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Biodiesel Synthesis with Iron Oxide Nano-Catalyst Catalyzed *Pongamia Pinnata* Seed Oil and Dimethyl Carbonate

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Abstract: The aim of this research was to investigate the biodiesel production from *Pongamia pinnata* seed oil and dimethyl carbonate with an iron oxide nano-catalyzed transesterification reaction. A significant biodiesel yield (96%) was obtained with optimal operating conditions as the dimethyl carbonate to oil molar ratio (5:1), iron oxide nano-catalyst (50 mg% based on oil weight), agitation speed of 150 rpm and 60°C temperature for 5 h reaction time. The produced methyl esters from the transesterification process were confirmed to be almost identical to commercial standard biodiesel by thin layer chromatography. The produced methyl esters were analyzed by gas chromatography-mass spectrometry using an internal standard. Properties of methyl esters were characterized such as kinematic viscosity at 40°C, specific gravity at 25°C, flash point, cloud point, pour point, copper strip corrosion and acid value. The properties of the produced biodiesel were within the specifications of the American biodiesel standard, ASTM D6751-02. The results showed that all of tested reaction variables in this study had positive effects. In this research studied, a novel method has developed for production of biodiesel under mild conditions using DMC and iron oxide nano-catalyst. Iron oxide nano-catalyst could be potential candidate for use in the large-scale biodiesel production.

Keywords: *Pongamia Pinnata* Oil, Iron Oxide Nano-catalyst, Dimethyl Carbonate, TLC, GC-MS

1. Introduction

Biodiesel, as a renewable source of bioenergy, has been produced from animal fats, vegetable oil, used cooking oils and algal lipids [1]. Biodiesel is a potential fuel because of its less polluting nature and lower amounts of carbon smoke [2]. Biodiesel is receiving increased acceptance in the market as an eco-environmentally friendly fuel [3]. Its physical properties are similar to those of petroleum diesel, which makes biodiesel a good alternative fuel [4].

Biodiesel seem to be useful, but its significant disadvantages include the cost of the oil sources and the limited availability of the oil crop for biodiesel production [5, 6]. Therefore, new feedstock suitable for biodiesel production must be found. One alternative to oil crops is the seed oil of *P. pinnata* because this plant contains high

amounts of lipids, which make it suitable for biodiesel production [7]. In India, animal fats, edible and non-edible oil can be used for biodiesel production. *P. pinnata* plants can be grown in wastelands. The use of *P. pinnata* oil can thus likely provide an eco-environmentally acceptable fuel that will lead to reductions in the current diesel engine emissions [8].

Prior research has shown that a highly specific surface, catalysis activity and large porosity are beneficial for nano-catalysts to bind with the sub-layer, which enhances the efficacy of biodiesel production [9-11]. Catalytic technologies are critical to this chemical process [12].

The use of dimethyl carbonate (DMC) instead of methanol as an acyl acceptor in the biodiesel production process can solve the problem of the glycerol surplus, and using DMC produces glycerol-free biodiesel and glycerol carbonate (GC)

as a valuable secondary product [13]. The generation of glycerol is avoided through the reaction of the oil feedstock and DMC to produce GC, which does not require separation from the methyl ester [4, 14]. DMC is a neutral, non-toxic, and non-corrosive compound that exhibits good solvent properties [15, 16].

In this research, an iron oxide nano-catalyst is used in the transesterification reaction using *P. pinnata* seed oil as the feedstock. The effects of the nano-catalyst, reaction temperature, reaction time and DMC-to-oil molar ratio on the yield of biodiesel are examined. The stability of the nano-catalyst and its effects on yield levels are measured and discussed.

2. Materials and Methods

2.1. Materials

Iron oxide, with a 50 nm particle size (BET), was purchased from Sigma-Aldrich Company. Solvents and other analytical reagents were purchased from Merck. The chromatographic standards for fatty acid methyl esters were purchased from Bellefonte, USA. The gases used in the chromatographic analyses were purchased from White Martins. TLC silica gel 60 F_{254} aluminum sheets were purchased from Merck Company.

