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Biodiesel from *Thevetia peruviana* Seed Oil with Dimethyl Carbonate Using as an Active Catalyst Potassium-Methoxide

(Biodiesel daripada Minyak Biji *Thevetia peruviana* dengan Dimetil Karbonat Digunakan sebagai Pemangkin Aktif Kalium-Metoksid)

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ABSTRACT

The transesterification of Thevetia peruviana *seed oil with dimethyl carbonate (DMC) for preparing biodiesel has been studied using as an active catalyst potassium-methoxide ($KOCH_3$). The effects of reaction conditions: Molar ratio of dimethyl carbonate to* Thevetia peruviana *seed oil, catalyst concentration, reaction time and agitation speed on dimethyl esters (DMC-Tp-BioDs) yield were investigated. The highest DMC-Tp-BioDs yield could reach 97.1% at refluxing temperature for 90 min with molar ratio of DMC-to-oil 5:1 and 2.0% w/w $KOCH_3$ (based on oil weight). The fuel properties of the produced DMC-Tp-BioDs were compared with the ASTM D6751-02 biodiesel standard.*

Keywords: Biodiesel; dimethyl carbonate; oil extraction; optimization conditions; potassium methoxide; Thevetia peruviana *seed oil*

ABSTRAK

Pengesteran trans daripada minyak biji Thevetia peruviana *dengan dimetil karbonat (DMC) untuk menyediakan biodiesel telah dikaji digunakan sebagai suatu pemangkin aktif kalium-metoksid ($KOCH_3$). Kesan tindak balas keadaan: hasil nisbah molar dimetil karbonat minyak biji* Thevetia peruviana, *pemangkin kepekatan, masa reaksi dan kelajuan pergerakan dimetil ester (DMC-Tp-BioDs) dikaji. Kadar hasil tertinggi DMC-Tp-BioDs boleh mencapai 97.1% pada perefluksan suhu untuk 90 min dengan nisbah molar DMC-kepada-minyak 5:1 dan 2.0% w/w $KOCH_3$ (berdasarkan berat minyak). Sifat bahan api yang dihasilkan DMC-Tp-BioDs dibandingkan dengan piawaian ASTM D6751-02 biodiesel.*

Kata kunci: Biodiesel; kalium metoksid; karbonat dimetil; keadaan pengoptimuman; minyak biji Thevetia peruviana*; pengekstrakan minyak*

INTRODUCTION

Biodiesel has attracted much attention during the past decade as a renewable, biodegradable and non-toxic fuel (Bobade & Khyade 2012; Martini & Shell 1998; Singh & Singh 2010; Umer et al. 2008). Biodiesel is an environment friendly fuel, which has almost no sulphur, no aromatics, low emission profile and has about 10% built-in oxygen (Fukuda et al. 2001; Raghunath et al. 2008). It has similar physico-chemical properties of conventional fossil fuel and can consequently, entirely or partially substitute fossil diesel fuel in compression ignition engines (Pasqualinoa et al. 2006).

Thevetia peruviana is a small tree commonly used as an ornamental plant in many tropical countries and belongs to the Apocynaceae family (Tewtrakul et al. 2002). *Thevetia peruviana* grows naturally in India (Neelam & Anil 2014). It is commonly found in the tropics and sub-tropics but it is native to Central and South America (Neelam & Anil 2014). It is available in Nigeria, where it is mainly grown as an ornamental plant (Ibiyemi et al. 2002). The fruits are usually green in color and become black on ripening and the fruit contains between one to four seeds in its kernel (Basumatary & Deka 2014). The plant starts flowering after one and a half year from plantation and thereafter blooms thrice a year (Balusamy et al. 2007). *Thevetia peruviana* seeds have high oil content with major glyceride of palmitric, steric and linoleic acid (Ibiyemi et al. 2002). *Thevetia peruviana* seed oil is non-edible because of the presence of cardiac glycoside (toxins) (Basumatary 2015; Usman et al. 2009).

Dimethyl carbonate (DMC) is a unique molecule having versatile reactivity, non-toxic and biodegradable produced by an environmentally benign process (Tundo & Selva 2002; Wang et al. 2006). It was reported that a methyl ester phase was obtained without glycerol production by using dimethyl carbonate (Fabbri et al. 2007). Glycerol carbonate and glycerol dicarbonate are also formed as byproducts but only in low concentrations (Fabbri et al. 2007).

