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Extraction and Quality Evaluation of Biodiesel from Six Familiar Non-Edible Plants Seeds †

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† Dedicated to the 100th Anniversary of Chemistry at Nankai University.

Abstract: Biodiesel produced from non-edible plant sources is cost-effective, biodegradable, environment friendly, and compatible with petro-diesel, but new sources and extraction processes still need to be discovered. Here, we explored the fuel properties of seeds from six non-edible plant sources, including *Sapindus mukorossi* (Soapnut, SP), *Vernicia fordii* (Tung, TO), *Ricinus communis* (Castor, CA), *Toona sinensis* (Juss. TS), *Ailanthus altissima* (Heaven tree, AA), and *Linum usitatissimum* L. (Lin seed, LS) from China. The optimum extraction conditions were obtained by optimizing the most important variables (reaction temperature, ratio of alcohol to vegetable oil, catalyst, mixing intensity, and purity of reactants) that influence the transesterification reaction of the biodiesel. All six plants contained high seed oil content (SOC; % *w*/*v*) with the highest in the TO-54.4% followed by SP-51%, CA-48%, LS-45%, AA-38%, and TS-35%, respectively, and all expressed satisfactory physico-chemical properties as per international standards of ASTM D6751 and EN14214. Our data provide a scientific basis for growing these plants in unproductive agricultural lands as an alternative energy sources for biodiesel production either standalone or blended with petro-diesel.

Keywords: *Sapindus mukorossi*; *Verniciafordii*; biodiesel; non-edible plant sources; transesterification reaction

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1. Introduction

The impact of emissions of greenhouse gases on climate change and growing energy needs has compelled the world community to focus on alternate energy sources, such as biodiesel production from plant, algae [1], and other waste materials [1,2]. Biofuel production from biomass is an effective strategy to reduce both crude oil consumption and pollution [3]. The demand and production of biofuels have grown rapidly (approximately 23% per annum) during the last two decades [4,5]. Globally, more than 350 oil-bearing plant sources [6] have been explored for biodiesel production using different extraction and optimization strategies. The vegetable plants used as biodiesel feedstock include soybean, rapeseed, sunflower, palm oils, mustard, peanut, sunflower, and cottonseed [7]. The challenge, however, remains to resolve as far as the high viscosity of vegetable oils is concerned. Generally, the high viscosity at room temperature is considered unsuitable in diesel engines. During the combustion process, oxygen in the air quickly reacts with the outer surface of the oil droplet and releases a huge amount of heat, and further initiates the intricate reactions (charring, choking, and polymerization). The oils with higher viscosity tend to form larger drops and further elevate the polymerization reaction, particularly those with a higher degree of unsaturation [8]. The risk gets even higher as more viscous fuel will weaken the atomization process, and consequently, power loss and smoke production [9], and premature injector fouling [10] will occur. Moreover, thermal decomposition of glycerol produces acrolein (a highly toxic substance) [11], which has been considered as a cancer-causing substance [12]. Previously, many researchers have used edible resources for research purposes, but they faced issues such as the food versus fuel debate and high cost of feedstock; resultantly, the commercialization of biodiesel suffered a major blow [13–16].

The optimization of higher viscosities and other properties of biodiesel is a significant step in order to curtail and manage large drop formation, ignition, and thermal efficiency. Among all the biodiesel production processes, transesterification is the most reliable, cost-effective, and easy method because it produces biodiesel of high yield and possesses compatible properties with petro-diesel [17]. Although vegetable oils are portable, renewable, and contain a high amount of neutral triacylglycerols (TAG), free fatty acids [1] heat, and reduced sulfur constituents, but still they are costly, highly viscous, and possess low volatility and reactions are involved in the unsaturated hydrocarbon chains [18]. Additionally, the conversion of vegetable oils into fatty acid methyl esters (FAME) is an effective way to overcome difficulties for using biofuels [19]. The molar ratio of methanol/oil, reaction temperature, reaction time, amount of catalyst, type of catalyst, and stirring speed have a profound effect on the transesterification process [20,21] and warrant the appropriate optimization. Although FAME is a major constituent of biodiesel, the composition can still be variable when process conditions and feedstock are considered [1]. However, vegetable oil sources pose a significant threat to food scarcity to fulfill human needs. Hence, it was felt to focus on non-edible plant sources as appropriate energy alternatives.

