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Optimization and Characterization of Novel and Non-Edible Seed Oil Sources for Biodiesel Production

Inam Ullah Khan and Syed Aftab Hussain Shah

Abstract

Biodiesel mainly comes from edible oil, and there is little research on its yield from non-edible sources with low-cost oil. It is paramount to investigate the non-edible oil resources which may lead to advance the commercial feasibility of biodiesel and cost effectiveness as well as resolve the food issues. This chapter describes four novel non-edible seed oil sources comprising *Koelreuteria paniculata*, *Rhus typhina*, *Acacia farnesiana* and *Albizzia julibrissin* for biodiesel production. We aimed to optimize different reaction parameters for oil extraction, alkali-catalyzed transesterification process for maximal biodiesel production and finally evaluate its compatibility with mineral diesel. The optimization factors in transesterification included the molar ratio of methanol to oil, reaction time, stirring intensity, catalyst concentration and temperature. Two methods have been described including Soxhlet and mechanical for extraction of seed oil. The synthesized esters were evaluated and characterized through the nuclear magnetic resonance (NMR; ^1H and ^{13}C), Fourier transform infrared (FT-IR) and gas chromatography–mass spectrometry (GC–MS) and the total conversion of crude oil to fatty acid methyl esters (FAMES) were established. The inductively coupled plasma-optical emission spectrometry (ICP–OES) and Elemental Analyzer (EA) were used for evaluation of elemental concentration. The physico-chemical characterizations of the biodiesel, i.e., flash point, pour point, cloud point, and density were within the American Society for Testing and Materials (ASTM; D6751) and European Standards ((EN14214). *Koelreuteria paniculata* produced highest biodiesel oil content by Soxhlet extraction (28–30%) followed by the *Albizzia julibrissin* (19–24%), *Acacia farnesiana* (23%), *Rhus typhina* (20–22%). The density ranged from 0.83–0.87 @ 15°C (g/cm³) and the kinematic viscosity ranged from 3.75–6.3 (mm²/s) among all the plant sources. *Koelreuteria paniculata* had highest Na (5456.2), Cr (1246.8), Ni (658.36), and Al (346.87) elemental concentrations (µg/g) than other plant sources. The elemental percent of C, H, N, and O of biodiesel ranged from 72.54–76.86, 11.25–13.34, 1.97–2.73, and 9.86–12, respectively. In conclusion, these non-edible plant seeds offer a cheap source of renewable energy and can be easily grown on barren and wastelands and contribute to efficient biodiesel production to mitigate the energy crisis.

Keywords: *Acacia farnesiana*, *Albizzia julibrissin*, *Rhus typhina*, *Koelreuteria paniculata*, Biodiesel

1. Introduction

The global energy need has been confronting major challenges owing to population growth and industrialization [1, 2]. Green house gases and their emissions as well as developing energy safety mechanisms have perpetually turned the focus on research and technological development in this sector. The researcher community is applying renewable energy practices as an alternate to petroleum fuels with bio-diesel, bioethanol, biomass, biogas, and synthetic fuels with the aim to curtail net CO₂ emission, and improve air, soil, water and global warming [3]. The American Biodiesel Standard Specification (ASTM 6751) defines biodiesel (also named fatty acid methyl ester; FAMES) as fuel comprising of monoalkyl esters of long-chain fatty acids acquired from vegetable oils or animal fats [4]. The International Energy Agency (IEA) provided the estimates about global market share of biofuels to be increased from 1% (2004) to 7% by 2030 [5]. The need for utilizing biodiesel is associated with its lower exhaust emissions (CO_x, SO_x) and particulate matter [6]. Moreover, it possesses tremendous biodegradability [7], lubricity, storage [8], and higher flash point [6], oxygen content than diesel [9–11]. The higher oxygen content reflects the low carbon emissions, particulate emissions, CO, aromatic hydrocarbons, sulfur, smoke, and noise [12]. The major issues for biodiesel production and commercialization from vegetable oils comprise their availability and manufacturing cost [13].

The raw materials of biodiesel can be classified into three major groups including vegetable oil (edible or non-edible oil), animal fat, and edible waste oil [14]. These sources possess triglycerides [15] which carry great potential. Biodiesel obtained from vegetable oil has a viable market share in USA and European countries [16]. The scientific community is facing eminent challenge remains for suitable raw materials, their extraction and finally characterization for efficient and cost-effective biodiesel production. The transesterification [17] is a specialized method for biodiesel production from vegetable sources through conversion of one ester to another having low viscosity than the mineral diesel. The transesterification reaction involves catalyst between triglycerides, and short-chain alcohols, which produce monoesters, branched-chain, and long-chain triglyceride molecules that are further converted into glycerol and monoesters [18]. The three-step reaction forms monoglycerides and diglycerides as intermediates. As methanol contains lower charge, it is efficiently used for commercial production of biodiesel. Potassium hydroxide (KOH) as a predominate role in transesterification reaction [19]. The palm, sunflower, coconut rapeseed, soybean, and flaxseed are some of the raw materials being employed for commercialization [20]. Vegetable oil contains complex structure so it cannot be directly used in diesel engines and it will further aggravate the food supply chain through depletion of forests and wildlife destruction. Thus, impetus, toward non-edible sources, has been shifted for biodiesel production.

Feedstock has greater significance for ample availability of biodiesel [21]. The redeeming traits of non-edible sources include their toxicity, no utility in human food as it contains Erucic acid as major constituent of fatty acid; 56–66% [22], and its easy cultivation on poor soils [23], and cost-effectiveness. Moreover, it is very stable and possesses low melting point [24]. Biomass is a major energy source covering almost 10–14% of global need due to its easy combustion, less pollution and lower ash content [8]. However, it has equally low calorific value, thermal efficiency (10 to 15%), and comparatively large volume and transportation issues [8]. Chemically, biomass energy can be converted into liquid and gaseous forms [25].

Many studies have been conducted to explore the non-edible sources for bio-diesel production comprising *Croton megalocarpus* [26], *Prunus dulcis* [27], *Prunus sibirica* [28], *Rhazya stricta* Decne [29], rubber seed oil [30], *Silybum marianum* L. [31], wild *Brassica juncea* L. [32], *Jatropha curcas* and *Karanja* [33–35], waste tallow [36], and notably, algae [37]. However, high-quality biodiesel production still remains to decipher from existing economical non-edible sources [38].

2. Description of non-edible plant sources

Four novel and non-edible plant sources have been explored for bio-diesel production comprising *Koelreuteria paniculata* [39], *Rhus typhina* [40], *Acacia farnesiana* [41], and *Albizia julibrissin* [42]. The plant seeds were collected from China (Urumqi, Binhai new area near Nankai University's new campus Tianjin) and Pakistan (Lakki Marwat and Islamabad).