Molar ratio of alcohol-to-oil, catalyst concentration, purity of reactants as well as reaction time and reaction temperature with agitation speeds are some of the factors that could affect the rate of transesterification and biodiesel yield (Edward & Peggy 2013; Meher et al. 2006). The type and concentration of catalysts employed are critical in transesterification reactions. Alkaline catalysts such as KOH are preferred because of their less corrosive nature and much faster than acid catalysis (Canakci & Gerpen 1999; Edward & Peggy 2013; Ma & Hanna 1999). However, most of the studies showed best properties of biodiesel was obtained by using potassium hydroxide as catalyst (Encinar et al. 2005; Ugheoke et al. 2007). The homogenous alkaline catalyst is commercially used to catalyze the transesterification process (Abbaszaadeh et al. 2012).

The aim of this study was to optimize the biodiesel production using DMC with as an active catalyst $KOCH_3$ as environmentally safe method. The effects of different parameters were investigated. Characterizations of DMC-Tp-BioDs from *Thevetia peruviana* seed oil and compared it with ASTM standards.

Materials and Methods

Collection of *Thevetia peruviana* Seeds and Preparation of Powder

Mature fruits of *Thevetia peruviana* plants were collected from Dr. Babasaheb Ambedkar Marathwada University, Aurangabad (MS), India. *Thevetia peruviana* fruits were cracked and isolated the inside seed kernels. 500 g of *Thevetia peruviana* seed kernels were crushed using a grinder and made up of powder for oil extraction.

Chemicals and Analytical Reagents

All chemicals and reagents were analytical reagent grade. The chemicals and reagents used in this study were potassium hydroxide (chemical society certified, purity 99.99%), n-hexane (chemical society certified, purity 99.97%), dimethyl carbonate (chemical society certified, purity 99.99%), purchased from Spectrochem Pvt. Ltd. Mumbai, India. Pure biodiesel standards (Canola derived-CAS No: 129828-16-6 and Rapeseed derived- CAS No: 73891-99-3) were obtained from Sigma Chemical Company (St. Louis, MO).

Oil Extraction from *Thevetia peruviana* Seed Kernel

100 g of *Thevetia peruviana* seed kernel powder with 100 mL of n-hexane solvent were fed to a 1000 mL round bottomed glass reactor for soaking for 30 min soaking of 30 min some glass beads were added and mixed with n-hexane for 30 min. It is relatively easy to remove from the solids and oil with low energy use. It does appear to have greater ability to extract oil from solid. After mixing, 400 mL of n-hexane was added with 200 rpm stirring speed for 60 min at 55ºC. After 60 min, all supernatant were filtered with Whatman filter paper (no. 42). The solvent was removed at 45ºC under vacuum using a rotary evaporator (Eyela, N-N Series, Rikakikai Co. Ltd., Tokyo, Japan). The percentage weight of extracted oil was calculated.

Experimental Details

Preparation of Potassium- Methoxide Catalyst ($KOCH_3$)

When a chemical reagent potassium hydroxide (KOH) was used as catalysts, powdered KOH reagent was added to the reactant mixtures since the KOH was not dissolved in DMC. In this case, the catalyst was prepared as, KOH was dissolved in methanol as ratio (KOH: Methanol as 1:3 w/w) by stirring at 38ºC Subsequently, DMC was added to the solution. Since $KOCH_3$ is not soluble in DMC, $KOCH_3$ crystallized as more DMC was added to the solution. The fine particles of $KOCH_3$ were suspended in the single phase solution of methanol and DMC. Clear solution of methanol and DMC was formed. Some glass beads were added for control the bumping. After DMC was added, all methanols were removed by vacuum distillation at 38ºC. The pressure was carefully controlled to avoid bumping and was slowly reduced when the pressure was below 100 mbar. The only successful strategy was to remove the methanol from reactants by pressure. After all the methanol was removed, DMC and $KOCH_3$ was mixed (DMC-$KOCH_3$) with *Thevetia peruviana* seed oil and transesterification was performed.

Production of Biodiesel from *Thevetia peruviana* Seed Oil (DMC-Tp-BioDs)

Fresh *Thevetia peruviana* seed extracted oil was used for biodiesel production. The chemical reaction between the *Thevetia peruviana* seed oil and DMC produce biodiesel shown in Figure 1. 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. The reaction temperature was maintained at a prescribed temperature using a constant temperature bath and the reactants were agitated by a stirrer speed as 200 rpm. The molar ratio of DMC-to-oil was varied from 2:1 to 7:1. The molecular weight of the oil was approximated by 874.6 g/mol. The catalyst concentration was varied from 0.5 to 2.5% w/w based on oil weight. Figure 2 shows the mechanism of $KOCH_3$-catalyzed transesterification with dimethyl carbonate.