The non-edible plant sources have paramount importance for bio-diesel oil exploitation as far as their cultivation at different lands, availability, cost-effectiveness, lower sulfur and aromatic content, higher biodegradability, and negligible human utilization is concerned [1,22]. However, oil obtained from non-edible plant sources contains free fatty acids (FFAs), which are highly toxic and multiply the cost of biodiesel production [23]. Therefore, the selection of more appropriate non-edible plant sources is a challenging task for boosting biodiesel production at competitive levels.

Keeping the above context in preview, we used seeds from six non-edible plant sources, including *Sapindus mukorossi* (Soapnut, SP), *Vernicia fordii* (Tung, TO), *Ricinus communis* (Castor, CA), *Toona sinensis* (Juss. TS), *Ailanthus altissima* (Heaven tree, AA), and *Linum usitatisimum* L. (Lin seed, LS) for biodiesel oil production from China. These plants generally grow on barren agricultural lands but possess great potential for biodiesel feedstock. In this study, we have measured and evaluated (i) seed oil content (SOC) of seeds from all six plants; (ii) their relative fatty acid content, and (iii) their potential for biodiesel production. The oil extraction from seeds was undertaken using soxhlet and mechanical oil extractor. Both acid- and base-catalyzed transesterification processes were carried out. Various techniques were used for evaluating biodiesel characteristics comprising Fourier transform infrared (FT-IR), nuclear magnetic resonance (NMR), gas chromatography–mass spectrometry (*GC-MS*), inductively coupled plasma-optical emission spectrometry (ICP-OES), and elemental analysis. The study found that all six plants contained high seed oil content (SOC; % w/v), (TO-54.4%, SP-51%, CA-48%, LS-45%, AA-38%, and TS-35%), with satisfactory physico-chemical properties as per international standards of ASTM D6751 and EN14214; therefore, seeds from these plants offer a promising prospect to be used as alternative energy sources for biodiesel production either standalone or blended with petro-diesel.

2. Description of Plant Sources for Biodiesel Feedstock

2.1. *Sapindus mukorossi (Soapnut, SP)*

Sapindus mukorossi tree produces soapnut fruit. The plant inhabits tropical and sub-tropical regions comprising Asia, America, and Europe. *S. mukorossi* and *S. trifoliatus* are commonly found in India, Nepal, Bangladesh, and Pakistan. *S. trifoliatus* contains an average of 51.8% oil content of total seed weight [24]. Soapnut contains potential non-edible oil for biodiesel production [25,26]. Although soapnut fruit shells have been used for medicinal [27], surfactant [28], as well as laundry purposes [29], after using pericarp portion, seeds are wasted, which makes them feasible for use as biodiesel (Figure 1a,b).

Figure 1. (**a**,**b**). *Sapindus mukorossi* (Soapnut) plant and seed photographs.

2.2. *Verniciafordii (Tung, TO)*

Vernicia fordii (Tung nut) belongs to the Euphorbiaceae family and native to China, Burma, and Vietnam (Figure 2a–c). It is a deciduous tree having 20 m height at maximum. Its seeds and whole nuts contain 21 and 41 wt% oil, respectively [30], with the average yield is 450–600 kg per hectare [31]. It has been used for lighting in lamps as well as wood paints and varnishes [32]. Its oil contains the unusual conjugated fatty acid, eleostearic acid (9,11,13-octadecatrienoic acid; 63.8%), with linoleic (11.5%), oleic (8.6%), and behenic (8.4%) acids [33].

Figure 2. (**a**–**c**) *Vernicia fordii* (Tung nut) plant and seed photographs.

2.3. *Ricinus communis (Castor, CA)*

Ricinus communis, a small wooden tree with 6 m maximum height, belongs to the Euphorbiaceae family and generally known as the castor oil plant (Figure 3a,b). Although it has African origin, it can be widely observed in the tropical and subtropical regions of the world. Its seeds contain higher oil content ranging from 48% to 60%, with 500–1000 L of oil/acre production potential. The salient features of *Ricinus communis* oil are 90% ricinoleic acid (18 carbon atoms and hydroxyl group at position 12), more oxygen atoms suitable for transesterification, and challenging high viscosity (240.12 mm^2/s at 313 K) [34].