2.1 *Koelreuteria paniculata*

Koelreuteria paniculata belongs to the family Sapindaceae. It is a novel non-edible seed oil source that can be investigated for biodiesel production (Figure 1). It is less likely that pests may destroy it. It can grow at different soil

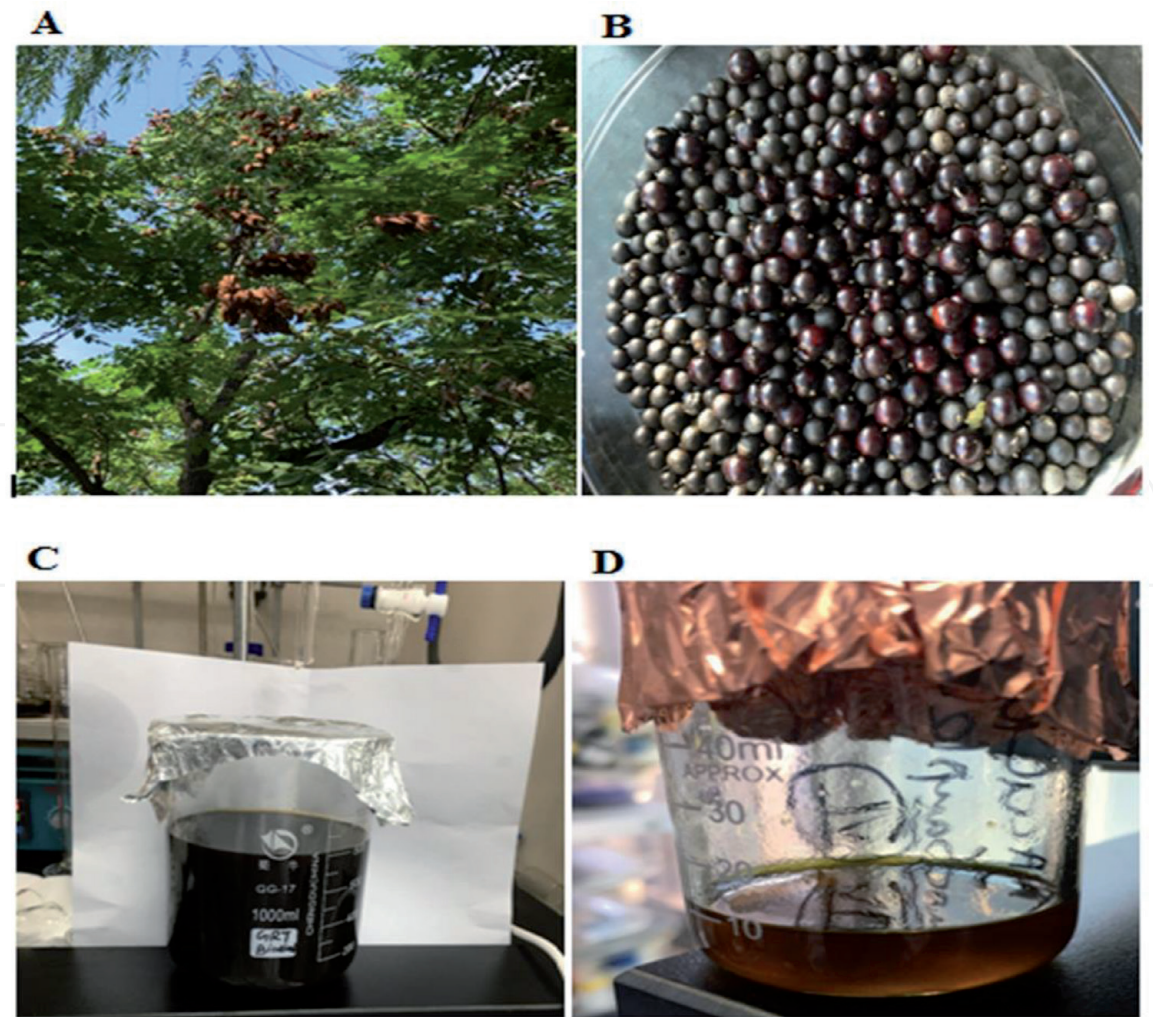


Figure 1.
Koelreuteria paniculata. (A) Plant; (B) seeds; (C) biodiesel; (D) glycerin.

environments, even at high pH soils. It possesses gorgeous inflorescence with ellipsoid pods and contains abundance of seeds. About 15–20kg seeds are produced annually from a single plant. The feasibility of planting *K. paniculata* trees ranges from 400 to 500 per hectare area with average production of 115,000 kg seeds and 30,000 kg per hectare oil production. *K. paniculata* species have origin from China, Japan, and Korea. *K. paniculata* is often used as an ornamental plant and declared as an invasive due to the inexhaustible seed production and offspring. *K. paniculata* is suitable to grow in unused and desolate lands and its seeds can be effectively used as alternate energy source, which contain 28–30% oil constituents [39, 43].

2.2 *Rhus typhina* L.

Rhus typhina L. (Staghorn sumac) is belongs to Anacardiaceae family (Figure 2). It has greater distribution in subtropical and temperate regions around the world, notably in Africa and North America. It has a maximum height ranging from 30 to 35 feet. Its seeds contain enormous potential as feedstock for biodiesel production [40]. *R. typhina* plant grows rapidly and produces abundance of seeds. About 300–400 trees can be planted, with approximate 78,000 kg seeds

