BRIEF REPORT

Corresponding Properties of Fatty Oils of *Cleome viscosa* and *Jatropha curcas* as Resources of Biodiesel

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Abstract Fatty oils of the seeds of *Cleome viscosa* and *Jatropha curcas* from Aravali range were methylated and analysed by GC and GC–MS. Thirty-four constituents of *C. viscosa* oil and 26 constituents of *J. curcas* oil were detected. *C. viscosa* and *J. curcas* oils had 22.0 % and 25.8 % saturated, 21.6 % and 42.2 % monounsaturated, and 53.8 % and 31.3 % polyunsaturated fatty acids, respectively. Major fatty acids of these plants were identified as palmitic acid, stearic acid, oleic acid, and linoleic acid. In addition, some minor and trace constituents and alkanes were also identified.

Keywords C. viscosa · J. curcas · Fatty oil · Fatty acid

Introduction

Due to rapid depletion of fossil fuels, there is a global awareness on the search for alternative sources of energy. The consumption of petroleum products in India has been steadily increasing and their prices are rising. Approximately, 40 % of world energy comes from petroleum products and 20 % from natural gas. It is estimated that petroleum products will last only for about 25 years and natural gas for about 60–70 years. In this context, sole dependence on fossil fuels is not desirable and alternative sources for energy have become necessary [52]. Further, the prices of petroleum products in India are also

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Genetic Genomics Laboratory, National Institute of Plant Genome Research (NIPGR), New Delhi 110067, India e-mail: sushil2000_01@yahoo.co.in increasing day by day. This will have an impact on Indian economy [52, 63]. Biofuels such as biodiesel and bioethanol are considered as alternatives for energy production [25, 27, 37, 44]. Development of indigenous sources for energy production will minimize the nation's fossils fuel bill, thus reducing the expenditure on foreign exchange reserves [63]. Plants having more than 20 % fatty oils in their seeds or seed kernels can be definitely tried for the production of biodiesel [64]. Some of the vegetable oils which have the potential for use as biodiesel are rapeseed oil, soybean oil, linseed oil, rice bran oil, karanj oil, Jatropha curcas oil, neem oil, sunflower oil, palm oil, etc. [25, 36, 43]. As some of these oils are used as edible oils, their use for the production of biofuels may create shortage of these oils for human consumption. This problem can be solved only if the nonedible oils are used for the development of biofuels [38, 58] or by abundant production of edible oils.

Technically, biodiesel is the vegetable oil methyl esters. Fatty oils of vegetable origin contain glycerol esters of fatty acids known as triglycerides. Removal of glycerol molecules from the triglycerides and methylation of the resulting fatty acids gives the so-called biodiesel [26, 55]. Fatty oils obtained from vegetable sources cannot be used directly in engines because of high viscosity. High viscosity of the oils causes unfavourable operational problems

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and results in high engine deposits and thickening of lubricating oil [55]. In order to reduce the viscosity of the oil, the oil has to be chemically modified in order to bring the combustion related properties closer to the properties of diesel oil and increase the volatility of the oil. This is done by transesterification of the oil using methanol and catalyst. If ethanol is used for transesterification, fatty acid ethyl esters are formed and the ethylated fatty oils can also be used as biodiesel [51, 55, 59]. The advantages of biodiesel are that it increases the engine life, and is ecofriendly, clean burning, nontoxic, biodegradable and renewable fuel [23, 28, 34]. Presently, the fatty oils of J. curcas and Pongamia pinnata are considered highly suitable for use as biodiesel [25, 54]. Cultivation of these two plants is being promoted for the production of biodiesel. Already large plantation of these tree plants have been taken up in several parts of India [45, 56]. We found in previous study that the plant Cleome viscosa, belonging to the family Capparidaceae whose seeds contain more that 20 % of fatty oil, is also suitable for use as biofuel [47]. Our previous work also showed that C. viscosa can be domesticated as a crop for large scale utilisation [49]. Properties of biodiesel depend on fatty acid compositions of oils [46]. In addition to previous works, this paper compares the fatty acid composition of C. viscosa, which has been extensively surveyed in previous work [48] with fatty acid composition of J. curcas oil.

