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Oleo Chemistry of Seed Oils of *Sida Cordifolia* and *Ervatamia Coronaria:* Assessment For Their Biodiesel Profile

Kariyappa S Katagi[1]*, Mahesh Akki[2], Mohankumar H.B[2], Vinayak K. Bangari[2]
Siddhaling B. Shiradoni[2] and Ravindra S. Munnoli[3]

1. Department of Chemistry, Karnatak University's, Karnatak Science College, Dharwad - 580 001, **INDIA**
2. Post Graduate Department of Studies in Chemistry, Karnatak Science College, Dharwad -580 001, **INDIA**
3. Department of Chemistry, VDR Institute of Technology Haliyal-581 329, **INDIA**

Email: kskatagi@gmail.com

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ABSTRACT

In this work new feedstock for biodiesel production has been screened. Non edible seed oil species like Sida cordifolia (SC) and Ervatamia coronaria (EC) plant species which yields 30.7% and 41.6% seed oil respectively. The molecular weight (MW) of oil is calculated based on the percentage component fatty acids of the seed oils. The prominent parameters of bio-diesel such as cetane number (CN), lower heating value (LHV) and higher heating value (HHV) of these Fatty Acid Methyl Esters (FAMEs) are empirically determined. The bio-diesel property of FAMEs of these seed oils is compared with existing bio-diesels. This confirmed the suitability of these seed oils for the generation of biodiesel. The seed oils selected in this investigation convene the major specification of biodiesel standards organizations like American (ASTM), Germany (DIN) and European (EN). This work reports the suitability of these candidates for the bio-diesel productivity.

Keywords: *Sida cordifolia, Eravatamia coronaria,* unusual fatty acids, industrial utilization, biodiesel.

INTRODUCTION

The global plant oils production over last 40 years has amplified from 23 million metric tons in 1967 to 129 million metric tons in 2007 [1]. It is estimated that about 90% of world's population will be located in developing countries by 2050. Under such circumstances the biomass energy will remain a sustainable energy feedstock to mitigate the future energy crises [2]. The seed oils have much higher density than starch and hence more energy per volume is obtainable. The seed oil has 2.25 times more energy than starch or proteins [3]. The markets in which plant oils find their usage, include a range of products such as, surfactants, soaps, detergents, lubricants, solvents, paints, cosmetics, and chemicals. In addition there are good numbers of *unusual fatty acids* produced by wild plant species or minor seed oils (non edible). The unusual fatty acids include those with chain lengths amid 8 and 22 carbons containing double bonds or conjugated systems or with functional groups such as a*cetylenic* bond or epoxy group or *hydroxyl* group or *cyclopropenoid* entity. The seed oils containing unusual fatty acids have wider industrial applications like protective coatings, plastics, urethane derivatives, dispersants, cosmetics, lubricant additives,

pharmaceuticals, textiles, variety of synthetic intermediates, and stabilizers in plastic formulations etc. Oral Kurganbekovich Beisenbaev et al reported that, gossypol resin is a slop of vacuum distillation of fatty acids derived from cotton seeds by processing of seeds and cottonseed oil [4, 5].
Fossil fuels are depleting in a very high-speed. Alternative energy sources are the only solution to alleviate energy crisis. Bio-fuels are dragging much attention worldwide. Animal fats and vegetable oils are most widely occurring lipids. Chemically, vegetable oils / animal fats are triglyceride molecules wherein three fatty acid groups / esters attached to glycerol molecule.