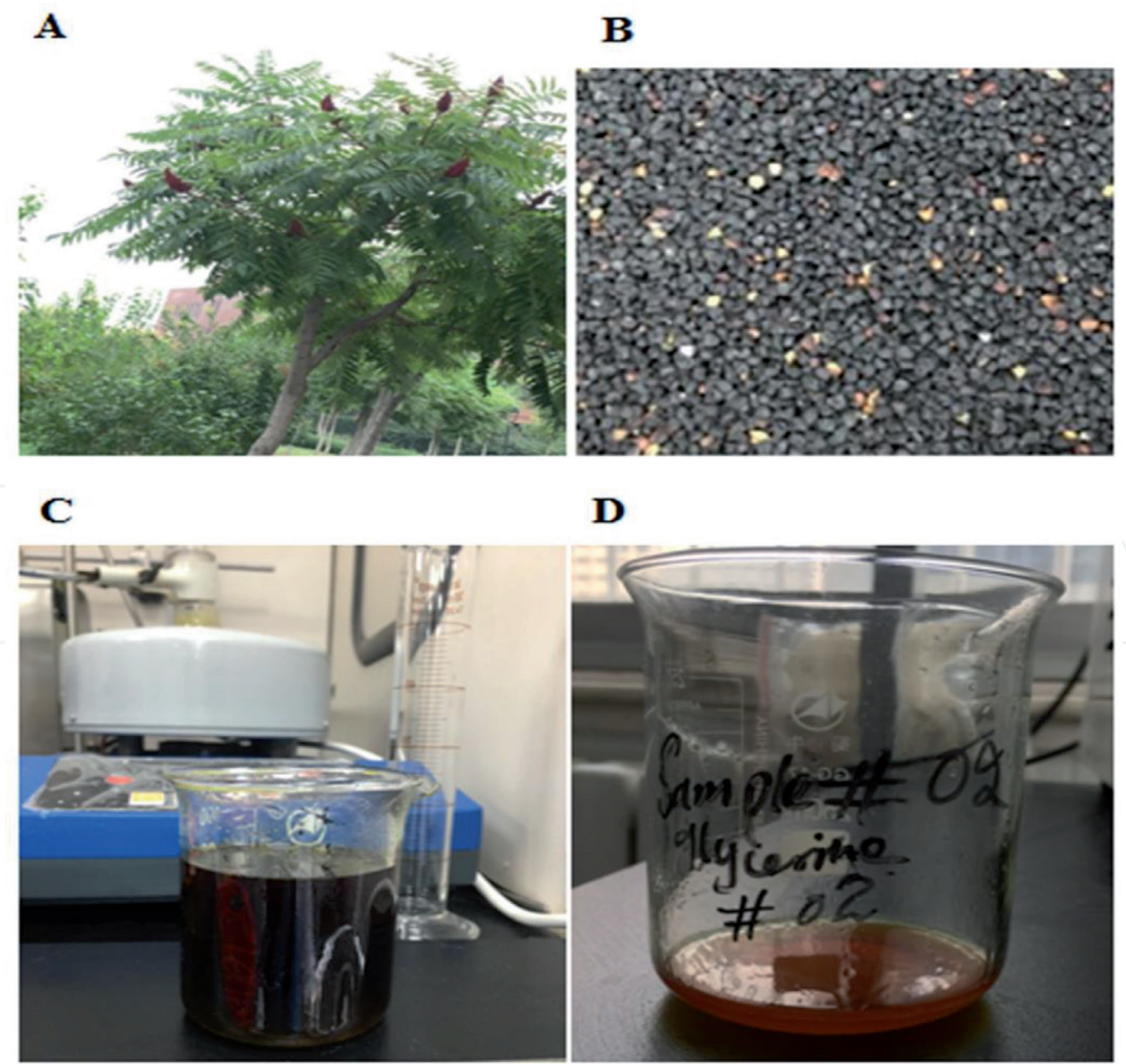


Figure 2.
Rhus typhina (staghorn sumac). (A) Plant; (B) seeds; (C) biodiesel; (D) glycerin.

and 17,160 kg production per hectare, respectively. *R. typhina* oil yield has been reported as 9% w/w on the basis of acetone/water extract 9:1 v/v [44] and 12% [45]; wherein we obtained 22% oil contents [40].

2.3 *Acacia farnesiana* L.

Acacia farnesiana L. belongs to family Leguminosae (Mimosoideae) and it is native to North America [46]. This tall semi-evergreen native shrub or small tree is commonly referred to as sweet acacia, Huisache, etc., with soft, medium-green feather-like, finely divided small leaves. The slightly thick stem is rich in chocolate brown or gray, with long and pointed needles. The small, puff-like yellow flowers are very fragrant, appear in clusters in late winter, and then occasionally spread out after each new flush, providing nearly four seasons of flowering. An area of about one hectare wills 91,500 kg of seeds yield, and the efficiency of oil per hectare is approximately 21,250 kg. The fruit is an elongated pod, 3 to 6 inches long, dry, and covered with hard skin, brown. Green color attracts birds; squirrels and other mammals have no obvious littering problems and stick to the trees, which is very beautiful. The long-lasting fruit has a smooth appearance and contains seeds cherished by birds and other wildlife (Figure 3).

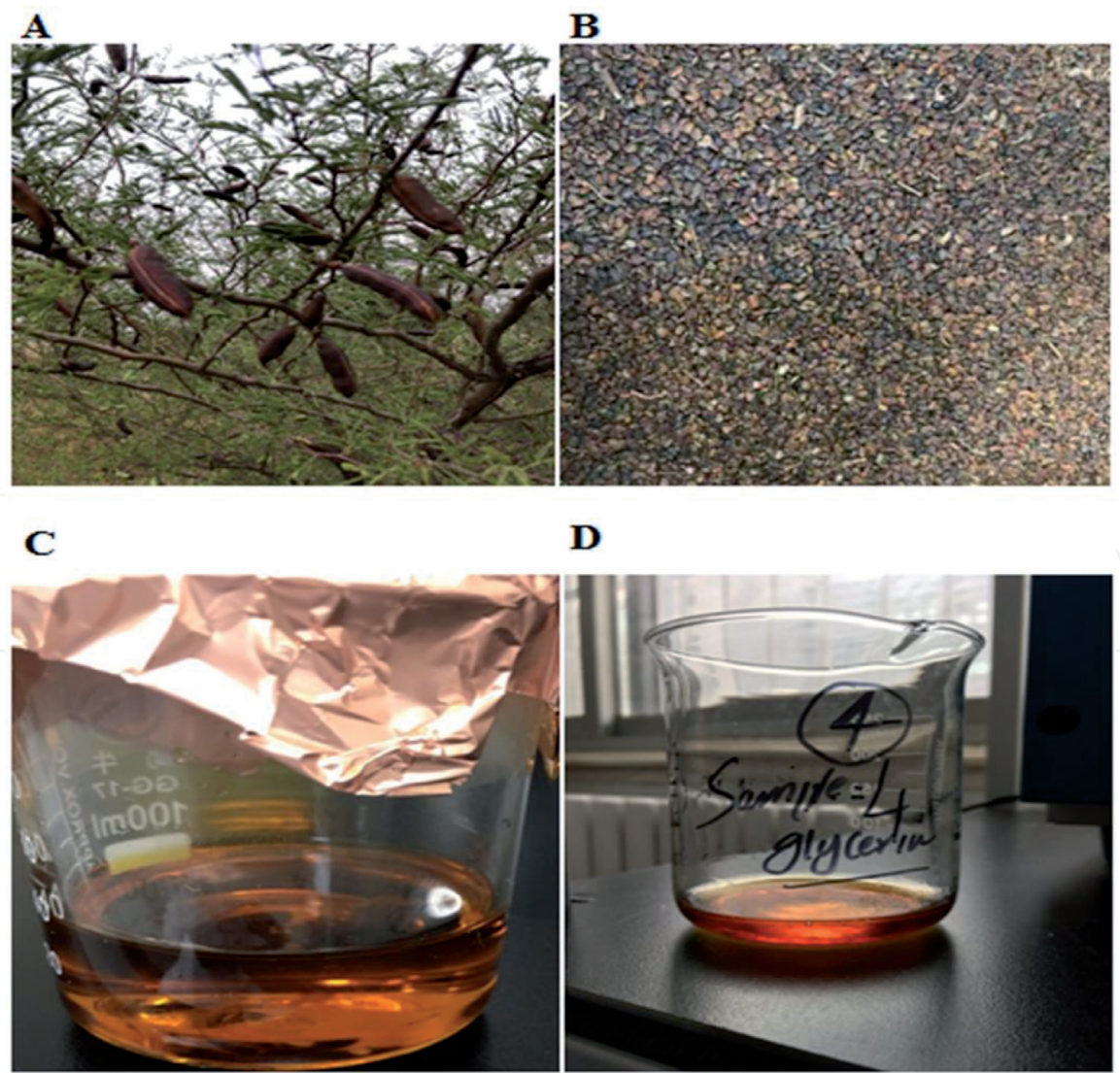


Figure 3.
Acacia farnesiana. (A) Plant; (B) seeds; (C) biodiesel; (D) glycerin.

