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BIODIESEL PRODUCTION AND FUEL PROPERTIES FROM NON-EDIBLE CHAMPACA (*MICHELIA CHAMPACA*) SEED OIL FOR USE IN DIESEL ENGINE

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ABSTRACT

In the present paper investigations were carried out to determine fatty acid composition and fuel properties of non-edible, renewable, champaca seed oil (CSO), champaca seed oil biodiesel (CBD), for its use in diesel engine. Methyl ester of seed oil was analyzed by Gas Chromatography (GC) - Mass Spectroscopy (MS) for the determination of fatty acid composition. The major fatty acids found in CSO were, palmatic , 32.52%, linoleic, 30.72%, palmitoleic, 13.39%, stearic, 8.88%, oleic , 6.03%, palmitelaidic, 5.18%, ecosenic, 0.71% and myristic, 0.57% and the total unsaturated fatty acid composition accounts for 56.03%. The biodiesel was produced by two step process i.e. acid pretreatment process followed by base-catalyzed transesterification process as the free fatty acid (FFA) content found to be 5.30% (corresponding to acid value of 10.55, mg KOH/g) . The first step of process was carried out with methanol and sulphuric acid as catalyst, followed by second step, base-catalyzed transesterification process with methanol and sodium hydroxide as catalyst the biodiesel yield was found to be 83.50 %. The fuel properties of biodiesel produced were determined as per the ASTM standard test procedures and compared with diesel, ASTM D6751-09a, biodiesel standard specifications and were found to meet the required standards.

1. INTRODUCTION

For the socio- economic development of any country energy plays an extremely significant role. Demand for energy and its resources, is increasing every day due to the rapid growth of population, and urbanization. The energy demand is met by the fossil fuels since from their exploration. At the end of 2013 the highest primary energy consumption in the world is crude oil with 32.9 % of global energy consumption and the reserve to production ratio of oil is 53.3 [1]. Mainly the oil is consumed by transportation sector, tractors & agriculture implements and decentralized power plants. With the present production and consumption pattern all the oil reserves may exhaust after 50-60 years. Due to limited reserves of fossil fuels, environmental degradation, and instability in fuel prices, there is a growing need for ensuring the uninterrupted availability of energy and protection of the environment.

Indigenously produced biodiesel, mono alkyl ester of vegetable oil or animal fat is gaining importance worldwide to meet the energy demand. There are more than 300 different species of trees which produce oil bearing seeds [2] only few of them being characterized for use in diesel engine. Which include Jatropha (*Jatropha curcas*) oil [3-13], Karanja or Honge (*Pongamia pinnata/glabra*) seed oil [4,9,14-20], Polanga (*Calophyllum inophyllum*) seed oil [21,22], Rubber (*Havea brasiliensis*) seed oil [23-26], Mahua (*Madhuca indica*) seed oil [9,27-30], Simarouba(*Simarouba glauca*) oil [29,30], Tobbaco (*Nicotiana*

tabacum) seed oil [31], Bitter almond (*Prunus dulcis*) oil [32], Neem (*Azadirochta indica A*). seed oil [9,10,33-36], Castor (*Ricinus communis*) seed oil [37, 38], Okra (*Hibiscus esculentus*) seed oil [39], Kusum (Scheichera triguga) oil [40] Bitter gourd (*Momordica Charantia*) seed oil [41], and Amoora (*Aphanamixis polystachya)* seed oil [42].

The present investigation aims at determining the fatty acid composition and characterizing biodiesel produced from non-edible, vegetable oil obtained from mechanical extraction of Champaca (*Michelia champaca*) seed for use in diesel engines for which very limited work has been done [43].

Champaca *(Michelia champaca*) is an evergreen or semi-deciduous, small to medium sized tree belongs to the family Magnoliaceae. The tree is native to India, but cultivated throughout the tropical and sub-tropical zone of South-east Asia extending from Nepal, India, Sri Lanka, Bangladesh, China, Indochina, Myanmar, Thailand, Malaysia to Indonesia. It is distributed in primary lowland to montane rain forest from 600-2000 m altitude. It grows best under very moist conditions on deep fertile, well-drained preferably sandy loam soils [44]. *Michelia champaca* flower, fruits and seeds were shown in Fig.1 a, b and c respectively.