Vegetable oils could only replace a small fraction of transport fuel. But the major problem posed by direct use of vegetable oil is its higher viscosity compare to common diesel fuel. The methods applied to reduce the viscosity of seed oil are; dilution, micro emulsification, pyrolysis, catalytic cracking and trans esterification [6]. Transesterification consists of a sequence of three consecutive reversible reactions involving conversion of triglycerides to diglycerides, and diglycerides to monoglycerides. Finally, glycerides are converted into glycerol and one ester molecule in each step. The transesterification method is vastly practiced throughout the globe for the production of biodiesel.
Biodiesel contains about 77% carbon, 12% hydrogen and 11% oxygen by weight [7]. It has low viscosity and improved heating value which result in shorter ignition delay and longer combustion duration thus low particulate emissions. Biodiesel has become more attractive recently because of its eco-friendly nature. Its advantages includes; its domestic origin, renewability, biodegradability, higher flash point, inbuilt lubricity and blending capability with petro diesel. The use of pure bio-diesel especially in the transport sector brings down the emissions of CO_2 by 80%. It has high cetane number than that of petro diesel.
Biodiesel is a realistic fuel for future. It posses more or less analogous properties to that of petro diesel, but low exhaust emissions, non-toxic, free of sulfur and aromatics. It has more than 10% oxygen weight which reduces the carbon monoxide, oxides of sulphur and volatile organic content. Its flash point is more (423 K) compared to petro diesel (337 K). It is non-flammable hence non-explosive also. The mechanical problems associated with biodiesel are oxidative stability, poor low-temperature properties, and bit higher NO_x exhaust emissions. The latter problem may be solved over time with the introduction of new exhaust emission control technologies [8,9,10].
To reduce the processing and raw material cost of biodiesel it is recommended to use *non edible seed oils* as raw materials, if unusual fatty acids are identified during analysis of seed oil, prior to transesterification process the isolation of industrially important unusual fatty acid/s to be carried out. Then, the rest of the material subjected for transesterification. Finally, the recovery of glycerol is important. Glycerol may be used as a chemical feedstock in the various products like polyurethanes, polyesters, polyethers, lubricants, wrapping and packaging materials, foods, drugs, cosmetics, tobacco products and products that displace existing petroleum derived materials. The manifestation of the biodiesel industry has generated a additional glycerol, which has initiated numerous efforts to find new products, and newer markets using glycerol as the resourceful chemical [11,12,13,13].

Biodiesel fuels obtained from different sources, varies with fatty acid profiles. Generally it contains C_{14}–C_{22} lower alkyl fatty acid esters. FAMEs of vegetable oils have outstanding advantages over clean engine fuel alternatives. Currently, the commonly occuring fatty esters in biodiesel are those of palmitic acid, stearic acid, oleic acid, linoleic acid, and linolenic acid. Usually, *Soybean, palm, sunflower, safflower, cottonseed, rapeseed,* and *peanut* oils are considered potential alternative fuels for diesel engines. The major seed oils sources like *Coconut, corn, cottonseed, canola , olive, peanut , safflower, soybean,* and *sunflower* species are the multipurpose oils which are used for both biodiesel purpose as well as edible purpose. This created more demand, escalating price, etc., of these seed oils due to competition of these seed oils for food and fuel. On the other hand the non edible seed oils which are already in for the purpose are *babassu , copaiba, honge, jatropha , jojoba, karanja, pongamia, mahua, nagchampa, neem, petroleum nut, rubber tree seeds, nicotinica tabacum, deccan hemp, cerebra oddulum* etc., Researchers are looking forward to new avenues for raw materials, especially non-edible materials for the production of biodiesel [15].

Biodiesel can provide just such an interim solution. However, no feed stock seems to exist that fulfils all requirements. It is becoming apparent that there may be an optimal fatty acid profile that provides optimal fuel properties while minimizing environmental risks and other negative effects. Knoth suggested that a mixture consisting primarily of methyl oleates and mixture of shorter chain esters derived from palmitoleic acid and esters derived from decanoic acid might be an ideal mixture. To meet this requirement transgenic crops are suggested [16,17,18,19].

The aim of this paper is to apply the already developed theoretical models (substantiated with experimental results) of fuel parameters on selected non edible seed oils. The individual non edible seed oils and their readily available fatty acid profile are utilized for assessment of their biodiesel profile. The non edible seed oils which yield more than 30% seed oil have been selected and subjected for screening their candidature for biodiesel. After the meticulous survey we have selected *Sida cordifolia* (SC) and *Ervatamia coronaria* (EC) for this investigation.