After the completion of the reaction, the reactants mixture (biodiesel, excess DMC and unreacted oil) was allowed to cool to room temperature and reactants mixture was transferred into separating funnel for separation of biodiesel for 2 h. The top layer including DMC-Tp-BioDs fraction was removed in a separated bottle, purified, weight and analyzed by Gas chromatography-mass spectrometry (GC-MS).

The purification has removed reactant residues and other impurities. For the purification stage, DMC-Tp-BioDs from transesterification process was purified by two different methods. First method was warm water purification using 50% vol. warm water (37°C) that was stirred together with crude DMC-Tp-BioDs. The second was dry purification method using 0.5% wt. magnesium silicate as adsorbent for the impurities in the crude DMC-Tp-BioDs.

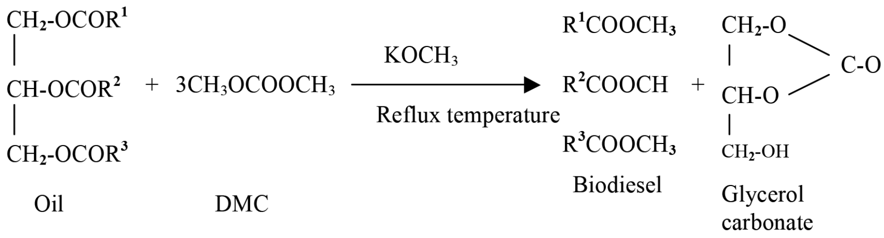

FIGURE 1. The chemical reaction between the *Thevetia peruviana* seed oil and DMC with $KOCH_3$ catalyst at reflux temperature produce biodiesel

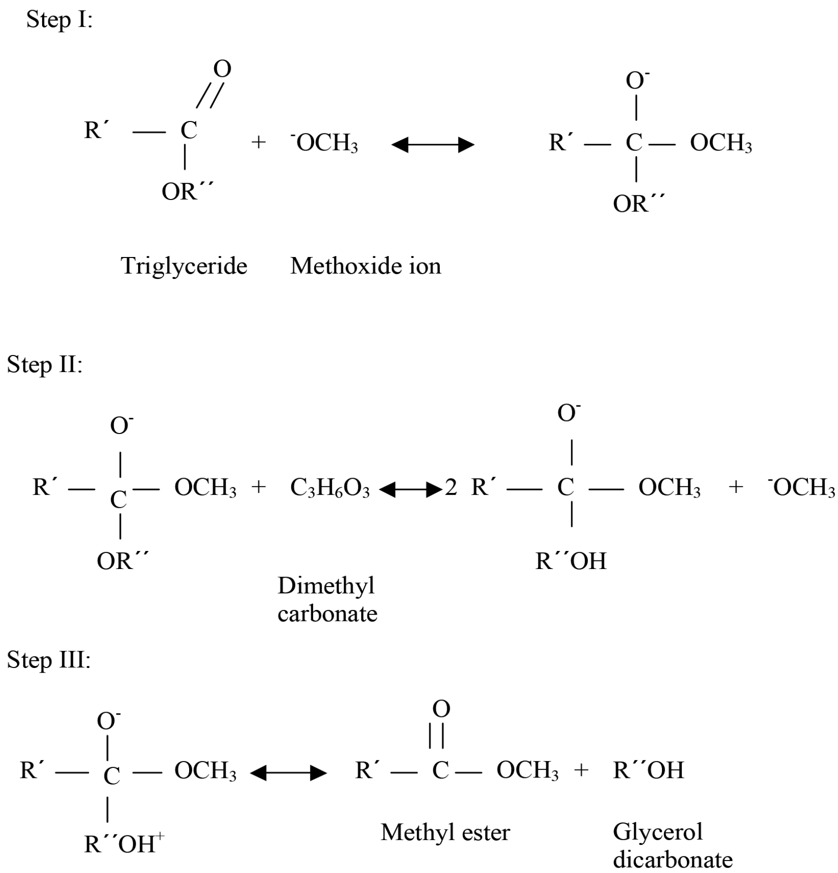

FIGURE 2. Mechanism of potassium methoxide catalyzed transesterification of triglyceride with dimethyl carbonate (where R´ = long chain alkyl group, R´´= glycerol backbone attached to fatty acids)