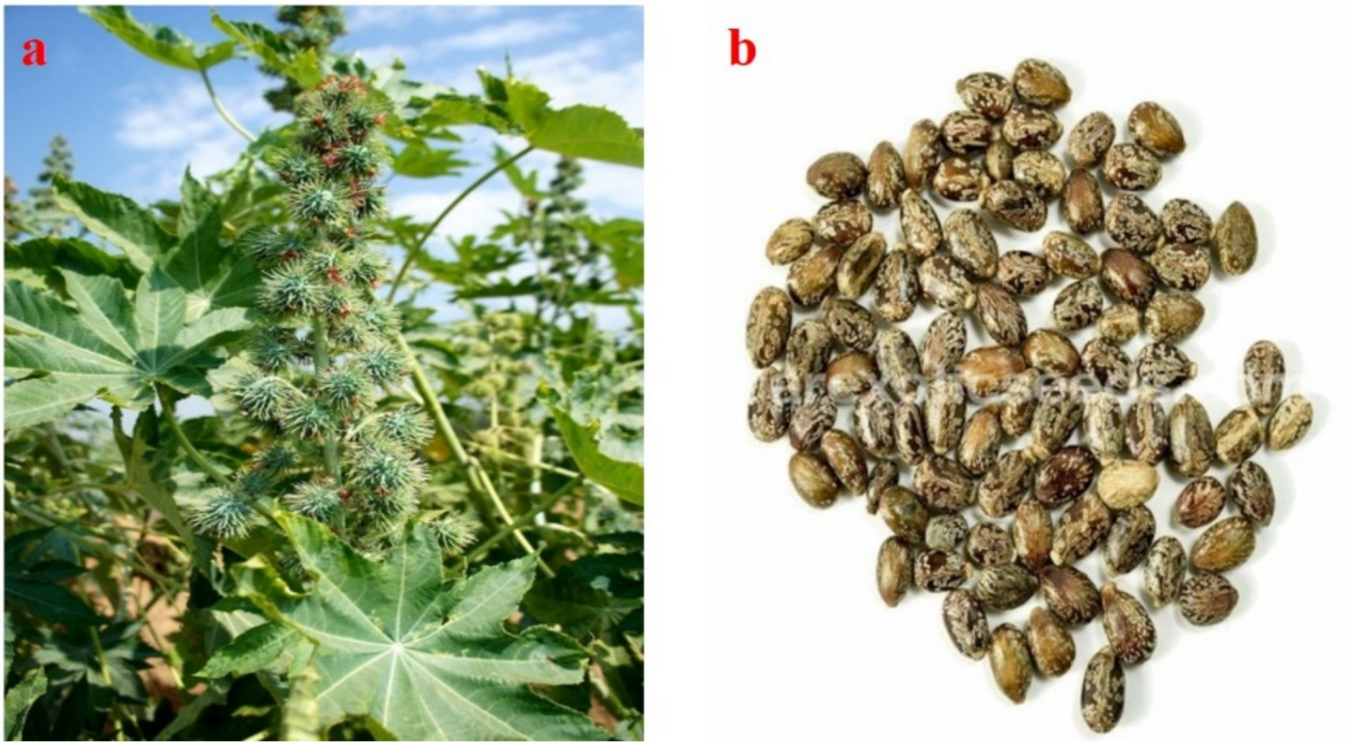


Figure 3. (**a**,**b**) *Ricinus communis*, castor bean plant and seed photographs.

2.4. *Toona sinensis (Juss.,TS)*

Toona sinensis (Juss.), a deciduous woody plant, belongs to the Meliaceae family (Figure 4a–c) and known as Chinese toon or Chinese mahogany [35]. Its stems and leaves are used in the treatment of dysentery, enteritis, carminative, and itchiness in Traditional Chinese Medicine. The terpenoids, phenylpropanoids, flavonoids, and anthraquinones are major phytochemical constituents of *Toonasinensis* [36,37]. It has also been used as anti-tumor, antioxidant, anti-diabetic, anti-inflammatory, antibacterial, and antiviral [36,38–40].