Sida Cordifolia: It belongs to the family *Malvaceae*. *Sida cordifolia* is an erect perennial that reaches 50 to 200 cm (20 to 79 in) tall, with the entire plant covered with soft white felt-like hair that is responsible for one of its common names, "flannel weed". The stems are yellow-green, hairy, long, and slender. The yellow-green leaves are oblong-ovate, covered with hairs and 3.5 to 7.5 cm (1.4 to 3.0 in) long by 2.5 to 6 cm (0.98 to 2.36 in) wide. The flowers are dark yellow, sometimes with a darker orange center, with a hairy 5-lobed calyx and 5-lobed corolla [20].

Ervatamia Coronaria: It belongs to the family *Apocynaceae*. An ornamental shrub reaching to 3 m tall, native probably of northern India, but widely dispersed by man, and now cultivated as an ornamental in the West African region. In its native area the wood is burnt as incense and is used to make perfume, and medicinally to provide a refrigerant (cooling) drink. A decoction of the roots is used in Indonesia to stop diarrhoea. Pulp surrounding the seed is used in the Himalayan area to produce a red dye [21].

Component Fatty Acids In Seed Oils: Listed below in Table-1 are the fatty acid components with their chemical formulae and molecular weights that make up the two seed oils under investigation. There are twelve fatty acids out of which seven are unsaturated acids.

Table 1. Fatty acids that constitute the two seed oils under investigation [22] [30].

Fatty acid	Molecular formula	Molecular weight
Myristic acid	$CH_3(CH_2)_{12}COOH$	230.30
Palmitic acid	$CH_3(CH_2)_{14}COOH$	256.40
Palmitoleic acid	$CH_3(CH_2)_5CH{=}CH(CH_2)_7COOH$	254.40
Stearic acid	$CH_3(CH_2)_{16}COOH$	284.40
Oleic acid	$CH_3(CH_2)_7CH{=}CH(CH_7)COOH$	282.46
Linoleic acid	$CH_3(CH_2)_4CH{=}CHCH_2CH{=}CH(CH_2)_7COOH$	280.45
Linolenic acid	$CH_3(CH_2)_4CH{=}CHCH_2CH{=}(CH_2)_7COOH$	278.40
Arachidic acid	$CH_3(CH_2)_{18}COOH$	312.45
Paullinic acid	$CH_3(CH_2)_5CH{=}CH(CH_2)_{11}COOH$	310.51
Behenic acid	$CH_3(CH_2)_{20}COOH$	428.35
Malvalic acid	$C_{18}H_{32}0_2$	280.44
Sterculic acid	$C_{19}H_{34}0_2$	294.47

MATERIALS AND METHODS

Oil extraction: The seeds of *Sida cordifolia* and *Ervatamia coronaria* were ground, powdered and the oil content extracted by extraction with light petroleum ether (B.P. 40-60 ^{0}C) in a Soxhlet extractor for 24 h. The organic extract has to be filtered and dried over anhydrous Na_2SO_4. The petroleum ether removed under vacuum. The % oil for each 100 g of dry seeds is calculated.

Transesterification: This is a reversible reaction. A strong base / a strong acid are used as a catalyst. Sodium or potassium methanolate can be used for efficient conversion of fatty acids present in oils to their corresponding FAMES.

Triglyceride	+ **Alcohol**	⇌	**FAMEs or FAEEs**	+	**Glycerol**
(Seed oil)	(Methanol / Ethanol)		(Biodiesel)		(Byproduct)

Procedure: The seed oil is treated with alcohol (6:1 ratio) usually methanol or ethanol in presence of acid or base as catalyst and refluxed until completion of the esterification reaction. Then the mixture is transferred to separating funnel then it is allowed to stand for overnight. The lower layer containing glycerol, methanol and most of the catalysts was drained out. The upper layer containing methyl esters, some methanol, traces of the catalyst are cleaned thoroughly by washing with de-ionized water. Then, the residual methanol is removed by rotary evaporation at around 70^{0}C. Thus obtained product containing FAMEs is used as biodiesel.