2.2. Pongamia Pinnata Seed Oil Contents

P. pinnata is a nitrogen-fixing plant with seeds containing 28-36% oil. The molecular composition of *P. pinnata* oil is oleic acid (51.59%), palmitic acid (21.65%), stearic acid (7.5%), linoleic acid (6.64%), eicosanoic acid (1.35%), dosocasnoic acid (4.45%) and tetracosanoic acid (1.09%) [17].

2.3. Transesterification Process of P. Pinnata Oil Using Nano-catalysts

P. pinnata oil was used as a reactant in the biodiesel transesterification process. First the *P. pinnata* oil was boiled at 50°C until a uniform liquid was produced. The reaction was performed in a 1000 mL glass batch reactor equipped with a heat exchanger to condense and return the DMC vapor to the reactor using a constant temperature bath. The molar ratio of *P. pinnata* oil to DMC varied from 1:1 to 6:1. The iron oxide nano-catalyst concentration was 10 to 60 mg% based on oil weight. The reaction temperature was varied from 15 to 75°C for the batch operation under standard conditions with a constant agitation speed of 150 rpm. The reaction was allowed to run for various reaction times, from 1 to 6 h, to study the transesterification process. The sample was allowed to settle by gravity for 10 h after collection with a separation funnel, and it settled into a clear, golden liquid layer of biodiesel on top of a light brown glycerol carbonate layer. The glycerol carbonate was drained from the bottom of the separation funnel, and the remaining crude biodiesel was washed four times with Millipore water to remove the catalyst. To ensure the high purity of the biodiesel, a rotary evaporator was employed to remove excess DMC from the biodiesel. The high-purity biodiesel was dried with anhydrous sodium sulfate and then analyzed by TLC and gas chromatography-mass spectrometry (GC-MS), and the percentage of biodiesel yield was calculated. The biodiesel properties were determined by standard ASTM D6751-02 methods.

2.4. Overall Reaction of Transesterification of P. Pinnata Oil with DMC

"Figure 1", shows the biodiesel transesterification reaction of oil with DMC [18]. The products of the reaction are biodiesel and glycerol dicarbonate (GDC), which hydrolyzes to produce glycerol carbonate (GLC). The three reactions in the transesterification of triglyceride with DMC are as, the reaction of triglyceride (TG) with DMC to produce biodiesel and fatty acid glycerol carbonate (FAGLC); the further reaction of FAGLC with DMC to produce GDC and biodiesel; and the hydrolysis of GDC in an aqueous environment to produce GLC. GLC and GDC may be useful secondary products.

2.5. Analysis of Biodiesel Used Nano-catalyst by GC-MS

GC-MS has a combination of two different analytical techniques. Gas chromatography (GC) and mass spectrometry (MS) is used to analysis complex organic and biochemical mixture. *P. pinnata* seed oil biodiesel components were analyzed by GC-MS (model GC6890 N gas chromatograph, model MS5973 MSD mass selective detector). Separation was performed in a capillary column (DB-5MS, 30 m × 0.32 mm, 0.25 μm film thickness). The carrier gas was helium with a flow rate of 1.5 mL/min. The column temperature was programmed to increase from 120 to 280°C at a rate of 10°C per min. A sample volume of 0.1 μL in chloroform was injected using split mode with a split ratio of 1:10. The mass spectrometer was set to scan the m/z range from 50 to 550 using the electron impact (EI) ionization mode.

2.6. Properties of Biodiesel Produced from P. Pinnata Oil Using Iron Oxide Nano-catalyst

The biodiesel properties of the produced from *P. pinnata* oil using the iron oxide nano- catalyst were determined according to the ASTM D6751-02 specification. The specific gravity, kinematic viscosity, flash point, cloud point, pour point, copper strip corrosion and acid value were measured following ASTM D5002, ASTM D445, ASTM D93, ASTM D2500, ASTM D97, ASTM D130 and ASTM D664 [19].

3. Result and Discussion

3.1. Iron Oxide Powder and Nano Particles

Iron oxide nanoparticles are metal particles and comes in the size range of 10 − 100 nm (approx.). They are non-toxic in nature. Iron oxide nanoparticles readily form oxides. Iron

nanoparticles possess high magnetic nature, high surface area, electrical and thermal conductivity. They also have excellent dimensional stability. Iron oxide nanoparticles it is even more rapid than the bulk material. This characteristic limits its use to inert environments. "Figure 2", shows the Iron oxide powder (A) and Iron oxide 10- 100nm nanoparticles (B).