Materials and Methods

Oil Extraction

Oils of C. viscosa and J. curcas were extracted from 15 kg seeds with solvent extraction process using hexane by soxhlet apparatus. A rotary evaporator was used to separate oil from solvent at 65-70 °C. The seeds of C. viscosa accessions collected from New Delhi, Faridabad (Haryana) and Jaipur (Rajasthan) [49] and grown for evaluation at NIPGR were pooled together for oil extraction The seeds of J. curcas were collected from trees raised from elite clones obtained from National Botanical Research Institute, Lucknow and grown at NIPGR farm.

Identification of Constituents of Oils

Oils were analysed as in Table 1 by different parameters such as acid value, saponification value, calorific value, viscosity, refractive index, density and specific gravity with ASTM methods. The fatty acids present in the oils of these populations were identified as their methyl esters by converting the fatty acids present in the form of triglycerides to methyl esters by treating the fatty acids with methanol using boron trifluoride as catalyst. Shimadzu gas chromatograph equipped with FID using fused silica column AB—Innowax (60 m \times 0.25 mm i.d \times 0.25 µm film thickness) and gas-mass spectrometry (Shimadzu GC-MS model QP 2010 plus and staphase used was SPTM—2560 column tionary 100 m \times 0.25 mm i.d \times 0.20 µm film thickness) analyses of methylated oils were carried out. NIST and the literature [2] were used to compare and identify oil components of their mass spectra.

Conversion of Oil into Biodiesel

300 ml reactor flask provided with steering and reflux condenser heated over mantle at 65 °C was used to convert oil into biodiesel using 2 % H₂SO₄ and methanol [26, 33] for 24 h. Glycerine was separated from reaction product. Crude biodiesel was purified by water and dried by rotary evaporation.

Characterisation of Biodiesel

Standard methods referred in Table 2 were used to characterize the biodiesel.

Table 1 Physicochemical characteristics of the fatty oils of C. viscosa and J. curcas from Aravali range Aravali range	Characteristic	C. viscosa	J. curcas	Reference
	Acid value (mg/KOH/g)	50	5	[22]
	Viscosity at 40 °C (mm ² /s)	30	33	[21]
	Density at 15 °C (g/cm ³)	1	1	[15]
	Specific gravity (g/cm ³)	1	1	[15]
	Refractive index at 20 °C	1.5	1.5	[14]
	Carbon residue (mass%)	0.5	0.3	[5]
	Lubricity (µm)	108	127	[41]
	Saponification value (mg/KOH/g)	212	215	[12]
^a Reference [60]	Calorific value (MJ/kg)	40	40^{a}	[13]

^a Reference [60]

 Table 2 A comparison of parameters of C. viscosa biodiesel, with those specified by IS and ASTM for commercial biodiesel

S. no.	Parameter	Unit	C. viscosa	IS 15607 ^a	ASTM 6751 ^b	Reference(s)
1	Flash point (closed cup)	°C	175 ± 4	≥120	<u>≥</u> 93	[20]
2	Kinematic viscosity at 40 °C	mm ² /s	4.5 ± 0.5	2.5-6.0	1.9–6.0	[21]
3	Cloud point	°C	21.2 ± 1.4	NR	NM	[18]
4	Oxidation stability	h	1.0 ± 0.6	≥6	<u>≥</u> 3	[32]
5	Carbon residue (100 % sample)	Mass%	0.04 ± 0.03	<u>≤</u> 0.05	<u>≤</u> 0.05	[5]
6	Acid number	mg/KOH/ g	0.1 ± 0.04	<u>≤0.50</u>	≤0.50	[22]
7	Sulphur	Mass%	0.0015 ± 0.001	≤0.0050	≤0.0015 (15 Grade) ≤0.05 (500 Grade)	[6]
8	Copper strip corrosion level	Number	1 ± 1	≤1	≤3	[7]
9	Cetane number		55.2 ± 3.0	≥51	≥47	[40]
10	Free glycerine	Mass%	0.0045 ± 0.001	≤0.02	≤0.02	[11]
11	Total glycerine	Mass%	0.0240 ± 0.1	≤0.25	≤0.24	[11]
12	Ester content	Mass%	96.5 ± 0.1	96.5	NR	[30]
13	Phosphorus content	Mass%	<10 ppm	≤0.001	≤0.001	[10]
14	Sulfated ash	Mass%	0.005 ± 0.006	≤0.020	≤0.020	[<mark>9</mark>]
15	Water and sediments	vol%	0; 0	Water ≤0.05 (wt%)	<u>≤</u> 0.05	[<mark>8</mark>]
				Total contaminants ≤0.0024 (wt%)		
16	Methanol content	vol%	0.001 ± 0.001	<u>≤</u> 0.20	≤0.20	[31]
17	Ca + Mg, combined	ppm	<1	NM	<u>≤</u> 5	[10]
18	Na + K combined	ppm	<1	NM	<u>≤</u> 5	[10]
19	Density at 15 °C	g/cm ³	0.89 ± 0.01	0.86–0.90	NR	[15]
20	Iodine value	Number	116.3 ± 0.03	NM	NR	[<mark>61</mark>]
21	Refractive index at 20 °C	Ratio	1.46 ± 0.01	NR	NR	[14]
22	ASTM colour value	Number	1.8 ± 0.2	NR	NR	[<mark>16</mark>]
23	Specific density at 15 °C	Ratio	0.89 ± 0.002	NR	NR	[15]
24	Calorific value	MJ/kg	39.7 ± 0.2	NR	NR	[13]
25	Cold filter plugging point (CFPP)	°C	$+9.1 \pm 2.7$	NR	NR	[19]
26	Lubricity	μm	198 ± 9.9	NR	NR	[41]
27	Fatty acid composition	%		NR	NR	[3, 4]
	C16:0		8.1 ± 1.5			
	C18:0		4.5 ± 1.7			
	C18:1		19.9 ± 0.9			
	C18:2		67.4 ± 4.4			
28	Pour point	°C	$+3 \pm 3$	NR	NR	[17]