The selected seed oils were investigated for fuel properties as per American (ASTM), Germany (DIN D6751) and European (EN 14214) standards. The highly useful analytical data for this work such as % SFAs, %USFAs, IV, SN, CN, LHV, HHV are deployed in this work. The SN and IV were calculated empirically with the help of Eqs. (1) and (2) respectively or referred from the literature. SN depends upon the molecular weight and the percentage concentration of fatty acid components present in FAMEs of oil. However, IV, according to Eq. (2), depends upon three variable percentage concentrations of unsaturated fatty acid components, their molecular weight and the number of double bond(s) present in them. Percent seed oil content SN, IV and fatty acid compositions of the selected seed oils is collected from the literature [22,30].

Biodiesel Property Of *Sida Cordifolia* And *Ervatamia Coronaria* Seed Oils Based On Fames: The selected seed oils were investigated for fuel properties as per American (ASTM), Germany (DIN D6751) and European (EN 14214) standards.

Iodine value and Saponification number: The SV and IV of fatty acid of these seed oils are calculated and used to establish their suitability for biodiesel. Thus, SV and IV of seed oil are calculated from the equations (1) and (2) respectively based on compositions of fatty acid methyl ester [23] results obtained are very close to experimental values.

$$SV = \sum \frac{560 \times A_i}{M_{wi}} \qquad [1]$$

$$IV = \sum \frac{254 \times N_{db} \times A_i}{M_{wi}} \qquad [2]$$

where, Ai is the percentage of component fatty acids, D is the number of double bonds and Mwi is the molecular mass of each component.

Cetane number (CN): The CN measures how easily ignition occurs and the smoothness of the combustion. The CN affects a number of engine performance parameters like combustion, stability, drivability, white smoke, noise and emission of carbon monoxide and hydrocarbon. Generally, biodiesel has higher CN than conventional diesel fuel, which results in higher combustion efficiency. It is an important parameters of the biodiesel / petro diesel fuel. It's significance is useful during selection of FAMEs to use as biodiesel. The CN of FAMEs is calculated [24] using the equation (3) and is known closely match to the experimental values.

$$CN = 46.3 + \frac{5458}{SV} - 0.225 \text{ x } IV \qquad [3]$$

Usually, FAMEs with higher CN are preferred. But the increase of CN thereby IV decreases it means degree of unsaturation goes down leading to the solidification of FAMEs at elevated temperature. To avoid this situation, US biodiesel standard specified the upper limit of CN up to 65 only [25].

Higher heating value (HHV): It is known that straight and processed vegetable oils used in diesel engines are the complex chemical mixture of FAMEs. The HHV of biodiesel is calculated using equation (4) in accordance with regression model [26].

$$HHV = 49.43 - (0.015 \text{ x } IV) - (0.041 \text{ x } SV) \qquad [4]$$

Lower heating value (LHV): The LHV of straight and processed vegetable oils is estimated with respect to equations (5) and (6) respectively based on bond energy values of chemical structure of different FAMEs. The method established for the calculation of lower heating value is quite general and its predictive ability is more precise.

For FAMEs,

$$LHV = 0.0109 \left(\frac{C}{O}\right)^3 - 0.3516 \left(\frac{C}{O}\right)^2 + 4.2000 \left(\frac{C}{O}\right) + 21.066 - 0.100\ N_{db} \qquad [5]$$

$$LHV = 0.0011 \left(\frac{H}{O}\right)^3 - 0.0785 \left(\frac{H}{O}\right)^2 + 2.0409 \left(\frac{H}{O}\right) + 20.992 - 0.100\ N_{db} \qquad [6]$$

Where, C, H & O are the number of carbons, hydrogen and oxygen respectively. N_{db} has the same meaning as stated above.