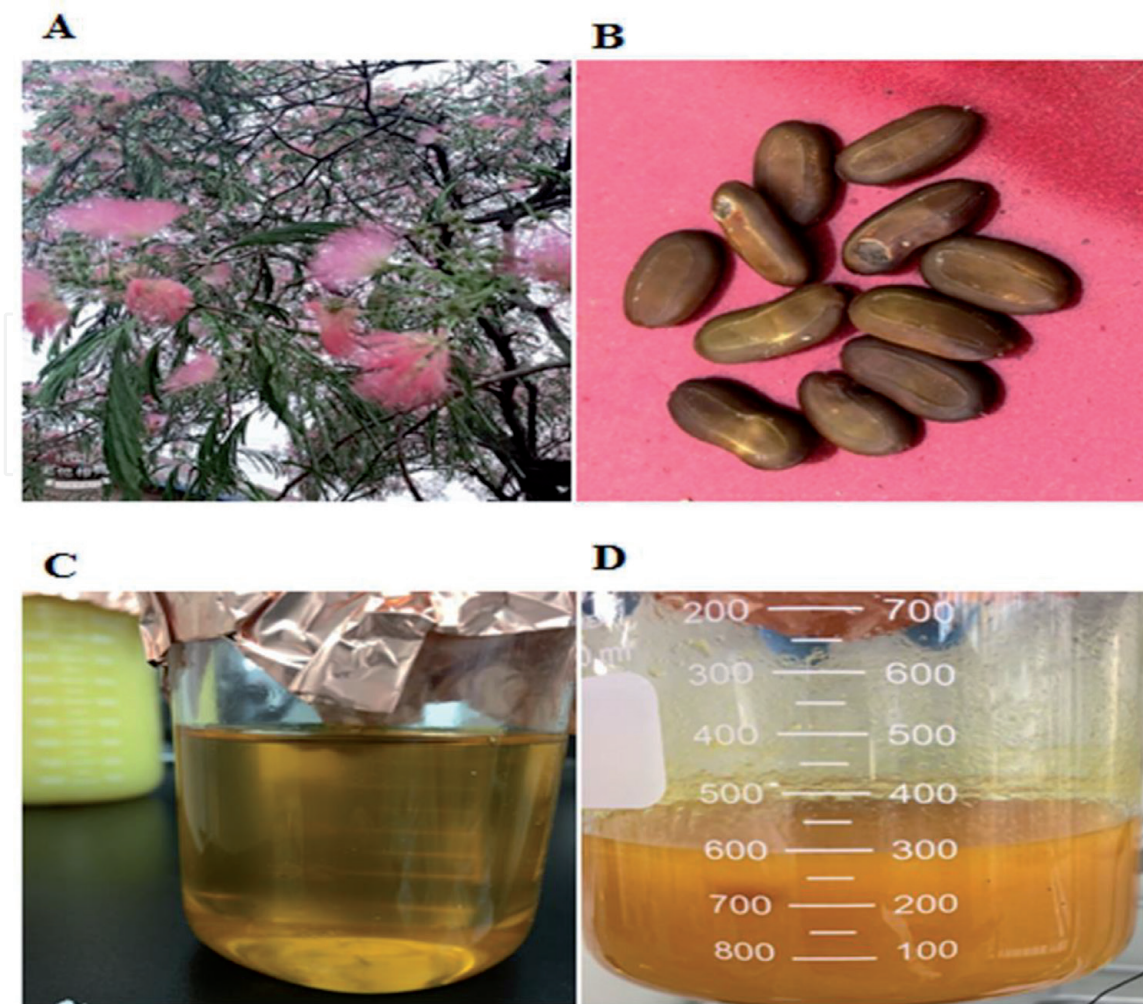


Figure 4.
Albizzia julibrissin. (A) Plant; (B) seeds; (C) biodiesel; (D) glycerin.

2.4 *Albizzia julibrissin*

One of the plants, *Albizzia julibrissin* (Mimosa) belong to Fabaceae, which contains non-edible seeds, can be evaluated for biodiesel production (**Figure 4**). *A. julibrissin* is a deciduous tree, wide and 12 m (40ft) high, but usually 3–6 m (10–20ft), smooth bark, gray. *A. julibrissin* fruit comprises of lentil pods with enlarged seeds. Each pod is 8–18 cm long and 1.5–2.5 cm wide and can be observed from June to February. Each pod usually grows 5–10 elliptical seeds, about 1.25 in length. Some authors assert that mimosa produces many seeds [47]. *A. julibrissin* can produce 8,000 seeds per year. The average *A. julibrissin* seed per pound is 11,000 to 11,500 [48, 49]. Wind, gravity, and water are major contributory factors for dispersal of seeds and pods. *A. julibrissin* is native to Asia and found in Turkey, Azerbaijan, China, Japan, Taiwan, and other temperate regions, Bhutan in Asia, India, Nepal, Pakistan, Myanmar, Japan and other tropical areas [42, 50].

3. Oil extraction methodologies and outcomes from non-edible plant sources

3.1 Oil extraction

After shelling, drying and pulverizing with a grinder (Xiantaopai XTP-10000A, Zhejiang, China) the seeds were processed for extraction. The oil extraction from

Acacia farnesiana, *Albizia julibrissin*, *Rhus typhina* and *Koelreuteria paniculata* was carried by Soxhlet (chemical; 90°C for 7 h using different solvents; **Figure 5A**) and mechanical extraction of by two different electric oil expeller machines comprising Fangtai Shibayoufang FL-S2017 China (less power extractor) and Fangtai Shibayoufang J508, China (high power extractor), (**Figure 5B**). Pre-treatment of seed is essential for mechanical and Soxhlet extraction, which can increase the amount of oil recovery. After 2–3 revolutions, a considerable yield of crude seed oil was obtained. The oil removed from the seed by mechanical presses requires additional handling of extraction and filtration to produce a purer raw feedstock. Further following steps were conducted to get the biodiesel from these sources comprising filtration, rotary evaporation for access methanol, heating, transesterification, settling, separation, and washing.

The oil production was calculated by the following equation.

$$\text{Conversion\%} = \text{Obtained seed oil weight (g)} / \text{Total seed weight (g)} \times 100 \quad (1)$$

The comparative oil content (%) of 4 plants, obtained by using mechanical oil extraction and Soxhlet extraction methods from four plant sources is given in **Table 1**.

3.2 Fourier-transform infrared spectroscopy (FT-IR)

FT-IR spectroscopy data of the mid-infrared region of biodiesel samples to recognize functional groups and the bands analogous to various stretching and bending vibrations is highlighted in **Table 2**.

3.3 Nuclear magnetic resonance (NMR)

The FAMES NMR spectrum was acquired by (Bruker Avance III 400 NMR Spectrometer, Karlsruhe, Germany) at 400 MHz (^1H -NMR) or 100 MHz (^{13}C -NMR). Denatured chloroform was used as solvent and tetramethylsilane as the internal standard. The biodiesel ^1H NMR (300 MHz) spectrum was noted with a cycle delay of 1.0 s, and eight times scans with a pulse duration of 30°, (**Table 3**). A carbon ^{13}C NMR (75 MHz) spectrum was recorded with pulse duration of 30° and a cycle delay of 1.89 s, followed by scanning for 160 times (**Table 4**).