ANALYSIS OF DMC-TP-BIODS BY GC-MS

The DMC-Tp-BioDs contents were analysed by GC-MS, model GC6890N coupled with mass spectrometer, model MS5973 MSD (mass selective detector). Separation was performed on a capillary column DB-5MS (30m × 0.32 mm, 0.25 μm of film thickness). The carrier gas was helium with flow rate of 1.5 mL/min. The column temperature was programmed from 120-300ºC at the rate of 10ºC/min. A sample volume of 0.1 μL in chloroform was injected using a split mode, with the split ratio of 1:10. The mass spectrometer was set to scan in the range of m/z 50-550 with electron impact (EI) mode of ionization.

DETERMINATION OF DMC-TP-BIODS PROPERTIES

The fuel properties of the DMC-Tp-BioDs produced were determined following ASTM D6751-02. The determinations of density, kinematic viscosity, flash point, cloud point, pour point, copper strip corrosion, ash content, acid value, water content, carbon residue and ester contents were made in accordance with ASTM D 4052, ASTM D 445, ASTM D 93, ASTM D 2500, ASTM D 5950, ASTM D 130, ASTM D 874, ASTM D 664, ASTM D 6304 and ASTM D 4530. All the analysis was performed in triplicate and the data reported.

RESULTS AND DISCUSSION

EXTRACTION OF THEVETIA PERUVIANA *SEED OIL*

52% of *Thevetia peruviana* seed oil was extracted by n-hexane. It was yellowish in color and clear. The acid value of the *Thevetia peruviana* oil was 1.21 mg KOH/g. It was reported that *Thevetia peruviana* seeds contain about 48% of oil (Dhoot et al. 2011). The quantity of crude oil is comparable to the values reported in linseed 40%, cotton seed 24% and groundnut seed 46% (Mohammed & Hamza 2008). This indicated that *Thevetia peruviana* seeds are a good source of oil. *Thevetia peruviana* seeds oil is a large scale mixture of more than 11 substances. Its main part consists of triglycerides (TG) of four fatty acids (FA), namely: palmitic acid, stearic acid, oleic acid and linoleic acid (Ibiyemi et al. 2002).

EFFECTS OF DMC-TO-OIL MOLAR RATIO AND REFLUX TEMPERATURE

The DMC-to-oil molar ratio (2:1, 3:1, 4:1, 5:1, 6:1 and 7:1) was varied in six experiments to decide the effect of DMC amount on DMC-Tp-BioDs production. Molar ratio of DMC-to-oil was approximately 5:1 (DMC/ oil, 0.88 w/w). In all experiments, 200 rpm agitation speed for 90 min, at 86ºC (reflux) reaction temperature and 2.0% w/w $KOCH_3$ concentration were employed. As shown in Figure 3, the most yields (97.1%) of DMC-Tp-BioDs were achieved with a molar ratio of 5:1. The molar ratio of DMC-to-oil as 2:1, 3:1 and 4:1 provided the lowest conversion, as 50.2, 61.5 and 81:1%, respectively. An increase in the ratio of DMC-to-oil as 6:1, the DMC-Tp-BioDs yield was the same as 5:1 molar ratio, even up to 7:1 did not result in higher DMC-Tp-BioDs yields (95.6%). In fact, higher quantities of DMC had a detrimental effect on DMC-Tp-BioDs yield, as indicated by steadily reducing DMC-Tp-BioDs yields when the DMC-to-oil molar ratios increased from 6:1 to 7:1. In additions to that, DMC have high solubility in *Thevetia peruviana* seed extracted oil. DMC-Tp-BioDs yield increased as reaction temperature increased, due to the enhancement of miscibility at high temperature. Other researcher investigated the highest biodiesel yield could reach 96.2% at refluxing temperature for 8 h with molar ratio of DMC and oil 9:1 (Zhang et al. 2010). Several researchers found that the increase in temperature influenced the reaction in a positive manner (Venkanna & Venkataramana 2009). Fabbri et al. (2007) reported 5 h of reaction with sodium methoxide as a catalyst in reacting dimethyl carbonate with soybean oil to produce biodiesel. 6:1 and 7:1 ratios should be avoided for economic reasons; thus, DMC-to-oil molar ratio of 5:1 was selected as being the best.