FIG. 1 A) CHAMPACA FLOWER, B) FRUITS, C) SEEDS

2. MATERIALS AND METHODS

2.1 Materials

The champaca seeds were obtained from the local market. The seeds were dried and crushed in a mechanical expeller. For complete extraction of oil the seeds were passed four times through the expeller. The neat oil is allowed to settle for 48 hours and after that oil is stored in an air tight container to avoid oxidation. All chemicals of analytical reagent (AR) grade were procured from local supplier, methanol (> 99% purity), sulphuric acid (95% pure), NaOH used being in the form of pellets.

2.2 Gas Chromatography (GC) – Mass Spectroscopy (MS) analysis of champaca seed oil

The vegetable oil extracted from a seeds composed of triglyceride, which is an ester derived from three fatty acids and one glycerol. The fatty acid composition of methyl esters of champaca seed oil (CSO) sample was analyzed by GC – MS at Spectroscopic analytical test facility, Division of biological sciences, Indian Institute of Science, Bangalore. GC-MS analysis was performed on a GC, Thermo Scientific, Trace GC ultra coupled with MS, DSQII. Experimental conditions for GC: Zebron ZB 5 ms fused silica capillary column (30 m Length x 0.25mm Inner Diameter x 0.25µm film thickness); helium was used as a carrier gas with flow rate of 1 ml/min and an injection volume of 1µl was employed in a split less injector mode; Initial temperature 40^OC (hold time 2 min) with an increase of 10^OC/min to 300^OC (hold time 10 min). Mass spectra were taken in electron ionization mode; quadrupole; with a scan mass range from 30 m/Z to 600 m/Z and positive polarity. Total GC run time was 37 min. Software used in the analysis was, Xcalibur and Automated Mass Spectral Deconvolution and Identification System (AMDIS) software. The spectrum of the unknown compound was based on comparison with the spectrum of the known compounds stored in the National Institute Standard and Technology (NIST) 2011, library. Concentrations of the identified fatty acids were determined through area normalization.

2.3 Biodiesel production from CSO

The production process employed mainly depends on the amount free fatty acid (FFA) content in the oil. The feedstock with high amount of free fatty acid (FFA), could not be transesterified by the traditional alkaline catalysts, as alkaline catalysts form soap when they reacts with FFA and formation of soap prevents the separation of ester and glycerin [45]. Four different techniques are available for production of biodiesel with high amount of FFA. The enzymatic method, these methods requires expensive enzymes. Glycerolysis, the drawback of this method is the high temperature and that the reaction is relatively slow. Acid catalysis, the transesterification of triglycerides is very slow, taking several days to complete and another problem of this technique is that the water production. Acid catalysis followed by alkali catalysis, solves the reaction rate problems, once FFA being reduced an alkali catalyst being used for transesterification. This method can convert high FFA feedstock into biodiesel quickly and efficiently [46]. For feed stocks with high FFA two-step acid pretreatment followed by base-catalyzed transesterification process being the best approach for the production of biodiesel [45]. Hence in the production of biodiesel from CSO a two step process being employed.

The biodiesel from CSO was produced in the laboratory scale batch reactor equipped with thermometer and condenser, the heating and stirring was done with a hot plate magnetic stirrer system. The schematic biodiesel production process as shown in the Fig.2, the esterification and transesterification process were carried out in a three neck batch reactor, after the completion of reaction the mixture was poured into the separating funnel. In the separating funnel the phase separation takes place by gravity and after phase separation it is being purified to obtain the pure biodiesel.
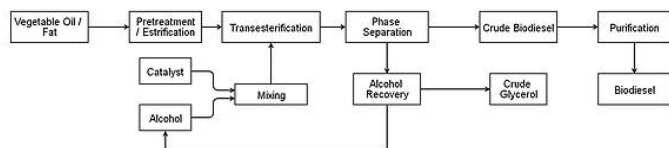
FIG. 2 SCHEMATIC DIAGRAM OF BIODIESEL PRODUCTION PROCESS

2.3.1 Acid pretreatment process: In the acid pretreatment process the reaction was carried out with the reaction conditions as tabulated in the Table. 1. After completion of the reaction the mixture was transferred to a separating funnel and allowed to settle for 60 minutes. After settlement of mixture, the FFA of lower layer was measured and taken for the next step for biodiesel production.

2.3.2 Base-catalyzed transesterification process: The second step, base–catalyzed transesterification reaction was carried out with the reaction conditions as tabulated in Table.1. After the completion of transesterification reaction the mixture was transferred into a separating funnel left for 60 minutes to separate into biodiesel and glycerol. The lower layer of glycerol was removed and the upper layer of crude biodiesel was washed several times with hot water at $50^{O}C$ to remove the impurities, such as residual catalyst, methanol, soap and glycerol. The removal of impurities was confirmed by measuring the pH of water. The biodiesel was dried by heating it to a temperature of $110^{O}C$ and allowed overnight for evaporation and cooling.