RESULTS AND DISCUSSION

More than 30% seed oil is recorded from *Sida cardifolia* (SC) *and Ervatamia coronaria* (EC) seed oils. The major component fatty acids present in SC seed oils is 0.8% myristic acid, 13.5% palmatic acid, 6.9% stearic acid, 15.7% oleic acid, 34.6% linoleic acid, 1.3%linolenic acid [22]. Similarly, in the seed oil of EC which comprised of 24.4% palmitic acid, 0.2% palmitoleic acid, 7.2% stearic acid, 50.5% oleic acid, 15.8% linoleic acid, 0.8% linolenic acid, 0.7% arachidic acid, 0.2% paullinic acid, 0.2% behenic acid [30]. Apart from normal fatty acids mentioned above, 12.6% and 14.5 % malvalic and sterculic acids are detected in case of SC [22]. Details of analytical data and component fatty acids in above mentioned seed oils and structure of corresponding fatty acids are shown in table.1 and table 2 respectively.

Table2. Fatty acid profile of seed oils of *Sida cordifolia* and *Ervatamia coronaria*

Source/ Seed species		Sida cordifolia	Ervatamia coronaria
% Seed oil		30.7	41.6
Molecular weight of oil (g mol^{-1})		875.37	866.51
% Total saturated fatty acids (TSFAs)		21.2	32.7
% Total unsaturated fatty acids (TUSFAs)		78.7	67.3
Saponification number (mg KOH g^{-1})		197.4	201.1
Iodine value (mg I_2 g^{-1})		81.2	76
% Component fatty acids:	14:0 (myristic)	0.8	-
	16:0 (palmitic)	13.5	24.4
	16:1 (palmitoleic)	-	0.2

18:0 (stearic)	-	7.2
18:1 (oleic)	15.7	50.5
18:2 (linoleic)	34.6	15.8
18:3 (linolenic)	1.3	0.8
20:0 (arachidic)	-	0.7
20:1 (paullinic)	-	0.2
22:0 (behenic)	-	0.2
Malvalic	12.6	-
Sterculic	14.5	-

The molecular weight of individual seed oil is calculated based on component fatty acids. The MW of SC and EC lies in the range of 866.51 - 875.31g mol^{-1} respectively. Iodine value of these seed oils is not exceeding 120 which best fit as per the limitation laid by EN 14214 for biodiesel. There is consistency in the SN. Generally, the FAMEs, which are mainly comprised of carbon chain lengths from 16 to 18, have boiling points in the range of 330–357^{0}C; thus the specification value of 360^{0}C is easily achieved. In the same context the FAMEs of these species meet the specification of boiling point limit of 360^{0}C specified in ASTMD6751 and in other biodiesel standards.

The CN of the FAMEs of these oils ranged from 55.67 and 56.34 respectively. Biodiesel standards of USA (ASTMD 6751), Germany (DIN 51606) and European Organization (EN 14214) have set CN value as 47, 49 and 51, respectively [23]. The upper limit of CN (65) has been specified in US biodiesel standard (ASTMPS 121–99). Moreover, the CN of petro diesel is 42.6. Over all the empirically calculated CN value of FAMEs of SC and EC meet the requirements of biodiesel standards.

The LHV and HHV of the FAMEs are appreciable in these seed oil species. For HHV, equation (4) shows that, the increase in the value due to increasing chain length in the fatty acid. The decrease is due to increase in the number of double bonds. The HHV of a fuel is a function of its carbon, hydrogen and oxygen content. The LHV of straight and processed vegetable oils is estimated with respect to equations (5) and (6) respectively based on bond energy values of chemical structure of different fatty acids or FAMEs present in fuel molecules. The empirically calculated results, for both the species under this study reflect that there is a consistency of LHV that is with 37 MJ Kg^{-1} in all the species under this investigation. This is slightly lower than the LHV of petro diesel (43 MJ Kg^{-1}). The European Biofuels Technology Platform 2011 reported the LHV for biodiesel as 37.1 MJ Kg^{-1} [28].