EFFECT OF CATALYST CONCENTRATION AND REFLUX TEMPERATURE

The effect of catalyst concentration on the conversion of DMC-Tp-BioDs was studied. The experiments were conducted with varying catalyst concentrations, $KOCH_3$ concentration on the oil bases (0.5, 1.0, 1.5, 2.0 and 2.5% w/w). The molar ratio of DMC-to-oil was 5:1 and the reaction temperature was 86°C (reflux) with 200 rpm for 90 min. As shown in Figure 4. The conversion increased with increasing catalyst concentration and the conversion was more, when the catalyst concentration was more than 1.5% w/w. At a catalyst concentration of 2.0% w/w of $KOCH_3$ on oil bases, the DMC-Tp-BioDs were achieved 97.1%. The catalyst concentration of 2.5% w/w of oil provided the same DMC-Tp-BioDs than that of 2.0% w/w of catalyst concentration. The dimethyl ester content increased with higher catalyst concentrations (Panchal et al. 2013). The results showed that the catalyst concentration had a significant effect on alkali-catalyzed methanolysis (Nakpong & Wootthikanokkhan 2010). For these reasons, 2.0% w/w of oil was considered to be the best catalyst concentration.

EFFECT OF REACTION TIME AND REFLUX TEMPERATURE

Freedman et al. (1986) observed that the increase in fatty acid esters conversion when reaction time was increased. In this experiment, reaction times were chosen such as 30, 60, 90 and 120 min. The reactions were carried out by using molar ratio of DMC-to-oil (5:1), 2.0% w/w of $KOCH_3$ on oil bases at 86ºC (reflux) temperature with agitation speed 200 rpm. From the results, 90 min reaction time gave

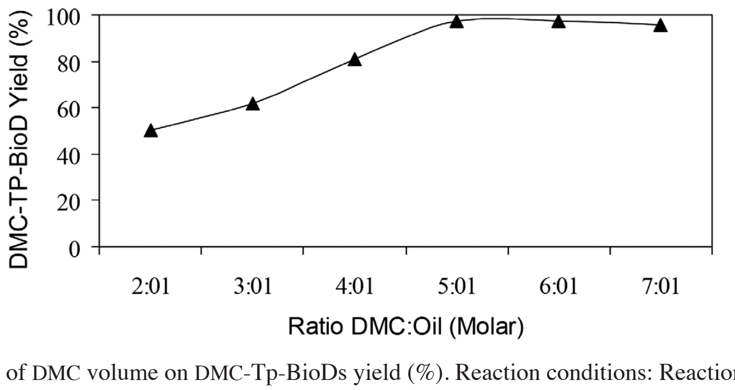

FIGURE 3. Effect of DMC volume on DMC-Tp-BioDs yield (%). Reaction conditions: Reaction temperature at 86ºC for 90 min, catalysts concentration 2.0% w/w, reaction agitation speed as 200 rpm, various volume of DMC- to-oil as 2:1, 3:1, 4:1, 5:1. 6:1 and 7:1 molar ratio

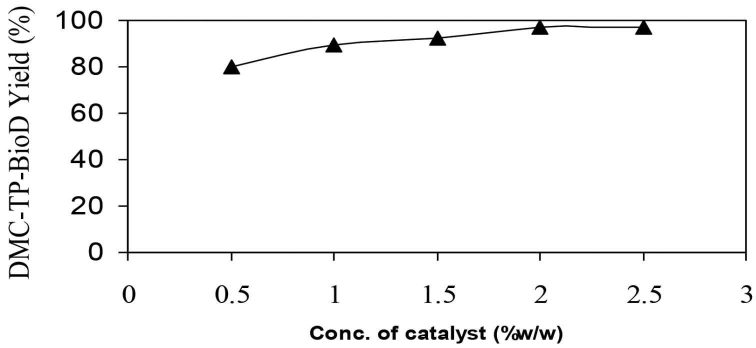

FIGURE 4. Effect of catalyst concentration on DMC-Tp-BioDs yield (%). Reaction conditions: DMC-to-oil molar ratio (5:1), reaction time 90 min at 86ºC, agitation speed as 200 rpm and various catalysts concentrations 0.5, 1.0, 1.5, 2.0 and 2.5% w/w

better DMC-Tp-BioDs yield than 120 min reaction time. As shown in Figure 5, the reaction proceeds faster until maximum yield are reached and then remain constant with any increase in the reaction time. This result showed good agreement with literature (Dennis et al. 2010). Hence, 90 min was chosen as optimum time for the reaction.