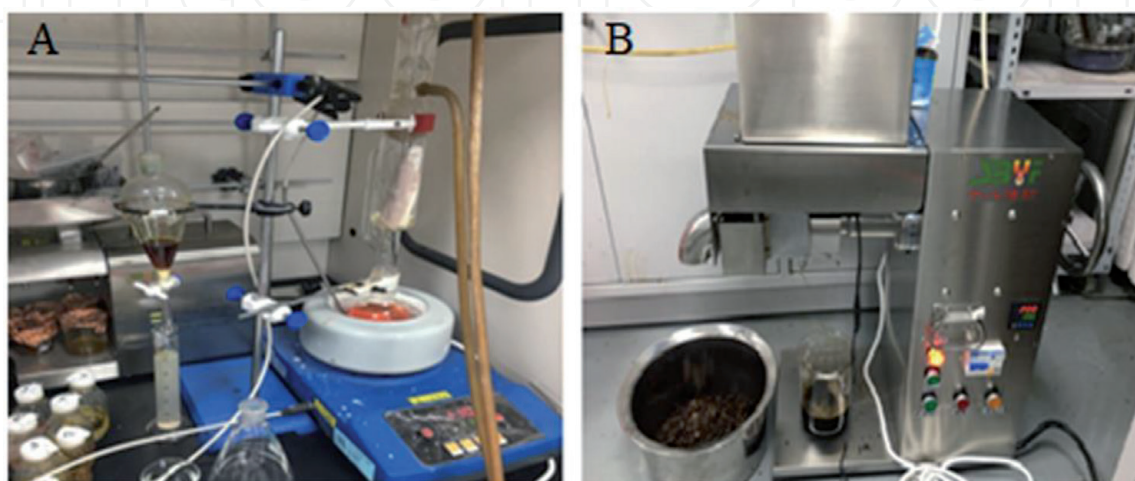


Figure 5.
(A) Instrument for Soxhlet extraction (chemical extraction); (B) instrument for mechanical oil extraction.

S. No.	Source Name	Mechanical Extraction (%)	Soxhlet extraction (%)	FFAs content (%)
1	<i>Koelreuteria paniculata</i>	18.7	28–30	0.91
2	<i>Rhus typhina</i>	13.3	22	1.0
3	<i>Acacia farnesiana</i>	8.3	23	0.4
4	<i>Albizzia julibrissin</i>	9.9	19–24	0.9

Table 1.
The oil content (%) of 4 plants, using mechanical oil extraction and Soxhlet extraction methods.

Peak no.	Wave number (cm ⁻¹)	Group attribution	Vibration type	Absorption Intensity
1	3465	–OH	Stretching	Weak
2	3006	=C–H	Stretching	Strong
3	2925	–CH ₂	Asymmetric stretching vibration	Strong
4	2854	–CH ₂	Symmetric stretching vibration Stretching	Strong
5	1743	–C=O	Shear type vibration	Strong
6	1641	–CH ₂	Bending vibration	Middling
7	1361	–CH ₃	Symmetric stretching vibration,	Middling
8	1170	C–O–C	Anti-stretching vibrations	Middling
9	1016	C–O–C	Vibration	Weak
10	723	–CH ₂	Plane rocking vibration	Weak

Table 2.
FT-IR data presenting various functional groups in FAMES.

Integration value	Chemical Shift ppm	Multiplicity	Inferences
3	0.89	Multiplet	CH ₃ is attached to aliphatic group.
16	1.30	Multiplet	Long aliphatic chain is present.
2	1.62	Quartet	CH ₂ group is attached with terminal CH ₃ .
3	2.04	Multiplet	CH ₂ of long chain aliphatic (Saturated) group.
2	2.30	Triplet	CH ₂ group is attached with CH of long aliphatic (Unsaturated/ olefinic group).
1	2.77	Triplet	CH group is attached with electron withdrawing carbonyl group.
3	3.66	Singlet	Methoxy(OCH ₃)group attached with electron withdrawing carbonyl group.
3	5.34	Multiplet	Olefinic hydrogen of long chain unsaturated aliphatic group

Table 3.
¹H NMR spectroscopic data depicting chemical composition of various methyl esters in biodiesel (FAMES) samples.

Peak No:	Peak area/ region/ ppm	Identified compound	Chemical structure
1	14.07	Terminal methyl carbon	-CH ₃
2	22.55–34.09	Methylene carbon	-CH ₂
3	51.37	Methoxy carbon	-OCH ₃
4	127.91–130.19	Olefinic carbon	C=C
5	174.24	Carboxyl carbon of ester	-COOCH ₃

Table 4.
¹³C NMR spectroscopic data depicting the chemical shift values matching to various structural features in FAMES.

3.4 GC–MS procedure

The outcome of biodiesel in our studies was evaluated by GCMS (QP2010SE, Shimadzu, Japan), furnished with a capillary column: PEG-20 M (30 m × 0.32 mm × 1 μm film thickness). Helium gas flow rate 1.2 mL/min; split ratio 40:1; the injector temperature and injection volume were 220°C and 1 uL; Furnace heat up mode was 100°C for 1 min, then from 100°C rises to 210°C at the increase rate of 10°C/min. Sensor heat mode was 210°C, and then for 20 min, the temperature was continuing at 210°C; ion source temperature of 200°C; for electron impact 70 eV ionization mode used; mass range of 35–500 m/z. The FAMES of all plant sources were identified with the mass spectrometry fragmentation design provided by the GCMS system software, as matched with those stored in the mass spectrometry library NIST14, and their fatty acid identity was further verified by matching with known standards and values [39–42].

The comparative GC based identified FAMES major compositions (%) of prepared biodiesel from four non-edible plant sources is given in **Table 5**.

3.5 ICP-OES procedure for elemental analysis in biodiesel

Inductively Coupled Plasma Spectrometer (Spectro-blue, Germany) and Elemental Analyzer (Vario EL CUBE, Germany) were used for the presence of metals in the biodiesel. For the ICP-OES test, 1 g of oil sample was taken for incinerating. The ashing process involved an increase in the oven temperature to 200°C in one hour; then the heat levels were maintained upto 500 °C for 2 h, and finally

FAMES major compositions (%)	Plant species			
	<i>Koelreuteria paniculata</i>	<i>Rhus typhina</i>	<i>Acacia farnesiana</i>	<i>Albizia julibrissin</i>
C16:0	9.7	14.0	6.85	10.598
C16:1	—	—	—	—
C18:0	1.8	3.2	2.36	2.12
C18:2	25.5	47.2	12.13	12.030
C18:3	3.6	1.1	1.23	—
C20:0	2.4	0.8	—	—
C20:1	48.5	0.5	—	—
C21:1	—	—	—	—

Table 5.
GC based identified FAMES major compositions (%) of prepared biodiesel.

to 800°C for 5 h. The ash was dissolved in 10 mL of 2% HNO₃. The prepared sample was used for elements finding and concentration test of the biodiesel.

The account of ICP-OES comparative element concentrations of 4 non edible oil plant species is given in **Table 6**.

3.6 Elemental analyzer (EA) procedure for elemental analysis

The element analyzer (Vario EL CUBE, Germany) was used to detect the H, N, C and O concentrations of biodiesel obtained from plant sources [39–42]. About 0.5 mL of biodiesel, 3 mL of concentrated HCl and 1 mL of nitric acid were taken in a tube and kept them at rest for 10–15min, to dissolve the oil in the solution. Fresh reagents can be used for sample preparation. The aqua regia amount was twice than the sample. About 1 mL of prepared solution was taken in a new tube and added deionized water making it up to 5 mL. The technique was repeated for 2–3 times until the sample appeared as clear and vivid and ready for evaluation of C, H, N, and O concentrations.

The comparative account of elemental analysis of biodiesel obtained from 4 non edible oil plant species is given in **Table 7**.

3.7 Physiochemical properties of biodiesel seed oil from four non edible oil plant species

The comparative account of physiochemical properties of biodiesel seed oil obtained from four non edible oil plant species is given in **Table 8**.

