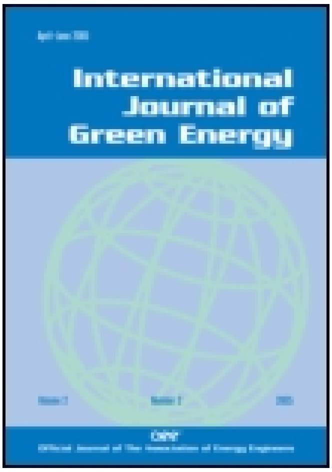

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PRODUCTION OF METHYL ESTER (BIODIESEL) FROM FOUR PLANT SPECIES OF BRASSICACEAE: OPTIMIZATION OF THE TRANSESTERIFICATION PROCESS

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Fatty acid methyl ester (FAME; Biodiesel) of four plant species of Brassicaceae family having more than 30% fixed oil in their seeds were examined. Base catalyzed transesterification using 1:6 molar ratio (oil:methanol) at 70°C was the optimal method to convert crude oil into FAMEs. FAME of oil of these four species, Brassica alba (white mustard), Brassica campestris (mustard), Brassica rapa (canola), and Eruca sativa (rocket seed oil), were found suitable for use as biodiesel and they meet the major specification of biodiesel standards of the United States. The selected plants have great potential for biodiesel use in ordinary diesel engine due to their engine efficiency, consumption, and performance, which is in accordance with mineral diesel.

Keywords: *Fatty acid methyl esters (FAMEs); Transesterification; White mustard; Mustard; Canola; Rocket seed oil; Biodiesel*

INTRODUCTION

The high prices of fossil fuels and their environmental concern to exhaust emissions has led to emergence of biodiesel as an alternative source of energy, which can supplement or replace fossil fuels. Oil seed plants have already been found suitable for the use as an alternative fuel in diesel engines (Srivastava and Verma 2007). This substitution required increased efforts in the research and development of various methods producing these fuels from plant oils and fats. At commercial level, various catalytic processes can be used to convert these seed oils into fatty acid methyl esters (FAMEs; Fukuda, Kondo, and Noda 2001).

FAMEs are characterized by the properties, such as viscosity, flash point, cloud point (CP), pour point (PP), specific gravity, cetane number, etc., to be used in diesel engine. FAMEs as an alternative fuel are environmentally safe and non-toxic (Chang et al. 1996). Various plant oils are in use in different countries for biodiesel production, such as soybean (Freedman, Butterfield, and Pryde 1986; Noureddini and Zhu 1997), sunflower (Antolin et al. 2002; Mohamed and Bornscheuer 2003), palm (Darnoko and Cheryan 2000), rapeseed (Kusdiana and Saka 2001), canola (Zahou, Konar, and Boocock 2003),

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cotton seed (Kose, Tuter, and Aksoy 2002), jatropha (Foidl et al. 1996), pongamia (Ahmad et al. 2009), and sesame (Ahmad et al. 2010). This article includes four rapeseed species, *Brassica alba* (white mustard), *Brassica campestris* (mustard), *Brassica rapa* (canola), and *Eruca sativa* (rocket seed oil), which contain 30% or more oil in their seeds. The free fatty acid (FAA) content of these oil seeds was calculated empirically and found to be less than 3% and can be used to establish their suitability for conversion into FAMEs. Although some studies exist on the conversion of rapeseed oil into FAMEs in Europe (Mcdonnel, Shane, and Mcnulty 1999), while in this work, the specific aim of the study was to compare quantitative and qualitative conversion, yield, byproduct and diesel engine efficiency, consumption and performance by using 100% biodiesel, and different blends. In this work, the production of FAMEs from rapeseed oil has been optimized. Optimum conditions for the transesterification of these plant oils into FAMEs were determined. After getting the optimum conditions, further enhancement of the process was studied in order to standardize the final product (biodiesel) according to American Society for Testing and Materials (ASTM).