Figure 4. (**a**–**c**). *Toona sinensis* (Juss.) plant and seed photographs.

2.5. *Ailanthus altissima (Heaven Tree, AA)*

Ailanthus altissima, a deciduous tree and belongs to the Simaroubaceae family (Figure 5a,b) and native to China and Taiwan [41]. It has application as biological control of invasive plants [41–43].

Figure 5. (**a**,**b**). *Ailanthus altissima* (Heaven tree, AA) plant and seed photographs.

2.6. *Linum usitatissimum L. (Linseed, LS)*

Linum usitatissimum L., an annual herb, belongs to the Linaceae family and occurs in cool environments (Figure 6a,b). It is native to China and North Vietnam but has spread to Europe and other regions as an invasive species [44]. The oil content obtained from it ranges from 35% to 45% of the overall weight. Its fatty acid profile of oil contains C16:0 (4.4%), C16:1 (0.3%), C18:0 (3.8%), C18: 1 (20.7%), C18: 2 (15.9%), C18: 3 (54.6%), and C20: 0 (0.2). Biodiesel produced has saponification number 109.1 (mg/g), iodine number 48.52 (g I2/100 g), and cetane number 0.916. However, its crude oil contains a higher proportion of gums as well as phospholipids compounds, hence affecting the catalytic activity drastically during biodiesel synthesis [45,46].

Figure 6. (**a**,**b**). *Linum usitatissimum* L. plant and seed photographs.

3. Materials and Methods

3.1. Plant Materials

The clean and infection free seeds from six non-edible plant sources, including *Sapindus mukorossi* (Soapnut, SP), *Vernicia fordii* (Tung, TO), *Ricinus communis* (Castor, CA), *Toona sinensis* (Juss. TS), *Ailanthus altissima* (Heaven tree, AA), and *Linum usitatissimum* L. (Lin seed, LS) were collected from Liaoning province, and Tianjin, China. The general pictorial displays of the procedures used during biodiesel are shown in Figure 7.