^a Indian Standard for testing 100 % biodiesel: 15607 (2007)

^b American Society for Testing and Materials standard for testing 100 % biodiesel: 6751 (2009)

Results

Physicochemical Characteristics of *C. viscosa* and *J. curcas* Fatty Oils

The physicochemical characteristics of the fatty oil of *C. viscosa* seeds are presented in Table 1 and compared the data are compared with those of the fatty oil of the seeds of *J. curcas*. The data for *C. viscosa* oil are comparable with the data for *J. curcas* seed oil. Table 1 shows that the value of lubricity is somewhat higher for jatropha oil (127) than

C. viscosa oil (108) and the carbon residue of *C. viscosa* oil is high compared to jatropha oil. Acid value of *C. viscosa* oil is much higher than *J. curcas* oil. However, the quality of biodiesel does not depend on acid value of oil, since acid value is used to select appropriate catalyst for conversion of oil into biodiesel (e.g. acidic or basic catalyst will be selected respectively for higher acidic and lower acidic value) [26, 33].

Other data such as viscosity, density, specific gravity, refractive index, and saponification value for both the oils are more or less similar.

Table 3 Constituents of themethylated fatty oils ofC. viscosa and J. curcas

Constituents	Percentage	
	C. viscosa	J. curca
Methyl myristate	0.1	0.2
Methyl pentadecanoate	Т	ND
Methyl palmitate	12.2	15.1
Ethyl palmitate	Т	ND
(9E)-methyl hexadecenoate	0.1	0.1
(9Z)-methyl hexadecenoate (methyl palmitoleate)	0.2	1.5
Methyl heptadecanoate (methyl margarate)	0.4	0.2
Tricosane	Т	ND
Methyl stearate	8.3	9.4
Tetracosane	Т	Т
Ethyl stearate	Т	ND
Methyl oleate	20.8	40.6
Methyl (11E)-octadecenoate	0.5	ND
Ethyl oleate	Т	ND
Pentacosane	Т	Т
Methyl linoleate	53.1	30.8
Ethyl linoleate	0.1	ND
Methyl arachidate	0.4	0.5
Hexacosane	0.2	0.1
Methyl (11Z)-eicosenate	0.2	0.1
Methyl linolenate	0.4	0.4
Methyl heneicosanate	Т	Т
Heptacosane	0.2	0.1
Methyl behenate	0.1	0.1
Octacosane	0.1	0.1
Methyl tricosanoate	0.1	0.1
Nonacosane	0.2	0.1
Methyl lignocerate	0.2	0.1
Triacontane	0.2	Т
Methyl pentacosanoate	0.1	Т
Hentriacontane	0.1	Т
Methyl hexacosanate	0.1	0.1
Dotriacontane	0.1	Т
12-Oxo ethyl stearate	1.2	ND
Saturated fatty acid	22.0	25.8
Monounsaturated fatty acids acid methyl (and ethyl) esters	21.6	42.2
Polyunsaturated fatty acid methyl (and ethyl) esters	53.8	31.3
Alkanes	1.1	0.4
Other	1.2	ND