MATERIALS AND METHODS

Four operations were studied in the experimental work, namely, (a) seed oil extraction, (b) transesterification, (c) fuel properties' standardization, and (d) engine efficiency. The raw material (rapeseeds) was collected from the cultivated farm of the National Agriculture Research Council (NARC), Pakistan. The oil was extracted from seeds using electric oil expeller (KEK P0015-10127, Germany). After extraction percentage oil contents of various sources of oil seed crops were determined. The extracted oil was filtered to remove the seed residues and other impurities through an electric filter apparatus (W. S. Automa 2). Filtered oil (1000 ml) was subjected to drying by heating at 120°C using hot plate (VWR, VELP—Scientifica, Germany).

The transesterification experiments were performed in 2000 ml beakers using 1000 ml of each rapeseed oil. The method used for the conversion of crude triglycerides into FAMEs (biodiesel) was transesterification as cited in the literature (Bradshaw and Meuly 1944; Allen, Rock, and Kline 1945; Trent 1945; Tanaka, Okabe, and Ando 1981; Freedman, Pryde, and Mounts 1984; Freedman, Butterfield, and Pryde 1986; Schwab, Bagby, and Freedman 1987; Wimmer 1992; Ma, Clements, and Hanna 1998a, 1998b; Ma, Clements, and Hanna 1999; Ahmad, Rashid, et al. 2008; Ahmad, Zafar, et al. 2008). The catalyst (NaOH, 6.5 g) was first dissolved in 200 ml of methanol (Ahmad et al. 2010, 2011). The heating was done in order to remove moisture contents in oil, otherwise every molecule of water will destroy a molecule of catalyst. The dissolved catalyst (sodium methoxide) was added to oil and was stirred vigorously for 20–30 minutes at 60°C. The reaction was arrested in each sample by adding 10–15 drops of acetic acid to avoid contamination. The reaction mixture was poured into a separator funnel. After 1–2 hours, the middle ester layer was poured into separate beaker and byproducts glycerin and soap were also separated. The crude FAMEs were washed in order to remove excess alcohol and residual catalyst by using 2000 ml water; this step was repeated thrice. After third washing, it was kept for 10–30 minutes. During this settling, the water phase containing the unreacted alcohol and catalyst would settle leaving a clear FAMEs phase on top. Finally, the FAMEs were dried with anhydrous sodium sulphate.

FUEL PROPERTIES

The FAMEs were analyzed for dynamic viscosity at 40°C (eta), kinematic viscosity at 40°C (ny), (VI-9730-945), density at 40°C (Rho) (density meter SBS-3500), Flash point (Flash point tester Pensky-Martens Closed Cup BTFT-3), PP °C (FG-K1-400-KW) and CP (CP detector FG-K12663-KW), color, specific gravity at 60°F (kg/l), sulfur contents (%), cetane index, and distillation using ASTM standards (D2500-81 & D97).

ENGINE PERFORMANCE ANALYSIS

The conventional diesel engine can be operated with biodiesel without modification in the engine (Clark et al. 1984). Engine performance of biodiesel and mineral diesel in terms of fuel consumption, efficiency, and power outputs was calculated during road run test.

RESULTS AND DISCUSSION

Determination of Free Fatty Acid Number (FAA)

The FFA content has significant effect on the transesterification of triglycerides with alcohol using catalyst (Goodrum 2002). Methyl esters' yield decreases from 97% to 6% with the increase of the FFA content in oil from 0.3% to 5.3% (Naik et al. 2007). The high FFA content may cause soap formation and makes the separation of products difficult and due to this there will be a significant decrease in FAMEs' yield. Thus, it is important to determine the FFA content of oil before transesterification. The calculated FAA in mustard, white mustard, canola, and rocket seed oils was 1.32%, 1.76%, 1.4%, and 2.54%, respectively. According to Anggraini and Wiederwertung (1999), if the FFA content is more than 3%, then the conversion efficiency decreases gradually.

Biodiesel Yield (%)

In this study, it is found that canola has maximum biodiesel yield (85%) due to its high oil (37%) contents. Crude canola oil color is golden yellow, having a burning taste and gives light pungent smell. Generally, the white mustard seed oil has a long chain of monosaturated fatty acids, erucic acid, oleic acid, and eicosenoic acid. Mustard, canola, and rocket seeds also have similar types of fatty acids like white mustard oil. The common types of fatty acids in mustard and canola were oleic, linoleic, eicosenoic, and erucic acids (Robbelen and Thies 1980). The oil recovery in mustard, white mustard, and rocket oil seeds was 35%, 36%, and 34%, respectively. Biodiesel yield through transesterification experiments revealed that the conversion of crude oil into FAMEs varies in white mustard 82%, mustard 81%, canola 85%, and rocket oil seed 83% (Figure 1). Conversion of crude oil into FAME was considered to be the maximum by using 1:6 molar ratio at 70°C.