3.2. Biodiesel Determination by Thin Layer Chromatography

Thin layer chromatography (TLC) is a rapid analytical technique that allows a qualitative indication of oil and biodiesel content in the mixture. "Figure 3", shows the TLC image for a biodiesel from transesterification reactions with the 50 mg% iron oxide nano-catalyst based on oil weight with optimum reaction conditions. The TLC procedure used a solvent containing hexane, ethyl acetate and glacial acetic acid at a ratio of 90:10:1 (v/v/v), respectively. Biodiesel produced from the *P. pinnata* seed oil using the iron oxide nano-catalyst showed higher vigor than did the oil, as indicated on the TLC plate with a clear phase separating the biodiesel from the feedstock. From the TLC analysis, it was confirmed that 50 mg% based on oil weight was the most suitable iron oxide nano-catalyst concentration for biodiesel production from *P. pinnata* oil. TLC was used to assess the conversion of oil to biodiesel [20] and to explore the use of the dominant fatty acids in safflower, soybean, sunflower, canola, corn and waste sunflower oil as biodiesel feedstock [21].

3.3. Optimization of the Transesterification Reaction

3.3.1. Effect of Iron Oxide Nano-catalyst on Biodiesel Yield

The effect of nano-catalyst loading on the transesterification of biodiesel was investigated with iron oxide nano-catalyst by varying the catalyst concentration from 10 to 60 mg% based on oil weight. The reaction was carried out at 60°C for 5 h with a 5:1 DMC to oil molar ratio. As shown in "Figure 4", when the nano-catalyst amount was increased from 10 to 50 mg% based on oil weight, the corresponding conversion to biodiesel gradually increased and obtained a maximum yield of 96%. Varying the concentration of the nano-catalyst from 10 to 40 mg% based on oil weight did not result in a notable difference in the biodiesel percent yield. The biodiesel yield was found to decrease when the iron oxide nano-catalyst concentration was in excess of 60 mg% based on oil weight. Based on these results, 50 mg% of iron oxide nano-catalyst was optimal for these reaction parameters. Wang et al. [22] synthesized acid-functionalized magnetic nano-particles and demonstrated their use as heterogeneous nano-catalysts for production of biodiesel. The biodiesel production of blend refined oil over novel promoted Cs–Ca/TiO$_2$–SiO$_2$ nano-catalysts was also investigated [23].

3.3.2. Effect of Reaction Temperature on Biodiesel Yield

Increases in temperature increase the kinetic energy of reactant particles, thus increasing the frequency of collision between reactants, which affects the rate of mass transfer between phases [24]. The reaction temperature was varied from 30 to 75°C, with a constant iron oxide nano-catalyst concentration, a DMC to oil molar ratio of 5:1 and an agitation speed of 150 rpm. For the iron oxide nano-catalyst, the biodiesel yield gradually increased as the temperature increased at 60°C, after which it tended to decrease slightly, as shown in "Figure 5". At 60°C, the highest biodiesel yield of 96% was obtained, and the same yield was obtained at 75°C; however, the iron oxide nano-catalyst gave only a 90% yield at 90°C. Thereafter, the yield decrease with temperature was greater. At low temperatures (30 to 45°C), the reaction was not completed, so the yield was small. An increase in reaction temperature distributes energy to overcome the energy barrier for a reaction to proceed [18, 25]. However, at higher reaction temperatures (75 and 90°C), a low biodiesel yield was obtained because the boiling point of DMC has at nearly the same temperature, and DMC may be lost as vapor [26]. Hence, 60°C was the optimum temperature for producing biodiesel from *P. pinnata* oil when using the iron oxide nano-catalyst.