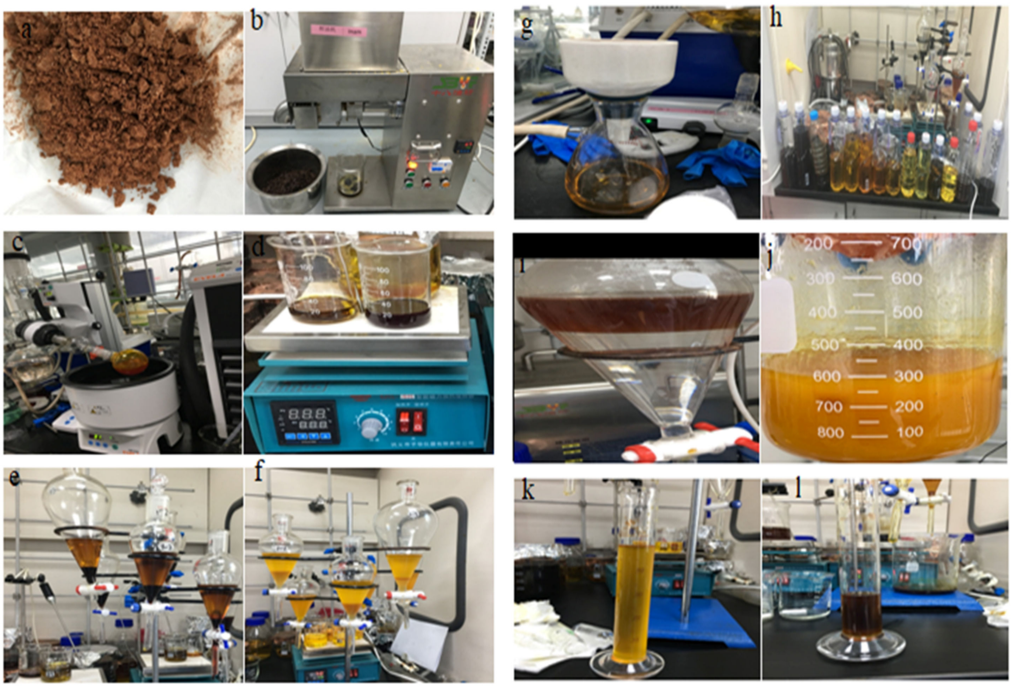


Figure 7. (**a**) Seed powder; (**b**). Mechanical extractor for seed oil content (SOC); (**c**). Rotary evaporator; (**d**). Pre-treatment of crude oil before transesterification (Heat up crude oil to remove moisture content); (**e**). Transesterification (settling and separation of resultant mixture); (**f**). Transesterification; (**g**). Crude oil filtration; (**h**). Different oil and biodiesel samples; (**i**). Washing of biodiesel; (**j**). Glycerine; (**k**). Graduated cylinder for measuring biodiesel; (**l**). After layers making each layer is detached and measure by the graduated cylinder.

3.2. Preparation of Seeds for Feed Stocking

In this study, after washing the seeds with distilled water, these were allowed to dry under sunlight for 48 h and later on were oven-dried at 60 °C for removal of moisture. The seeds were then ground using a grinder (XIANTAOPAI XTP-10000A, Zhejiang, China). The size of seed particles after grinding ranged from 0.21 to 1.0 mm. The seeds were again processed for oven-drying at 60 °C for 90 min to minimize the moisture content, and the seeds were then processed for oil extraction to maximize the purity and minimize any contamination after the grinding and pulverization.

3.3. Oil Extraction

The seed oil content (SOC) from all six seed sources was extracted using soxhlet [47] and mechanical oil extractor (Fangtai Shibayoufang FL-S2017 China and Fangtai Shibayoufang J508, Guangdong, China) (Table S1, Supplementary Materials). The oil extraction occurred at 90 °C for 7 h, and different solvents were also used during this process which comprised petroleum ether, acetone, dichloromethane, and ethyl acetate. Petroleum ether was finally used for all plant sources during Soxhlet extraction. Filter papers (pore size 30–50 µm) were used for removing the impurities, and solvent was removed at 80 °C by employing a rotary evaporator (Tokyo Rikakikai Co. Ltd. N-1210B, Tokyo Japan) under lower pressure (0 to 0.01 MPa). Finally, the oil extracted was stored and allowed to dry over anhydrous sodium sulfate prior to use.

3.4. Evaluation of Potential of the Six Plants as Biodiesel Resources

We assessed the potential of each plant as a biodiesel resource by its seed oil content (SOC). The detailed procedure of Soxhlet and mechanical extraction are given in Supplementary Materials S1 and S2.

3.5. Acid-Catalyzed Esterification Process

The esterification process is most appropriate for the unrefined or waste cooking oils with high free fatty acid (FFAs). Sulfuric acid (H_2SO_4) is commonly used as a catalyst. The procedure is devoid of using pre-treatment of oil with an alkali for lowering its FFA content. The process has few challenges to overcome, including slow rate, need of methanol-to-oil molar ratio; water production causes hindrance in the esterification of triglycerides following reaction of FFA with the alcohol. The overall biodiesel yield is hampered due to the burning of oil followed by treating it with the higher levels of the acid [48].

The methanol (469 g) and H_2SO_4 (11.4 g) were added into soapnut oil, tung oil, linseed oil, Haven tree, Toona sinensis, and castor oil (for all 2751 g) as a prerequisite of esterification pre-treatment and mixed vigorously at 60 °C for 1 h as a reaction time. Then, esterified soapnut oil was processed for methanolysis for 1 h, the molar ratio of methanol to 6, reaction temperature (60 °C), and KOH catalyst quantity on the basis of oil weight of 1% (w/w). The reactor had a Pyrex glass structure having 17.2 cm inside diameter and 5.5 L volume and properly connected with water jacket for marinating the reaction temperature. The reactor's design was founded on the shape factor criteria of a standard six-blade turbine [49]. The solution was allowed to settle down overnight, and thus separation phase was achieved. Later on, an ester phase was carried out, and saturated sodium chloride solution (three times the volume of the ester phase) was used to wash the outcome to remove any residual methanol, KOH, or glycerol. Ultimately, the water traces in the soapnut oil methyl esters (*SPME*) were removed by adding up anhydrous magnesium sulfate, subsequently to filtration [50].