EFFECT OF AGITATION SPEED AND REFLUX TEMPERATURE

Panchal et al. (2013) reported that agitation speed was the important reason for biodiesel production. Vigorous mixing was required to increase the area of contact between the two immiscible phases. Effect of agitation speed were studied in the range 50, 100, 150, 200 and 250 rpm, keeping all other conditions same as molar ratio of DMC-to-oil (5:1), 2.0% w/w of $KOCH_3$ on oil bases at 86ºC (reflux) temperature with 90 min. DMC-Tp-BioDs yield was found to increase with increase in the rate of agitation speed up to a point and then decreased. Agitation speed 200 rpm produced the highest percentage of DMC-Tp-BioDs yield (97.1%). However, no further increase in percentage conversion was observed at 250 rpm. As shown in Figure 6, based on these findings, 200 rpm was taken as best agitation speeds for better yield of DMC-Tp-BioDs from *Thevetia peruviana* seed oil.

ANALYSIS OF DMC-TP-BIODS BY GC-MS

GC-MS chromatogram of the DMC-Tp-BioDs obtained from transesterification is presented. In general, the chromatogram displays very good peak shapes and the compounds are very well separated from each other. A total of 12 peaks are observed in the chromatogram, however, some of them could not be identified because their intensities are too low to be considered satisfactory. The compounds identified are presented in Table 1. Identification of these dimethyl esters indicated that transesterification of fatty acids in the *Thevetia peruviana* seed oil took place as expected. The most abundant composition was Hexadecanoic acid DMC-ester with the content of 37%, Dimethyl myristate (32.85%), Heptadecanoic acid (10.53%), Octadecanoic acid (7.87%), Eicosapentaenoic acid (4.99%) and Docosahexaenoic acid (1.43%) were dominating acid DMC-esters and the total content of DMC-Tp-BioDs was 97.1%. This could result in the high quality of DMC-Tp-BioDs produced from *Thevetia peruviana* seeds oil.

PROPERTIES OF DMC-TP-BIODS

The properties of DMC-Tp-BioDs are summarized in Table 2. DMC-Tp-BioDs properties were comparable to those

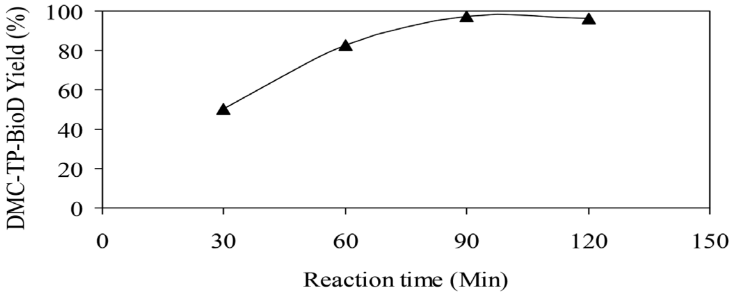

FIGURE 5. Effect of reaction time on DMC-Tp-BioDs yield (%). Reaction conditions: DMC-to-oil molar ratio (5:1), catalysts concentration 2.0% w/w, reaction temperature at 86ºC, agitation speed as 150 rpm and various reaction times 30, 60, 90 and 120 min

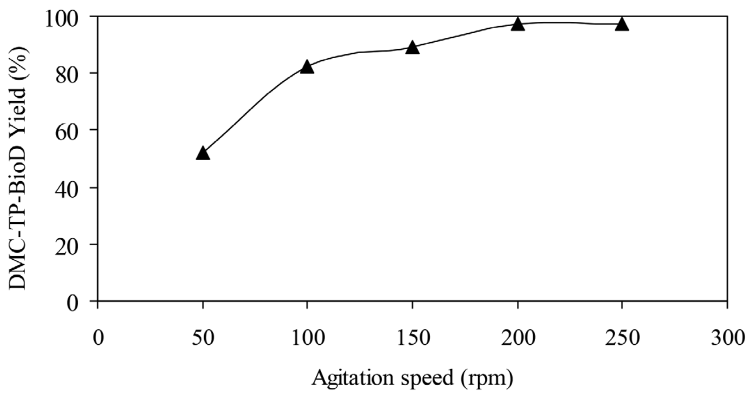

FIGURE 6. Effect of reaction agitation speed on DMC-Tp-BioDs yield (%). Reaction conditions: DMC-to-oil molar ratio (5:1), catalysts concentration 2.0% w/w, reaction temperature at 86ºC with 90 min, various reaction agitation speed as 50, 100, 150, 200 and 250 rpm