3.3.3. Effect of DMC-to-Oil Molar Ratio on Biodiesel Yield

The reactant molar ratio is an important variable that affects the yield (%) of biodiesel produced by transesterification [27]. The effect of the DMC to oil molar ratio on the biodiesel production is shown in "Figure 6". A biodiesel yield of 96% was obtained by increasing the molar ratio to 5:1. Generally, increasing the amount of alcohol increases the rate of reaction, resulting in a good yield of biodiesel in a short reaction time [28]. A decrease in the yield was observed when the molar ratio was increased beyond 5:1. Since excess DMC causes difficulties in heating, the DMC-to-oil molar ratio was maintained for a better transesterification process. Therefore, a DMC-to-oil molar ratio of 5:1 is suggested to be suitable for biodiesel production from *P. pinnata* oil. An excess of DMC possibly increased the polarity of the reaction suspension, thus developing a reverse reaction and decreasing the percentage of biodiesel yield [29]. Alia and Aziz [30] investigated the optimization of the transesterification reaction between palm oil and DMC with the aim of achieving a higher biodiesel yield.

3.3.4. Effect of Reaction Time on Biodiesel Yield

Balaji Panchal et al. [31] investigated the increases in the transesterification of fatty acid esters with increasing reaction times. The reaction time has an important effect on the biodiesel yield (%) from *P. pinnata* oil. In this experiment, reaction times of 1, 2, 3, 4, 5 and 6 h were used; all other conditions were kept constant. As shown in "Figure 7", the conversion increased with reaction time from 1 to 5 h. The highest biodiesel yield, 96%, was obtained within 5 h, thereafter, biodiesel production slightly decreased. As reported by Patil et al. [32], an extended reaction time may result in the over-heating of the reaction suspension and

solvent losses. Further, a longer reaction time decreases the biodiesel product due to the reversible reaction of transesterification, which causes a loss of methyl esters [33]. On the basis of the above results, the 5 h reaction time was considered as optimum for biodiesel production from *P. pinnata* oil using an iron oxide nano-catalyst, although shortening the reaction time may be more favorable for industrial applications.

3.3.5. GC-MS Analysis of the Biodiesel Produced from P. Pinnata Oil

GC-MS was used to identify the methyl esters in the biodiesel produced from *P. pinnata* seed oil, DMC and an iron oxide nano-catalyst. "Figure 8", shows the GC-MS results for biodiesel produced under optimal conditions. Ten characteristic peaks of methyl esters appeared in the GC-MS fragmentation pattern. The most abundant methyl ester components were 40.13% oleic acid methyl esters; 23.50% eicosenoic acid; 8.80% arachidic acid; 5.48% linoleic acid; 4.98% palmitic acid; 4.32% steric acid; 2.78% palmitoleic acid; 1.98% behenic acid; 0.96% alpha and gamma linoleic acid; and 0.87% docosanoic acid. The total yield of methyl esters was 96%, suggesting high-quality biodiesel production from *P. pinnata* seed oil when using an iron oxide nano-catalyst. Hence, the iron oxide nano-catalyst has suitable for biodiesel production from *P. pinnata* oil and has recommended for industrial production of biodiesel.

3.3.6. Properties of Biodiesel Produced from P. Pinnata Oil Using the Nano-catalyst

Fabbri et al. [34] examined the chemical and physical properties of biodiesel. In the present research, the kinematic viscosity of the produced biodiesel has 4.9 mm^2/s at 40°C, and the specific gravity has 0.82 gm/cm^3 at 25°C. The fuel properties of the biodiesel from *P. pinnata* oil are shown in Table. *P. pinnata* oil biodiesel has a higher pour point and

kinematic viscosity than doe's pure biodiesel [35, 36] and a higher cloud and pour point than doe's diesel fuel [37-39]. The pour and cloud points of the produced biodiesel using an iron oxide nano-catalyst ware 2 and 5, respectively. The acid value of the *P. pinnata* oil using iron oxide nano-catalyst biodiesel produced in the research has 0.30 mgKOH/g. The flash point of the *P. pinnata* oil using iron oxide nano-catalyst was 119°C. Biodiesel has a high flash point, which usually exceeds 150°C, whereas conventional diesel fuel generally has a flash point in the range of 55 to 66°C [40]. The flash point is a measure of the flammability of fuels and an important parameter for assessing hazards during fuel transport and storage [41, 42].