Elements	Concentration (µg/g)				
	Petro-diesel	KPOB	RTOB	AFOB	AJOB
Sb	—	5744.02	7845.2	—	—
Na	868.3	5456.2	70.29	868.3	868.3
K	213.3	6.14		213.3	213.3
Cr	2.5	1246.8	3.41	—	—
Ni	12.4	658.36	46.98	12.4	12.4
Al	—	346.87	55.56	—	—
Sn	—	378.61	838.6	—	—
Mn	1.5	92.05	5.58	1.5	1.5
Ti	—	64.40	–124.11	—	—
Li	1.6	43.93	105.5	1.6	—
V	—	42.79	15.42	—	—
Cu	99.6	24.63	31.82	—	—
Ca	21.4	14.90		21.4	21.4
Mg	35.6	32.10	32.74	35.6	35.6
Bi	—	19.90	29.05	—	—
Zn	9.5	13.08	49.89	9.5	—
Co	21.2	10.69	7.96	21.2	21.2
Cd	—	—	1.59	—	—

Abbreviations: *Koelreuteria paniculata* Biodiesel Oil (KPOB); *Rhus typhina* Biodiesel Oil (RTOB); *Acacia farnesiana* Biodiesel Oil (AFOB); *Albizia julibrissin* Biodiesel Oil (AJOB).

Table 6.
ICP-OES element concentrations of 4 non edible oil plant species.

Ultimate analysis	KPBD	RTBD	AFBD	AJBD
C%	72.54	74.89	76.37	76.86
H%	12.73	413.02	13.34	11.25
N%	2.73	1.97	2.18	2.03
O%	12		8.11	9.86
HHV	23.39		23.39	23.39

Abbreviations: *Koelreuteria paniculata* Biodiesel Oil (KPOB); *Rhus typhina* Biodiesel Oil (RTOB); *Acacia farnesiana* Biodiesel Oil (AFOB); *Albizzia julibrissin* Biodiesel Oil (AJOB).

Table 7.
Elemental analysis of biodiesel from 4 non edible oil plant species.

Parameters	EN 14214	ASTM D-6751	Petro-diesel	KPOB	RTOB	AFOB	AJOB
Oil contents (wt. %)	—	—	—	28–30	20–22	23	19–24
Density @ 15°C (g/cm ³)	0.86–0.90	0.86–0.90	0.809	0.879	0.879	0.831	0.842
Kinematic viscosity @ 40°C (mm ² /s)	3.5–5.0	1.9–6.0	1.3–4.1	6.21	6.3	5.32	3.75
Flashpoint (°C)	Min. 120	Min. 130	60–80	147	168	158	160
Ignition value	—	—	—	175	—	—	
Acid value (mg KOH/g ⁻¹)	Max. 0.50	Max. 0.5	—	0.07		0.40	
Saponification value (mg KOH/g ⁻¹)	—	—	—	176.4	175.6	174.8	180.4
Iodine value (g I ₂ /100 mg)	Max. 120	Max. 120	—	80.7	85	142.5	118.5
Refractive index @ 20 °C	—	—	—	1.4901	—	—	
Cloud point (°C)	—	—	–15–5	2	7	7	9
Pour point (°C)	—	—	–2.0	–30	–11	–28	–12
Fire point (°C)	—	—	—		198	189	190
Cetane number	Min. 51	Min. 47	49.7	51	—	52	58
Free fatty acid (%)	—	—	—	0.91	1.0	0.4	0.9
HHV (MJ/kg)	—	—	—	23.39	23.73	—	—
Ash content (g/100 g)	—	—	—	0.002	0.3	0.002	0.003
Specific gravity @15 (°C)	—	—	—	0.88	0.855	0.831	0.842
Cold filter plug point (°C)	Max.19	Max.19	–16	–18	14	–25	3
Sulphated ash content (wt.%)	Max.0.02	—	—	0.003	—	—	
Oxidation stability (110°C, h)	Min. 6	Min. 3	25.8	—	18.3	–1.86	4.71

Abbreviations: *Koelreuteria paniculata* Biodiesel Oil (KPOB); *Rhus typhina* Biodiesel Oil (RTOB); *Acacia farnesiana* Biodiesel Oil (AFOB); *Albizzia julibrissin* Biodiesel Oil (AJOB).

Table 8.
Physiochemical properties of biodiesel (FAMES) samples.

4. Discussion

The great potential exists for using non-edible seed oil as biodiesel, which has been highlighted in this chapter. The plant sources explored were *Koelreuteria paniculata*, *Rhus typhina*, *Acacia farnesiana* and *Albizzia julibrissin*.

Biodiesel can be synthesized from vegetable oils, animal fats or algae oils [51]. The Soxhlet extraction allows the sample to repeatedly bring into contact with fresh portions of extracting materials, hence facilitating the equilibrium. It remains at relatively high temperature and no filtration is required [52]. In the present study, *Koelreuteria paniculata* produced highest biodiesel oil content by Soxhlet extraction (28–30%) followed by the *Albizzia julibrissin* (19–24%), *Acacia farnesiana* (23%), *Rhus typhina* (20–22%) than the mechanical extraction.

The biodiesel production largely depends upon the choice of appropriate, cost-effective and environment friendly catalysts [53] based on the nature of oil which greatly help in transesterification of oil. Density optimization is a vital biodiesel factor having huge impact on fuel quality as well the cost incurred [54]. The density for all plant sources ranged from 0.83–0.87 @ 15°C (g/cm³). The kinematic viscosity ranged from 3.75–6.3 (mm²/s) among all the plant sources. Our results show that densities of all plant sources were within the ASTM (D6751) and (EN14214) standards.

The 1H-NMR procedure is used to evaluate the un-saturation and “residual” fatty acid composition [55]. In our study, the biodiesel 1H NMR (300 MHz) spectrum was noted with a cycle delay of 1.0 s, and eight times scans with a pulse duration of 30°. 13C NMR spectroscopic data represented the chemical shift values matching to various structural features in FAMES.

The metals such as Cu, Co, Fe, Mn and Ni are known to catalyze oxidative degradation reactions in vegetable oils and biodiesel [56]. The presence of some metals, such as Al, Ca, Cr, Cu, Fe, Mg, Pb, V and Zn, among other, in fuel is undesirable due to their release into the atmosphere upon fuel combustion [57]. *Koelreuteria paniculata* had highest Na (5456.2), Cr (1246.8), Ni (658.36), and Al (346.87) elemental concentrations (µg/g) than other plant sources. Moreover, Al, Cr, Sn, V, Cu, Bi, Cd were not detected in *Acacia farnesiana* and *Albizzia julibrissin* and both showed comparable Zn, K, Ni, Mn, Ca, and Co concentrations with petro diesel. The metal elements in biodiesel result into engine degradation, operational as well as cause environmental pollution [58]. In our study, the elemental percent of C, H, N, and O of biodiesel ranged from 72.54–76.86, 11.25–13.34, 1.97–2.73, and 9.86–12, respectively.

Flash point of fuels is imperative to determine the prerequisites for transportation and storage temperature [59]. The flash point was highest in the biodiesel oil of *Rhus typhina* (168°C), followed by *Albizzia julibrissin* (160°C), *Acacia farnesiana* (158°C), *Koelreuteria paniculata* (147°C) which was comparable to EN 14214 (Min. 120°C), ASTM D-6751 (Min. 130°C) petro diesel (60–80°C) standards.