TABLE 1. Fatty acid profile of DMC-Tp-BioDs from *Thevetia peruviana* seed oil relative (%)

Retention time (min)	Relative percentage	Fatty acid name	Molecular formula
16.591	32.85	Dimethyl myristate	$C_{15}H_{30}O_2$
18.971	37	Hexadecanoic acid	$C_{17}H_{34}O_2$
20.222	10.53	Heptadecanoic acid	$C_{18}H_{36}O_2$
23.519	7.87	Octadecanoic acod	$C_{19}H_{38}O_2$
28.785	4.99	Eicosapentaenoic acid	$C_{21}H_{32}O_2$
29.739	1.43	Docosahexaenoic acid	$C_{23}H_{34}O_2$

TABLE 2. Measured properties of DMC-Tp-BioDs products in comparison with ASTM D6751-02

Sr. no	Properties	Units	Method	Limits	Results
1	Kinematic viscosity at 40ºC	mm^2/s	D445	1.9 - 6.0	4.45±0.01
2	Density at 25ºC	gm/cm^3	D4052	Reports	0.84±0.01
3	Flash point	ºC	D93	Reports	125±0.02
4	Cloud point	ºC	D2500	Reports	3±0.01
5	Pour point	ºC	D5950	Reports	-2±0.0
6	Copper corrosion strip	Number	D130	3max	1a±0.0
7	Acid value	mg KOH/g	D664	0.50max	0.28±0.01
8	Water content	wt. %	D6304	0.03max	0.02±0.0
9	Ash content	wt. %	D874	0.02max	0.01±0.0
10	Carbone residue	wt. %	D4530	0	0
11	Ester content	wt. %			97.1

of ASTM biodiesel standards. This indicates that biodiesel produced from *Thevetia peruviana* seed oil with DMC can be useful in the engines. Biodiesel with the best properties was obtained using potassium hydroxide as catalyst in many studies (Demirbas 2009; Encinar et al. 2007, 2005; Gupta et al. 2007; Refaat et al. 2008; Tomasevic & Siler-Marinkovic 2003). In the current study, DMC-Tp-BioDs had kinematic viscosity (4.45±0.01 mm^2/s) that met the American biodiesel specification ranges. Kinematic viscosity is a key fuel property because it persuades the atomization of a fuel upon injection into the diesel engine ignition chamber and ultimately, the formation of engine deposits (Knothe & Steidley 2005). The pour point has the DMC-Tp-BioDs as -2±0.0. In general, biodiesel has higher cloud and pour point than diesel fuel (Demirbas 2009). Pour point (PP) is one of the important parameters associated with engine performance in cold weather conditions (Nakpong & Wootthikanokkhan 2010). The flash point of DMC-Tp-BioDs biodiesel has 125°C. Flash point was measured according to ASTM D93 (Fernando et al. 2007). Flash point of *Pongamia pinnata* and *Jatropha curcus* seed oil biodiesel has 122ºC and 127ºC, respectively (Antony et al. 2011; Panchal et al. 2013). Water content of biodiesel reduces the heat of combustion and caused corrosion of vital fuel system components fuel pumps, injector pumps and fuel tubes. Moreover, sediment may consist of suspended rust and dirt particles or it may originate from the fuel as insoluble compounds formed during fuel oxidation (Atabani et al. 2013).

CONCLUSION

The results of the present study demonstrated that the optimum conditions elucidated for the transesterification of *Thevetia peruviana* seed oil were: 5:1 molar ratio of DMC-to-*Thevetia peruviana* seed oil, fixed at 86ºC (reflux) reaction temperature and 2.0% w/w $KOCH_3$ catalyst concentration on oil bases with 200 rpm for 90 min. This process gave over 97.1% conversions to DMC-Tp-BioDs was achieved from *Thevetia peruviana* seed oil. The analyses of GC-MS verified the chemical structural feasibility of DMC-Tp-BioDs as a substitute for fossil diesel. Fuel properties of the DMC-Tp-BioDs have been measured and these met the required standards for fuel use.

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