4. Conclusions

We found that this new approach could decrease both the production reaction time and temperature and that using a low iron oxide nano-catalyst concentration provides higher biodiesel yields that can even reach 96%. The production of biodiesel from *P. pinnata* oil with DMC was successfully conducted using an iron oxide nano-catalyst. A DMC to *P. pinnata* oil molar ratio of 5:1 is very effective and economically viable. Based on the experimental results, it is possible to conclude that a 5 h reaction time, 50 mg% iron oxide nano-catalyst based on oil weight, 60°C reaction temperature and 150 rpm agitation speed were the best experimental conditions for achieving the maximum biodiesel yield of 96%. The properties of the resulting biodiesel met the specifications of the biodiesel standard ASTM D6751-02. This biodiesel production process has a relatively low cost because of the *P. pinnata* oil starting material, a low iron oxide nano-catalyst concentration and a short reaction time, so we consider it to be an economical fuel solution.

Appendix

Table 1. Properties of biodiesel using iron oxide nano-catalyst with P.pinnata oil compared with ASTM D6751-02 specification.

| Biodiesel properties	Units	Methods	ASTM Limits	Obtained result
Kinematic viscosity at 40°C	mm^2/sec	D445	1.9-6.0	4.9
Specific gravity at 25°C	gm/cm^3	D4052	Reports	0.82
Pour point	°C	D93	Reports	2
Cloud point	°C	D2500	Reports	5
Flash point	°C	D5950	Reports	119
Copper strip corrison (3hrs at 50°C)	1	D130	Max no.3	1a
Acid values	mg KOH/g	D664	Max 0.50	0.3
Water and sediment% vol. max	% vol.	D2709	0.05	0.009
Carbon residue	wt%	D45309	0	0
Ash content	wt%	D874	Max 0.02	0.007
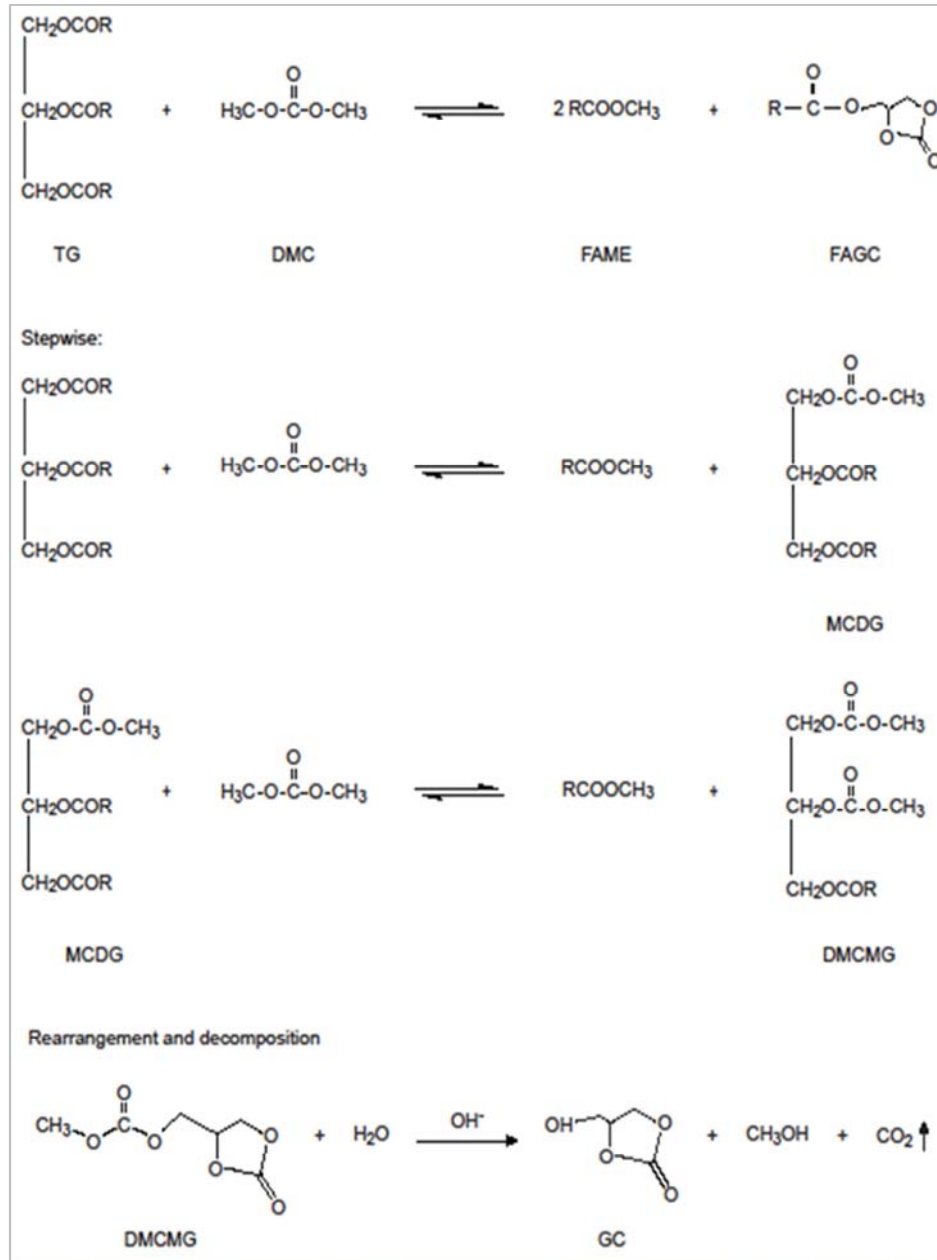
Figure 1. Transesterification reaction of triglyceride with DMC.