T trace amounts, *ND* not detected

Fatty Acid Composition of the Oils of *C. viscosa* and *J. curcas*

Fatty acid composition of the fatty oil of *C. viscosa* is one of the important characteristic for the assessment of the quality of biodiesel from the oil. Thirty-four constituents of the oil of *C. viscosa* and 26 constituents of *J. curcas* oil were identified. The identified compounds with their

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relative percentages in the oils are presented in Table 3. The major fatty acids of the oil of *C. viscosa* are palmitic acid (C16:0) (12.2 %), stearic acid (C18:0) (8.3 %), oleic acid (C18:1) (20.8 %), and linoleic acid (C18:2) (53.2 %). In addition, myristic acid, pentadecanoic acid, ethyl palmitate, (9E)-hexadecenoic acid, palmitoleic acid, heptadecanoic acid, (11E)-octadecenoic acid, ethyl oleate, ethyl linoleate, arachidic acid, (11E)-eicosenoic acid, linolenic

acid, heneicosanoic acid, behenic acid, tricosanoic acid, lignoceric acid, pentacosanoic acid, hexacosanoic acid, 12-oxostearic acid and the hydrocarbons tricosane, tetracosane, pentacosane, hexacosane, heptacosane, octacosane, nonacosane, triacontane, hentriacontane and dotriacontane were also found as minor and trace components in the oil of *C. viscosa*. Thus, the fatty oil of *C. viscosa* contains 22.0 % of saturated fatty acids, 21.6 % of monounsaturated fatty acids and 53.8 % of polyunsaturated fatty acids.

The main components of *J. curcas* seed oil from Aravali range are palmitic acid (15.1 %), stearic acid (9.4 %), oleic acid (40.6 %) and linoleic acid (30.8 %). Besides, the oil was found to contain myristic acid, pentadecanoic acid, margaric acid (C17:0), (9E)-hexadecenoic acid, palmitoleic acid, arachidate acid, (11Z)-eicosenoic acid, linolenic acid, heneicosanoic acid, behenic acid, tricosanoic acid, lignoceric acid, pentacosanoic acid and hexacosanoic acid, and the alkanes tetracosane, pentacosane, hexacosane, heptacosane, octacosane, nonacosane, triacontane, hentriacontane and dotriacontane were found as minor and trace constituents. Thus, the fatty oil of *J. curcas* from Aravali range contained saturated fatty acid (25.8 %), monounsaturated fatty acids (42.2 %) and polyunsaturated acids (31.3 %).

Yield and Quality of C. viscosa Biodiesel

The biodiesel yield from the oil of *C. viscosa* was 96.7 %. Table 2 provides the description of the biodiesel in terms of 28 parameters. Table 2 gives the values of the parameters expected to be met according to the Indian standards 15607 of 2007 and American Society for Testing and Materials 6751 of 2009.

Discussion

Comparison of the compositions of the fatty oils of *C.* viscosa and *J. curcas* (Table 3) shows that the oil of *C.* viscosa contained relatively low amounts of the monounsaturated fatty acid i.e. oleic acid and high amounts of the diunsaturated fatty acid i.e. linoleic acid than the oil of *J. curcas*, though the level of saturated fatty acids (palmitic acid, stearic acid, arachidic acid, etc.) in the two oils is somewhat similar (22.0 and 25.8 %). This indicated that the methylated fatty oil of *C. viscosa* has less stability during oxidative process and the biodiesel prepared from this oil is less stable but may have better performance in cold weather conditions [24]. Expectedly, the biodiesel of *C. viscosa* met more or less all the parameters set under Indian and American standards, except for the oxidation stability.

The seed yields of the annual crop of *C. viscosa* accession CVR14 was observed to be 1.2 ± 0.4 t/ha for the crops sown in July–August and harvested in October–November which took ~4 months [49]. The seed yield from 1 year and older perennial plantations of *J. curcas* has been reported to vary between 1.8 ± 1.8 and 6.3 ± 2.7 t/ ha [1, 29, 35, 39, 42, 50, 53, 56, 57, 62, 65].

As the *C. viscosa* plant grows wild in several parts of India during rainy season, commercial cultivation of this plant with higher yields may not be a problem if proper agricultural practices are developed. Future work on extensive screening of populations and analysis of the oils of the seeds of *C. viscosa* populations followed by breeding may result in the development of suitable varieties with high oil content and with high oleic acid content and low contents of linoleic acid and other polyunsaturated fatty acids in the oils.

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