Table 1   Reaction conditions for biodiesel production

Step	Type of alcohol	Type of catalyst	Alcohol to oil molar ratio	Catalyst concentration (w/w %)	Reaction temperature (^{O}C)	Reaction time (Mins)
First step	Methanol	Sulphuric acid	24:1	1.50	60	75
Second step	Methanol	Sodium hydroxide	6:1	0.65	65	75

2.4 Analytical and test methods

The mean molecular weight, saponification number (SN), iodine value (IV) and cetane number (CN) were determined from the fatty acid composition of oil using the Eqs. (1), (2), (3) and (4) respectively [32, 47].

$$MW_{oil} = 3 \times \sum(MW_i \times x_i) + 38 \qquad (1)$$

Where MW_{oil}, stands for molecular weight of CSO, MW_i and x_i stand for molecular weight and mass fraction of i th fatty acid respectively.

$$SN = \sum(560 \times A_i/MW_i \qquad (2)$$

$$IV = \sum(254 \times D \times A_i/MW_i \qquad (3)$$

$$CN = 46.3 + 5458/SN - 0.225 \times IV \qquad (4)$$

Where, A_i is the percentage, D is the number of double bonds and MW_i is the molecular mass of each component.

The fuel properties of CSO and champaca biodiesel (CBD) were determined as per the ASTM standards and the biodiesel yield was calculated using the Eq. (5).

$$Biodiesel\ yield = \frac{m_{biodiesel}}{m_{oil}} \times 100 \qquad (5)$$

Where, $m_{biodiesel}$, is the weight of CBD after purification and m_{oil}, is the weight of CSO.

3. RESULTS AND DISCUSSION

3.1 Oil content of champaca seeds

The oil extracted from the mechanical expeller was weighed after filtering; it was found that the champaca seeds contain low quantity of oil, 14.5 w/w % oil. The oil yield by soxhlet extractor with petroleum ether found to be 45.0% [43], hence chemical method to be employed for extraction in order to obtain maximum oil yield.
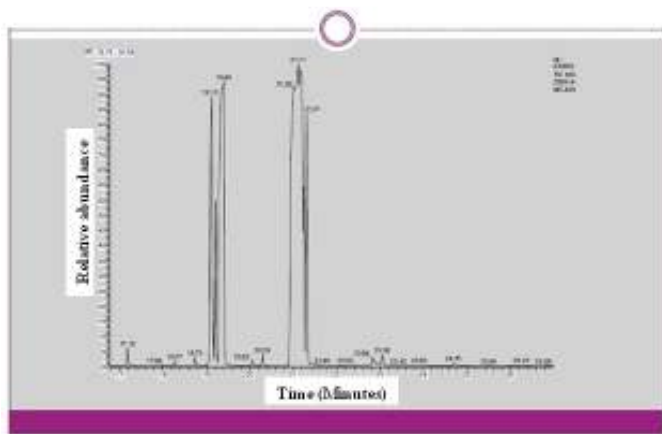
3.2 Fatty acid composition analysis

Generally three main types of fatty acids are present in triglyceride and they are, saturated, monounsaturated and polyunsaturated. The quality of biodiesel will be affected by the fatty acid composition of oil. The biodiesel fuels derived from fat or oils with significant amounts of saturated fatty compounds will display poor low temperature flow properties [48], where as highly unsaturated compounds have acceptable cold flow properties and they are more prone to oxidation [49].

The fatty acid profile of CSO was shown in Table.2 and Fig.3 shows the combined GC-MS chromatogram, where x-axis represents the retention time and y-axis represents the relative abundance. In order to determine the composition the sample was injected into the GC in which the molecules present in the sample separate as they pass through the column. Once the molecules elute from the GC column, they were taken into MS and ionized by electron impact. Using the AMDIS software, the spectrum of the unknown compound was compared with the spectrum of the known compounds stored in the NIST 2011, library. The major fatty acids present in the CSO were, palmatic , 32.52%, linoleic, 30.72%, palmitoleic, 13.39%, stearic, 8.88%, oleic , 6.03%, palmitelaidic, 5.18%, ecosenic, 0.71% and myristic, 0.57%. The total unsaturated fatty acid composition accounts for 56.03%. Due to high amount of unsaturated compounds the CBD may have acceptable cold flow properties and may be more prone to oxidation.