(A)
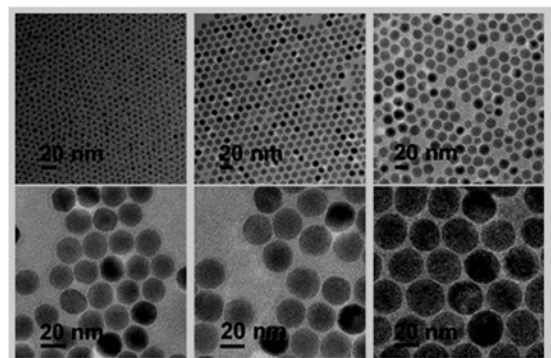
(B)

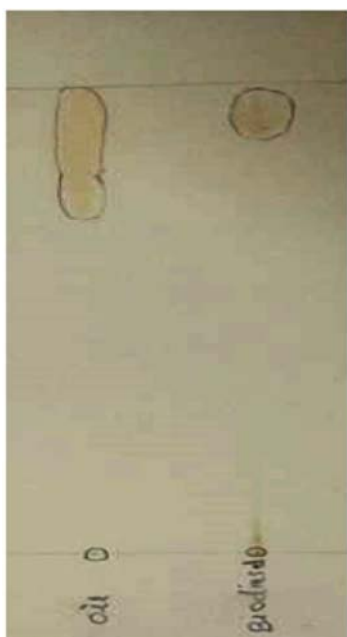*Figure 2. Images of Iron oxide powder (A) and Iron oxide 10- 100nm nano particles (B).*

Figure 3. *Biodiesel analysis by thin layer chromatography. Reaction conditions: 5:1 molar ratio of DMC to P. pinnata oil, 150 rpm agitation speed for 5 h, 50 mg% iron oxide nano-catalyst in P. pinnata oil and reaction temperature of 60°C.*
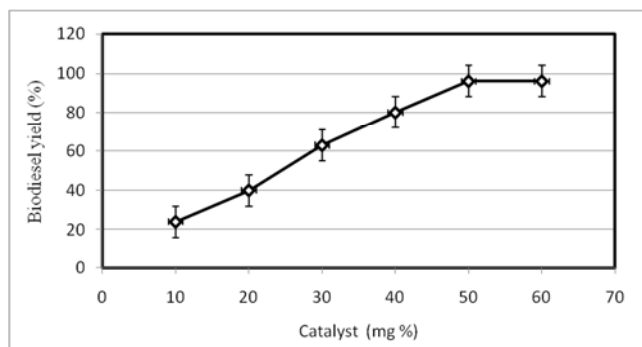