Biodiesel Fuel Properties

In this study, it was noted that fuel properties of rapeseed biodiesel and their blends were in accordance with mineral diesel. The comparative flow chart of various fuel properties of four Brassicaceous species was presented in Table 1. Various studies have been conducted elsewhere on biodiesel production, fuel properties, performance, and emission

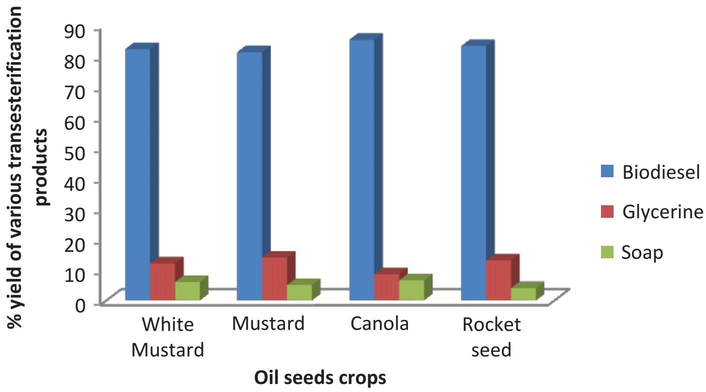

Figure 1 Biodiesel and byproducts' yield (%) (color figure available online).

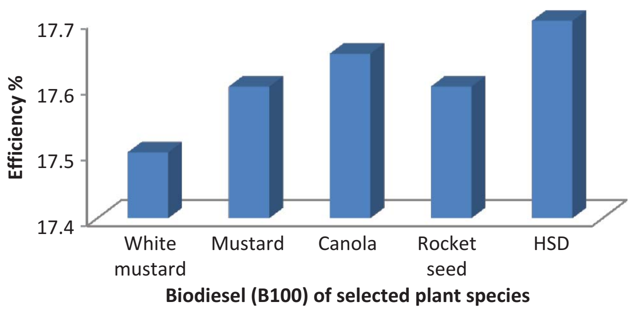

Figure 2 Comparative analysis of biodiesel performance with high speed diesel (color figure available online).

characteristics of diesel engine (Figure 2) (Puhan et al. 2007; Rao et al. 2007; Kabir et al. 2009; Fan, Burton, and Austic 2010; Satyanarayana and Muraleedharn 2010; Özcanlı, Keskin, and Aydın et al. 2011; Subbiah and Gopal 2011).

Viscosity. Viscosity affects the atomization of a fuel upon injection into the combustion chamber and thereby ultimately the formation of engine deposits. The higher the viscosity, the greater is the tendency of the fuel to cause such problems (Dunn and Knothe 2001; Knothe et al. 2001). The viscosity of vegetables oil reduces substantially after esterification. Srivastava and Verma (2007) found that the density and viscosity of vegetables oils after esterification become very close to petroleum diesel oil. The difference in viscosity between the parent oil and the alkyl ester derivatives can be used to monitor biodiesel production (Filippis et al. 1995). The kinematic viscosities of 100% white mustard oil biodiesel (WMOB), mustard oil biodiesel (MOB), canola oil biodiesel (COB), and rocket seed oil biodiesel (RSOB) were 6.719, 6.873, 4.814, and 7.850, respectively, which showed that these are in accordance with ASTM standards.

Density. The density at 40°C of values of WMOB, MOB, COB, RSOB and their blends is presented in Table 1. The respective densities of 100% WMOB, MOB, COB, and RSOB were 0.899, 0.868, 0.870, and 0.877, which is comparable to high speed diesel (HSD) as reported by Peterson et al. (1992) and Encinar et al. (2002).