The main shortcomings of biodiesel are related to the low-temperature performance and oxidation stability. The oxidation stability (110°C, h) was recorded as 18.3 in *Rhus typhina*, 4.71 in *Albizzia julibrissin*, –1.86 in *Acacia farnesiana* and these fall in the recommended values of EN 14214 (Min. 6), ASTM D-6751 (Min. 3) and petro diesel (25.8). This highlights that the saturated fraction of biodiesel has a positive effect on its stability as in the case of our results, but the long-chain saturates may compromise fuel performance at lower temperatures [60].

Metal-containing fuel additives and un-removed catalysts are the major contributors of sulfated ash [61]. The sulphated ash content (%) was not detected in *Rhus typhina*, *Acacia farnesiana*, *Albizzia julibrissin* and it was 0.003 in *Koelreuteria*

paniculata as compared to the EN 14214 (Max.0.02) standards, hence these plant sources can be used for biodiesel production.

5. Conclusions

The physico-chemical characterizations of the biodiesel, i.e., flash point, pour point, cloud point, and density, have been found within the ASTM (D6751) and (EN14214) standards. The values of fuel properties were comparable with mineral diesel. FT-IR, NMR, and GC-MS analysis established the total conversion of crude oil to FAMES. The elemental analysis of biodiesel ensured the feasibility for environment friendly usage. These sources have been optimized through optimization of transesterification reactions: oil to methanol ratio (6:1), a potassium hydroxide concentration (3.0%), temperature (65°C), stirring rate (700 rpm) and reaction time (60–80 min). The highest biodiesel yield was obtained from *Albizzia julibrissin* (98%) followed by *Acacia farnesiana* (96%), *Koelreuteria paniculata* (95.2%) and *Koelreuteria paniculata* (93.33%). FAMES of all sources were compatible to ASTM (D6751) and (EN14214) standards. These non-edible plant seeds offer a cheap source of renewable energy. These plants can be easily grown on barren and waste-lands and contribute to efficient biodiesel production to curtail the energy crisis. Keeping these findings in preview, we can assert that biodiesel obtained from these non-edible seeds has a huge potential as an alternative to petroleum diesel and can be efficient renewable source of fuel.

Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this chapter.

Authors' contributions

Inam Ullah Khan prepared the first draft of the chapter based on his published research work and other relevant literature. Syed Aftab Hussain Shah edited, formatted the entire chapter and worked on bibliography.

Ethical statement

This is to certify that chapter has adhered to the research ethics.

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References

- [1] Röllin H. Evidence for health effects of early life exposure to indoor air pollutants: What we know and what can be done. *Clean Air J.* 2017;27(1).
- [2] Xue B-j, Luo J, Zhang F, Fang Z. Biodiesel production from soybean and *Jatropha* oils by magnetic CaFe_2O_4 – $\text{Ca}_2\text{Fe}_2\text{O}_5$ -based catalyst. *Energy.* 2014;68:584-91.
- [3] Samadi S, Karimi K, Behnam S. Simultaneous biosorption and bioethanol production from lead-contaminated media by *Mucor indicus*. *Biofuel Res. J.* 2017;4(1):545.
- [4] Knothe G, Krahel J, Van Gerpen J. *The Biodiesel Handbook*: Elsevier; 2015.
- [5] Garcez CAG, de Souza Vianna JN. Brazilian biodiesel policy: social and environmental considerations of sustainability. *Energy.* 2009;34(5):645-654.
- [6] Živković SB, Veljković MV, Banković-Ilić IB, Krstić IM, Konstantinović SS, Ilić SB, et al. Technological, technical, economic, environmental, social, human health risk, toxicological and policy considerations of biodiesel production and use. *Renew. Sustain. Energy Rev.* 2017;79:222-247.
- [7] Tremblay AY, Montpetit A. The in-process removal of sterol glycosides by ultrafiltration in biodiesel production. *Biofuel Res. J.* 2017;4(1):559-564.
- [8] Sharma Y, Singh B, Upadhyay S. Advancements in development and characterization of biodiesel: a review. *Fuel.* 2008;87(12):2355-2373.
- [9] Song H, Quinton KS, Peng Z, Zhao H, Ladommatos N. Effects of oxygen content of fuels on combustion and emissions of diesel engines. *Energies.* 2016;9(1):28.
- [10] Nakano M, Okawa K. Study of oxygen-containing hydrocarbons in exhaust emission from a spark ignition combustion engine. *Int. J. Engine Res.* 2014;15(5):572-580.
- [11] Mwangi JK, Lee W-J, Chang Y-C, Chen C-Y, Wang L-C. An overview: Energy saving and pollution reduction by using green fuel blends in diesel engines. *Appl. Energy.* 2015;159:214-236.
- [12] Murayama T. Evaluating vegetable oils as a diesel fuel. 1994;5:1138-1145.
- [13] Knothe G, Gerpen J, Krahel J. *The Biodiesel Handbook*. Champaign-Illinois: AOCS Press; 2005. p. 89-90.
- [14] Verma D, Raj J, Pal A, Jain M. A critical review on production of biodiesel from various feedstocks. *J. Scient. Innov. Res.* 2016;5(2):51-58.
- [15] Roeges NP. A guide to the complete interpretation of infrared spectra of organic structures: J. Wiley; 1994. p. 1-356.
- [16] Akinsiku AA. Biodiesel fuel from differently sourced local seed oils: characterization, effects of catalysts, total glycerol content and flow rates. *Int. J. Scient. Eng. Res.* 2013;4(6):654-660.
- [17] Otera J. Transesterification. *Chemical Rev.* 1993;93(4):1449-1470.
- [18] Demirbas A. Biodiesel production from vegetable oils via catalytic and non-catalytic supercritical methanol transesterification methods. *Prog. Energy Combust. Sci.* 2005;31(5-6):466-487.
- [19] Ma F, Hanna MA. Biodiesel production: a review. *Bioresour. Technol.* 1999;70(1):1-15.
- [20] Körbitz W. Biodiesel production in Europe and North America, an

encouraging prospect. *Renew. Energy*. 1999;16(1-4):1078-1083.

[21] Sharma Y, Singh B. Development of biodiesel: current scenario. *Renew. Sustain. Energy Rev*. 2009;13(6-7):1646-1651.

[22] Wazilewski WT, Bariccatti RA, Martins GI, Secco D, de Souza SNM, Rosa HA, et al. Study of the methyl crambe (*Crambe abyssinica* Hochst) and soybean biodiesel oxidative stability. *Ind. Crops Prod*. 2013;43:207-212.

[23] Gui MM, Lee K, Bhatia S. Feasibility of edible oil vs. non-edible oil vs. waste edible oil as biodiesel feedstock. *Energy*. 2008;33(11):1646-1653.

[24] Vargas-Lopez J, Wiesenborn D, Tostenson K, Cihacek L. Processing of crambe for oil and isolation of erucic acid. *J. Am. Oil Chem. Soc*. 1999;76(7):801-809.

[25] McKendry P. Energy production from biomass (part 2): conversion technologies. *Bioresour. Technol*. 2002;83(1):47-54.