The HHVs of biodiesels for these species are 40.12 MJ kg^{-1} and 40.04 MJ kg^{-1} respectively which are slightly lower than those of petro diesel (43 MJ kg^{-1}), or petroleum (42 MJ kg^{-1}), but are higher than coal (32–37 MJ kg^{-1}). Table 3 shows various fuel profiles of FAMEs of these seed oils.

Table4. Biodiesel properties of *Sida cordifolia and Ervatamia coronaria* seed oils

Source / seed species	***Sida cordifolia***	***Ervatamia coronaria***
Cetane number	55.67	56.34
Higher heating value (HHV; MJ kg^{-1})	40.12	40.04
Lower heating value C/O (LHV; MJ kg^{-1})	37.36	37.64
Lower heating value H/O (LHV; MJ kg^{-1})	37.30	37.77

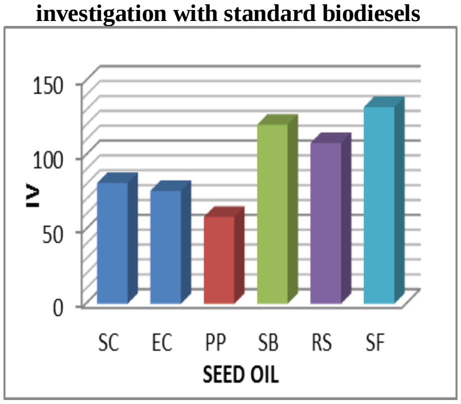
The overall screening of these selected species of the seed oils for biodiesel properties compared as shown are depicted in table 5. The calculated values are compared with existing biodiesel so also with the common diesel. The calculated values are very close to the standards. This reveals that, the selected seed oils at from the investigation can be a potential feed stock for the production of biodeisel.

Table 5. Comparison of properties of biodiesels from *Sida cordifolia* and *Ervatamia coronaria* with existing biodiesels and petro-diesel

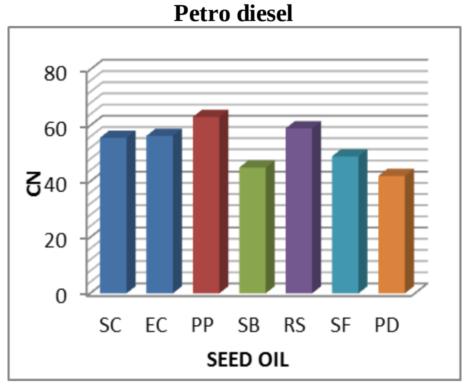
Fuel property	*Sida cordifolia* (SC)	*Ervatamia coronaria* (EC)	*Pongamia pinnata** (PP)	*Soyabean** (SB)	*Rapeseed** (RS)	*Sunflower** (SF)	Petro diesel (PD)
SV (mg KOH g^{-1})	197.4	201.1	182.3	194.6	197.1	193.0	NA
IV (mg I_2 g^{-1})	81.2	76	58.4	120.5	108.1	132.3	NA
CN	55.67	56.34	63.1	45.0	59.0	49.0	42.0
HHV (MJ kg^{-1})	40.12	40.04	40.5	39.8	37.0	40.6	46.0
LHV (MJ kg^{-1})	37.36	37.64	34.3	33.5	32.8	33.5	43.1

* indicates some of the data obtained from, Ref. [30] NA=Not applicable

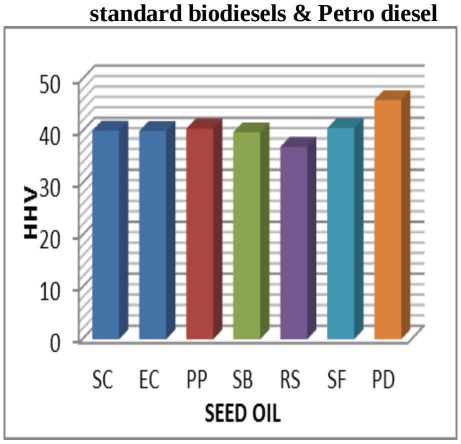
Comparison of Iodine Value of seed oils selected for investigation with standard biodiesels