Table 2 Fatty acid profile of champaca seed oil obtained by Gas Chromatography (GC) - Mass Spectroscopy (MS)

Fatty acid	Structure (Carbon atoms in fatty acid chain: Double bonds)	Formula	Weight percentage in CSO
Myristic	C14:0	$C_{14}H_{28}O_2$	0.57
Palmitic	C16:0	$C_{16}H_{32}O_2$	32.52
Stearic	C18:0	$C_{18}H_{36}O_2$	8.88
Palmitoleic	C16:1	$C_{16}H_{30}O_2$	13.39
Palmitelaidic	C16:1	$C_{16}H_{30}O_2$	5.18
Oleic	C18:1	$C_{18}H_{34}O_2$	6.03
Linoleic	C18:2	$C_{18}H_{32}O_2$	30.72
Ecosenoic	C20:1	$C_{20}H_{38}O_2$	0.71
Others	----	----	2.00
Total	-----		100.00

FIG. 3 GC-MS CHROMATOGRAM OF CSO

3.3 Physicochemical properties of CSO and CBD

The physical and chemical properties of CSO and CBD were determined as per the ASTM standard test procedures and tabulated in Table.3. The fuel properties of CSO and CBD were compared with commercially available diesel, simarouba oil and simarouba biodiesel as simarouba oil contains approximately same amount of FFA and it was found that most of the fuel properties were quite comparable to that of simarouba oil and simaroba biodiesel.

3.3.1 Iodine value and saponification number: Iodine value measures the number of double bonds in biodiesel and indicates the unsaturation level. The iodine value of CSO was calculated using Eq.(3) and tabulated in Table.3. it was found to be 122.71 mgI$_2$/g, as the iodine value is higher, which indicates the unsaturation of CSO. The heating of these higher fatty acids results in polymerization of glycerides, which necessitates the limitation of unsaturated fatty acids otherwise it leads to formation of deposits and deterioration of lubricating oil. The saponification of CSO was calculated using Eq.(2) and was found to be 209.17 mg KOH/g of oil, which indicates that the CSO is normal triglyceride.

3.3.2 Acid value and FFA content: Acid number or value is defined as the number of milligrams of KOH required to

neutralize the free acid present in one gram of oil. Increases in acid value indicates the oxidation of oil which leads to gum and sludge formation besides corrosion. The acid value of CSO and CBD found to be 10.55, mg KOH/g, and 0.44, mg KOH/g respectively. The acid value of CBD was within the acceptable limits of standard specification. The FFA content of CSO found to be 5.30% (corresponding to acid value of 10.55, mg KOH/g), the biodiesel was produced by two steps, acid pretreatment followed by base-catalyzed transesterification process. In the first step the mixture of oil and methanol was treated with H_2SO_4 and the FFA content was reduced to 0.96%. In the second step by base-catalyzed transesterification process the biodiesel was produced successfully with a yield of 83.50% as calculated using Eq. (5), the yield found to be lower. This may be attributed to the presence of high amount of FFA, as it produces more amount of soap hence lower yield of biodiesel.

3.3.3 Flash point: Flash point gives an idea about, nature of boiling point diagram of the system, the presence of highly volatile and flammable materials, the tendency of oil to form a flammable mixture with air, and explosion hazards during storage and handling. The flash point of CSO, CBD and diesel were 232, 158, and 54 degree Celsius respectively. The flash point of CSO and CBD found to be much higher in comparison with diesel, which helps in safe storage and transportation.

3.3.4 Density: The density of any fuel establishes the relationship between mass and volume, and it affects the efficiency of atomization of fuel. The density of CSO and CBD found to be 920 kg/m^3 **and 870 kg/m^3** respectively. The density found to be higher than that of diesel, which may be due to the presence of higher molecular weight triglycerides.

3.3.5 Kinematic viscosity: Viscosity of oil is an important property because, as it affects for flow of oil through pipelines, injector nozzles and orifices, and also affects the proper atomization of fuel in the cylinder, if range found to be more causes pumping pressures, and it decides the pump clearance, if out of range leads to pump seizer. The kinematic viscosity of CSO was found to be 47.94, mm^2/s, which is much higher than that of diesel, hence the direct use of CSO may lead to poor combustion, untimely wear of fuel pumps and injector. The viscosity of CSO was reduced by converting it to biodiesel and it was found to be 5.11, mm^2/s, which is within the limits of standard specification for biodiesel fuel.

