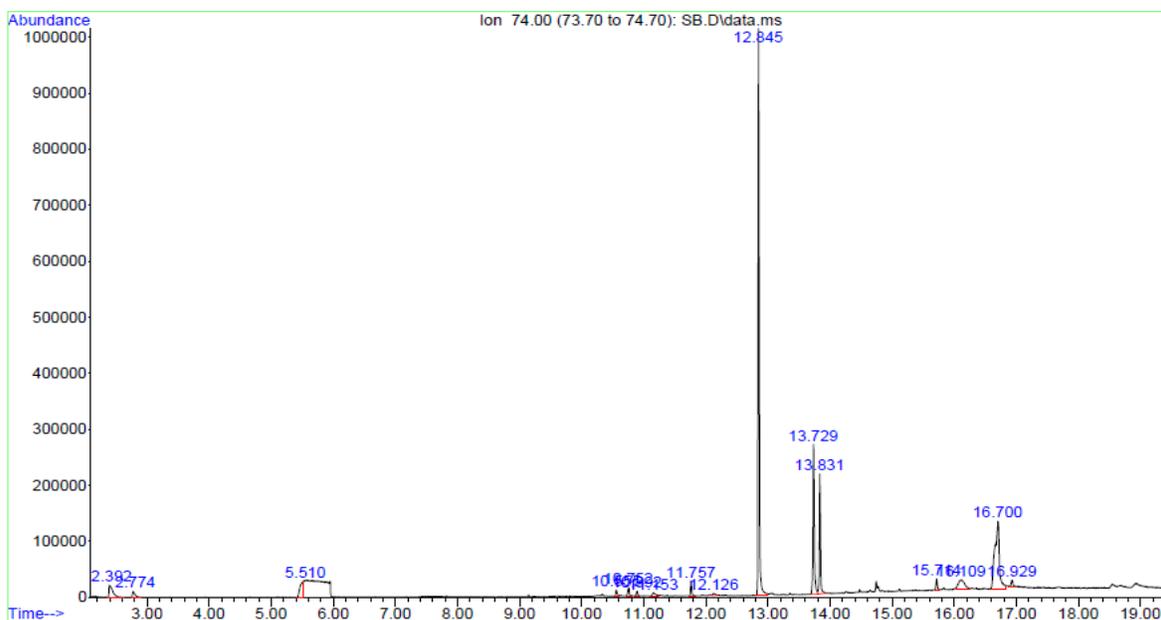


Fig. 10. Characterization of biodiesel by GC-MS

### Conflict of Interest

Authors declare there are no conflicts of interest.

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