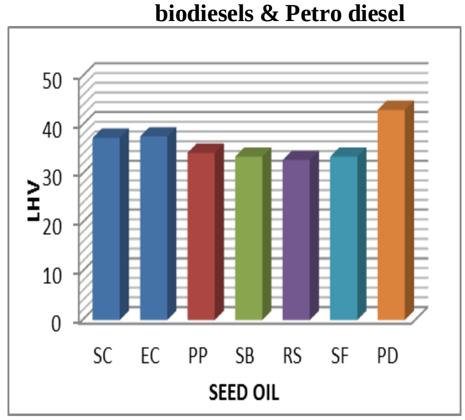
Comparison of Cetane Number of seed oils/FAMEs selected for investigation with standard biodiesels & Petro diesel



Comparison of Higher Heating Value of seed oils/FAMEs selected for investigation with standard biodiesels & Petro diesel



Comparison of Lower Heating Value of seed oils/FAMEs selected for investigation with standard biodiesels & Petro diesel



APPLICATIONS

Fatty acids are widely used as inactive ingredients in drug preparations, and the use of lipid formulations as the carriers for active substances is in high demand. The majority of lipids are used for manufacturing fat emulsions, mainly for clinical nutrition so also as drug vehicles and there has been an increase in the use of lipids as formulation ingredients. Fatty acids themselves or as part of complex lipids, are frequently used in cosmetics such as soaps, fat emulsions [29]. The byproducts of biodiesel viz., glycerol and seed cake have tremendous applications in chemical and cattle industries respectively.

CONCLUSIONS

The FAMEs of SC and EC seed oils meet the major specifications of US biodiesel standard (ASTMD 6751-02, ASTMPS 121-99), Germany (DIN V 51606) and European Standard Organization (EN 14214). The FAMEs of these seed oils is found to be the most suitable for biodiesel production since they meet the major specifications of biodiesel standards. However, still further research work is needed, to evaluate these FAMEs for other property measures like, tribological studies, and long-term engine testing before full-fledged alternative fuel. The two major co-products from the biodiesel process are protein-rich cakes/meals and glycerol. The cake has been major source of crude protein in commercial livestock and poultry production. If many of such plants are grown in large scale on suitable wastelands, the bio-diesel produced from such seeds can supplement or replace the petro-diesel.

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AUTHORS' ADDRESSES

1. **Dr. Kariyappa S Katagi**
Asst. Professor in Chemistry, Department of Chemistry
Karnatak Science College, Dharwad-580 001
Email: kskatagi@gmail.com
Mob.No. +91 9916248809
2. **Mahesh. Akki**
Post Graduate Department of Chemistry,
Karnatak Science College, Dharwad.
Email: maheshakki.1410@gmail.com
Mob: +918867188085
3. **Mohan Kumar. H. B**
Post Graduate Department of Chemistry,
Karnatak Science College, Dharwad.
Email: mohanhb92@gmail.com
Mob: +919060377657
4. **Vinayak. K. Bangari**
Post Graduate Department of Chemistry,
Karnatak Science College, Dharwad.
Email:vinay13s60032@gmail.com
Mob: +919738545135
5. **Shiddhaling. B. Shiradoni**
Post Graduate Department of Chemistry,
Karnatak Science College, Dharwad.
Email: shiradonisiddu@gmail.com
Mob: +917760842162
6. **Dr.Ravindra S. Munnolli**
Associate professor in Chemistry, Department of Chemistry,
VDR Institute of Technology, Haliyal-581329
Email: rsmunnolli@gmail.com
Mob: +91 9742741619