Flash point. Flash points of WMOB, MOB, COB, RSOB, and HSD are presented in Table 1. The 100% RSOB has the highest flash point (120°C) as compared with the

Table 1 Fuel Properties of WMOB, MOB, COB, and RSOB

Sr. no.	Testing methods	ASTM D975 standards	Diesel (HSD)	Fuel properties of pure biodiesel and various blends															
				WMOB B100	MOB B5	COB B10	RSOB B20	B100	B5	B10	B20	B100	B5	B10	B20	B100	B5	B10	B20
1	Dynamic viscosity at 40°C (eta)	3.2642	2.7870	6.192	4.312	4.26	3.611	5.972	3.347	3.239	3.141	4.191	3.290	3.155	3.100	6.885	3.346	3.503	3.121
2	Kinematic viscosity at 40°C (ny)	1.3–1.6	3.3597	6.719	4.821	4.412	4.221	6.873	4.201	4.105	4.028	4.814	3.935	3.204	3.041	7.850	5.002	4.178	4.012
3	Density at 40°C (Rho)	0.079	0.8295	0.899	0.852	0.843	0.831	0.868	0.836	0.832	0.811	0.870	0.836	0.832	0.822	0.877	0.836	0.811	0.801
4	Color comparison	2	2.0	2.0	1.5	1.8	2.0	2.0	2.0	2.0	2.0	1.0	2.0	2.0	1.5	2.0	2.0	2.0	2.0
5	Flash point, °C	60–80	74	110	90	85	80	80	79	76	74	115	95	90	95	120	100	95	90
6	Pour point, °C	−35 to −15	0	−6	0	0	0	−3	0	0	0	−9	0	0	0	−15	0	0	0
7	Cloud point, °C	−15 to 5	5	3	4	5.5	5	5	4	3	2	5	4	3.5	3	03	2.5	2	2
8	Specific gravity,kg/l at 60°F	0.85	0.847	0.892	0.863	0.842	0.836	0.885	0.853	0.843	0.822	0.886	0.852	0.834	0.826	0.893	0.852	0.821	0.811
9	Sulfur contents, %	0.05	0.5862	0.0130	0.0426	0.052	0.0422	0.01285	0.0519	0.0498	0.042	0.0110	0.0506	0.047	0.0414	0.0393	0.0563	0.0490	0.060

ASTM = American Standard Testing Materials; HSD = high speed diesel; B100 = pure biodiesel; B5 = 5% biodiesel; B10 = 10% biodiesel; B20 = 20% biodiesel; WMOB = white mustard oil biodiesel, MOB = mustard oil biodiesel, COB = canola oil biodiesel, RSOB = rocket seed oil biodiesel.

HSD (74°C). A higher flash point indicates a higher degree of safety for the storage, transportation, and usage of liquid fuel (Hossain and Devies 2010).

Cloud point and pour point. One of the major problems associated with the use of biodiesel is poor low temperature flow property, indicated by relatively high CPs and PPs (Dunn and Knothe 2001; Knothe et al. 2001). CPs and PPs of WMOB, MOB, COB, and RSOB were presented in Table 1, which indicate clearly that their values were comparable to HSD. Lee, Johnson, and Hammond (1995) argued that the CP is affected by the presence of monoglycerides in seed oils while PP remains unaffected.

Sulphur contents. Unlike fossil fuels the sulphur contents of plant oil is very low, which indicates its environmental friendly properties. Moreover, vegetable oils take away more carbon dioxide from the environment during their production than is added to it by their later combustion (Ahmad, Rashid, et al. 2008; Ahmad, Zafar, et al. 2008). The sulphur contents of 100% WMOB, MOB, COB, and RSOB were in traces as compared with HSD (Table 1).

CONCLUSION

In this study, crude oil obtained from four Brassicaceous plant species was successfully converted into FAMEs using base-catalyzed transesterification at 1:6 molar ratio (oil:methanol). The biodiesel produced from them can replace or supplement the petrodiesel more successfully as Indo-Pak Subcontinent has vast areas of unused non-forest land of different kinds like arid, semi-arid, and ravine land. The study may suggest to agriculturists, policy makers, and farmers to project their efforts to cultivate these four potential oil seeds on their unused land as energy crops in order to overcome the energy crises for global interest and perspectives.

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