[26] Aliyu B, Agnew B, Douglas S. Croton megalocarpus (Musine) seeds as a potential source of bio-diesel. *Biomass and Bioenergy*. 2010;34(10):1495-1499.

[27] Atapour M, Kariminia H-R. Characterization and transesterification of Iranian bitter almond oil for biodiesel production. *Appl. Energy*. 2011;88(7):2377-2381.

[28] Wang L, Yu H. Biodiesel from Siberian apricot (*Prunus sibirica* L.) seed kernel oil. *Bioresour. Technol*. 2012;112:355-8.

[29] Nehdi IA, Sbihi HM, Al-Resayes SI. *Rhazya stricta* Decne seed oil as an alternative, non-conventional feedstock for biodiesel production. *Energy Convers. Manag*. 2014;81:400-406.

[30] Reshad AS, Tiwari P, Goud VV. Extraction of oil from rubber seeds for biodiesel application: Optimization of parameters. *Fuel*. 2015;150:636-644.

[31] Fadhil AB, Aziz AM, Al-Tamer MH. Biodiesel production from *Silybum marianum* L. seed oil with high FFA content using sulfonated carbon catalyst for esterification and base catalyst for transesterification. *Energy Convers. Manag*. 2016;108:255-265.

[32] Al-dobouni IA, Fadhil AB, Saeed IK. Optimized alkali-catalyzed transesterification of wild mustard (*Brassica juncea* L.) seed oil. *Energy Sources, Part A: recovery, utilization, and environmental effects*. 2016;38(15):2319-2325.

[33] Rashed M, Kalam M, Masjuki H, Mofijur M, Rasul M, Zulkifli N. Performance and emission characteristics of a diesel engine fueled with palm, jatropha, and moringa oil methyl ester. *Ind. Crops Prod*. 2016;79:70-76.

[34] Sinha P, Islam MA, Negi MS, Tripathi SB. Changes in oil content and fatty acid composition in *Jatropha curcas* during seed development. *Ind. Crops Prod*. 2015;77:508-510.

[35] Foidl N, Foidl G, Sanchez M, Mittelbach M, Hackel S. *Jatropha curcas* L. as a source for the production of biofuel in Nicaragua. *Bioresour. Technol*. 1996;58(1):77-82.

[36] Bhatti HN, Hanif MA, Qasim M. Biodiesel production from waste tallow. *Fuel*. 2008;87(13-14):2961-2966.

[37] Chisti Y. Biodiesel from microalgae. *Biotechnology advances*. 2007;25(3):294-306.

[38] Chen Y-H, Chen J-H, Luo Y-M. Complementary biodiesel combination from tung and medium-chain fatty acid oils. *Renew. Energy*. 2012;44:305-310.

- [39] Khan IU, Yan Z, Chen J. Production and characterization of biodiesel derived from a novel source *Koelreuteria paniculata* seed oil. *Energies*. 2020;13(4):791.
- [40] Khan IU, Yan Z, Chen J. Optimization, transesterification and analytical study of *Rhus typhina* non-edible seed oil as biodiesel production. *Energies*. 2019;12(22):4290.
- [41] Khan IU, Yan Z, Chen J, Chen H, Shah SAH. *Acacia farnesiana* seed oil: a promising source of biodiesel production. *Int. J. Scient. Eng. Res*. 2021;12(1):277-295.
- [42] Khan IU, Yan Z, Chen J, Chen H, Shah SAH. *Albizia julibrissin*: a non-edible seed oil source for biodiesel production. *Int. J. Scient. Eng. Res*. 2021;12(1):327-341.
- [43] Martins-Noguerol R, DeAndrés-Gil C, Garcés R, Salas JJ, Martínez-Force E, Moreno-Pérez AJ. Characterization of the acyl-ACP thioesterases from *Koelreuteria paniculata* reveals a new type of FatB thioesterase. *Heliyon*. 2020;6(10):e05237.
- [44] Ruan C-J, Xing W-H, da Silva JAT. Potential of five plants growing on unproductive agricultural lands as biodiesel resources. *Renew. Energy*. 2012;41:191-199.
- [45] Zhang F, Ren H, Tong G. Production of biodiesel from the extract of the sumac fruit cluster. *Cellul. Chem. Technol*. 2018;52:1275-1279.
- [46] Clarke HD, Seigler DS, Ebinger JE. *Acacia farnesiana* (Fabaceae: Mimosoideae) and related species from Mexico, the southwestern US, and the Caribbean. *Syst. Bot*. 1989:549-564.
- [47] Weber E, editor. *Invasive plant species of the world: a reference guide to environmental weeds*: Cabi; 2017. DOI 10.1079/9781780643861.0000
- [48] Vines RA. *Trees, shrubs and woody vines of the southwest*. 5th ed. University of Texas at Austin; 1960. 1104 p.
- [49] Williams R, Hanks S. *Hardwood nursery guide*. Slightly rev., July 1994. *Agriculture handbook (USA)*. 1976. p. 473.
- [50] Cheatham S, Johnston M, Marshall L. *useful wild plants of Texas, the southeastern and southwestern United States, the southern plains and northern Mexico*, vol. I. *HerbalGram*. 1998.
- [51] Ozcanli M, Gungor C, Aydin K. Biodiesel fuel specifications: A review. *Energy Sources, Part A: recovery, utilization, and environmental effects*. 2013;35(7):635-647.
- [52] De Castro ML, Priego-Capote F. Soxhlet extraction: Past and present panacea. *J. Chromatogr. A*. 2010;1217(16):2383-2389.
- [53] Gupta J, Agarwal M, Dalai A. An overview on the recent advancements of sustainable heterogeneous catalysts and prominent continuous reactor for biodiesel production. *J. Ind. Eng. Chem*. 2020;88:58-77.
- [54] Pratas MJ, Freitas SV, Oliveira MB, Monteiro SC, Lima ÁS, Coutinho JA. Biodiesel density: experimental measurements and prediction models. *Energy Fuels*. 2011;25(5):2333-2340.
- [55] Knothe G, Kenar JA. Determination of the fatty acid profile by 1H-NMR spectroscopy. *Eur. J. Lipid Sci. Technol*. 2004;106(2):88-96.
- [56] Choe E, Min DB. Mechanisms and factors for edible oil oxidation. *Compr. Rev. Food Sci. F*. 2006;5(4):169-186.
- [57] Wang Y-F, Huang K-L, Li C-T, Mi H-H, Luo J-H, Tsai P-J. Emissions of fuel metals content from a diesel vehicle engine. *Atmos. Environ*. 2003;37(33):4637-4643.

[58] Sorate KA, Bhale PV. Biodiesel properties and automotive system compatibility issues. *Renew. Sustain. Energy Rev.* 2015;41:777-798.

[59] Santos SM, Nascimento DC, Costa MC, Neto AM, Fregolente LV. Flash point prediction: Reviewing empirical models for hydrocarbons, petroleum fraction, biodiesel, and blends. *Fuel.* 2020;263:116375.

[60] Wang W, Li F, Li Y. Effect of biodiesel ester structure optimization on low temperature performance and oxidation stability. *J. Mater. Res. Technol.* 2020;9(3):2727-2736.

[61] American Society for Testing Materials. ASTM 6751-Standard specification for biodiesel fuel (B100). Blend stock for distillate fuels. ASTM, West Conshohocken PA, USA; 2002.