3.3.6 Calorific value: The calorific value measures the available energy in a fuel and a critical property of fuel intended for use in weight-limited vehicles. Calorific value of CSO, CBD and diesel were found to be 37.95, 40.48, and 43.00 MJ/kg respectively. The calorific valve of studied oil and biodiesel found to be lower than that of diesel, which may be due to the difference in chemical composition or presence of oxygen molecule in molecular structure of oil.

Table 3 Physicochemical properties of CSO and CBD in comparison with commercially available diesel, Simarouba oil, Simarouba biodiesel and standard specifications

Property	CSO	CBD	Simarouba oil [30]	Simarouba biodiesel [30]	Commercially available diesel	Standards specifications for biodiesel fuel, ASTM D6751-09a	Test Method
Iodine value (mg I$_2$/g)	122.71	----	N/A	----	----	----	----
Saponification number (mg KOH/g of oil)	209.17	----	N/A	----	----	----	----
Acid number (mg KOH/g)	10.55	0.44	12.49	0.36	----	0.50 max	D5555-95
Flash point (°C)	232	158	237	152	54	130 min	D93-10
Density (kg/m^3)	920	870	908	878	830	870-900	D1298-99
Kinematic viscosity (mm^2/s) at 40°C	47.94	5.11	45.59	5.09	2.4	1.9-6.0	D445-09
Calorific value (MJ/kg)	36.28	39.51	36.24	40.62	43	----	D240-09
Cetane number	44.79	----	N/A	----	----	47 min	----
Calculated cetane index	----	53.50	----	N/A	50.98	47 min	D976-06
Sulfated ash (w/w, %)	----	0.001	----	N/A	----	0.020 max	D874-07
Carbon residue (w/w, %)	0.79	0.05		Nil	----	0.050 max	D189-06
Copper strip corrosion	----	3h,50°C/1a		3h,50°C/1a	----	No. 3 max	D130-04
Distillation temperature, 90% recovered (°C)				N/A		360 max	D86-09
Initial Boiling Point (IBP)		335			161		
10%		340			202		
20%		342			215		
50%		347			254		
90%		354			348		
Molecular weight (g/mol)	840.08	----				----	

3.3.7 Cetane number and calculated cetane index: Cetane number is used to measure of ignition quality of diesel fuels, high cetane number implies short ignition delay and it influences both gaseous and particulate emissions. Cetane index which is very close to cetane number is calculated based on 10, 50, 90% distillation temperatures and specific gravity. Cetane number of CSO calclated using Eq. (4) and found to be 44.79 which is lower in comparison with the standard specifications. The cetane index calculated using the distillation characteristics for CBD and diesel were found to be 53.50 and 50.98 respectively. Both are higher than the limits of standard specifications.

3.3.8 Sulfated ash: In order to indicate the concentration of known metal-containing additives in oils the sulfated ash being used. The sulfated ash of CBD found to be 0.001 w/w % which is within the limits.

3.3.9 Carbon residue: Carbon residue is the percentage of amount of carbon (coke) left by heating the oil to a high temperature in absence of air. It is a measure of the amount of carbonaceous deposits in the combustion chamber. The carbon residue for CBD was 0.05% which agrees the standards.

3.3.10 Copper strip corrosion: The oils contain the varying amount of sulfur and sulfur compounds. The copper strip corrosion test is a measure to assess relative degree of corrosiveness and it also indicates the presence of sulfur compounds. The copper strip corrosion test being conducted for CBD and it was found to meet the required standards specified.

4. CONCLUSIONS

The purpose of the present study was to investigate the fatty acid composition and biodiesel characterization of non-edible, renewable, vegetable oil of CSO for use in diesel engine. Based on the study the following conclusions were drawn:

The GC-MS analysis of methyl esters of oils shows that the major fatty acids found in CSO were, palmatic , 32.52%, linoleic, 30.72%, palmitoleic, 13.39%, stearic, 8.88%, oleic , 6.03%, palmitelaidic, 5.18%, ecosenic, 0.71% and myristic, 0.57%.

The CSO was converted successfully into biodiesel by the two step process, acid pretreatment followed by base-catalyzed transesterification process and the biodiesel yield at the process parameters was found to be 83.50%.

The physical and chemical properties of biodiesel produced were found to be close to that of diesel fuel and also they meet the ASTM standard specifications for biodiesel, hence it can be used for diesel engine in place of conventional diesel fuel.

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