Figure 4. *Effect of iron oxide nano-catalyst concentration on biodiesel yield. Reaction conditions: 5:1 molar ratio of DMC to P.pinnata oil, 150 rpm agitation speed at 60°C for 5 h and various iron oxide nano-catalyst concentrations. Data represent the mean ± standard deviation of triplicate observations.*
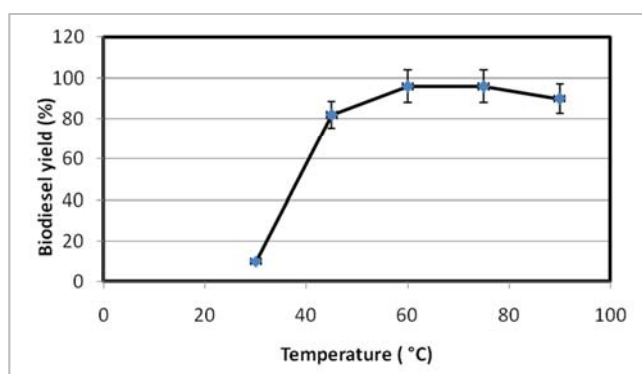

Figure 5. *Effect of reaction temperature on biodiesel yield. Reaction conditions: 5:1 molar ratio of DMC to P. pinnata oil, 150 rpm agitation speed for 5 h, 50 mg% iron oxide nano-catalyst in P. pinnata oil and various reaction temperatures in°C. Data represent the mean ± standard deviation of triplicate observations.*
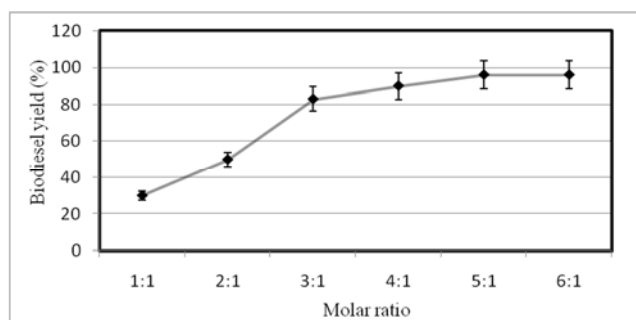

Figure 6. *Effect of the DMC to P. pinnata oil molar ratio on biodiesel yield. Reaction conditions: 50 mg% iron oxide nano-catalyst in P. pinnata oil, 150 rpm agitation speed at 60°C for 5 h and various DMC to P. pinnata oil molar ratios. Data represent the mean ± standard deviation of triplicate observations.*
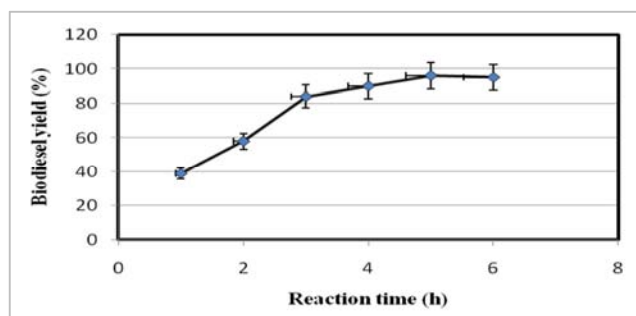

Figure 7. *Effect of reaction time on biodiesel yield. Reaction conditions: 50 mg% iron oxide nano-catalyst in P. pinnata oil, DMC to P. pinnata oil molar ratio, 150 rpm agitation speed at 60°C and various reaction times in hours. Data represent the mean ± standard deviation of triplicate observations.*
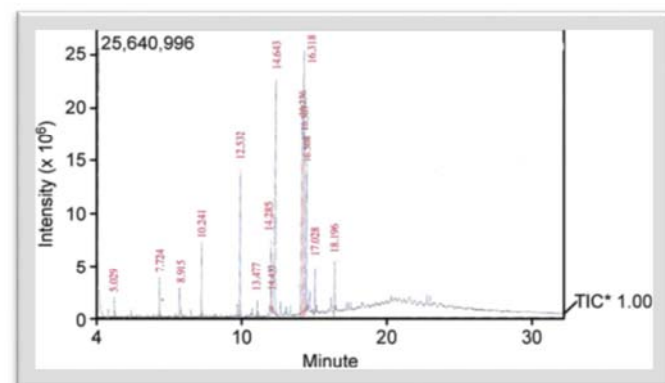

Figure 8. *Chromatogram of biodiesel from P. pinnata oil and DMC using an iron oxide nano-catalyst.*

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