3.6. Base-Catalyzed Transesterification Process

The seed oil biodiesel from all six sources was prepared by using crude oil (50 g), methanol (10 mL) at a molar ratio of 5:1, and KOH catalyst (2.3 w/v%). The reaction was conducted for 1 h at 65 °C under reflux, and the agitation rate was 600 rpm [51]. The procedure was conducted with an excess of methanol (99.99%), having methanol to oil ratio (5:1), and KOH ratio (2.9 w/w%), [52]. The reflux condenser had a reactor to cool down the methanol after coming out of the reaction mixture. The resultant reaction mixture was shifted into the funnel, and it was kept overnight in order to separate the biodiesel, soap, and glycerol constituents with glycerol at the bottom and biodiesel at the uppermost layer. Once the reaction got completed, we separated the crude glycerin using gravity, and KOH was also separated, followed by treating 3–4 times with hot distilled water. The phenolphthalein indicator was used for assessing the complete removal of the catalyst. The vacuum distillation procedure was applied on the leftover un-reacted methanol and moisture until the achievement of the final product and stable FAMEs weight loss. The crude FAMEs underwent further washing for 3–4 times heated de-ionized water, centrifugation, and drying with a vacuum dryer to ascertain its purity. The phase separation was fast and observable within 10 min. The biodiesel phase had a cloudy appearance; it, however, became pretty clean and clear after at least 20 h of setting duration. We used all analytical reagent-grade chemicals.

3.7. Fourier Transform Infrared (FT-IR) Study

The FT-IR bands spectroscopy was evaluated by FT-IR spectrometer (Bruker Vertex 70, Ettlingen, Germany) at a resolution of 1 cm^{-1}, scanning 15 times, and employing Nujol mull as a dispersive medium in the range of 400 to 4000 cm^{-1}, to originate the produced biodiesel which has been described through characterization of different functional groups.

3.8. Nuclear Magnetic Resonance (NMR) Study

The FAMEs NMR spectrum was carried out by NMR Spectrometer (Bruker Avance III 400, Karlsruhe, Germany) at 400 MHz (1H-NMR) or 100 MHz (13C-NMR). Denatured chloroform was used as solvent and tetramethylsilane as the internal standard. The biodiesel ^{1}H NMR (300 MHz) spectrum was documented with a cycle delay of 1.0 s and several scans of 8 times, with pulse duration of 30°. A carbon ^{13}C NMR (75 MHz) spectrum was recorded with a pulse duration of 30° and a cycle delay of 1.89 s, and a scan of 160 times [22,32,33].

3.9. Gas Chromatography–Mass Spectrometry (GC-MS) Study

The FAMEs outcome was evaluated by GC-MS (QP2010SE, Shimadzu, Tokyo, Japan), [22]. GC-MS conditions were followed as per our previous study [22] and are listed in Table 1.

Table 1. Gas chromatograph conditions [22].

Parameter	Descriptions
Column	QP2010SE, Shimadzu PEG-20M Length: 30 m Internal diameter: 0.32 mm Film thickness: 1 µm
Injector temperature	220 °C
Detector temperature (EI 250)	210 °C
Carrier gas	Helium, flow rate = 1.2 mL min^{-1}
Injection	V = 1 µL
Split	Flow rate = 40:1
Temperature program	Initial temperature = 100 °C Rate of progression = 10 °C min^{-1}. Final temperature = 210 °C, 20 min.

3.10. Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) and Elemental Analysis Study of Biodiesel

The presence of metals in the FAMEs was studied using Inductively Coupled Plasma Spectrometer (Spectro-blue, Kleve, Germany) and Elemental Analyser (Vario EL CUBE, Hanau, Germany) for all six plant sources [22].

4. Results and Discussion

The SOC (w/v%) and FFAs (%) of the six plants were 54.4% and 0.12% (*Vernicia fordii*; Tung), followed by 51% and 1.1% (*Sapindus mukorossi*; Soapnut), 48% and 0.8% (*Ricinus communis*; castor), 45% and 2.7% (*Linum usitatissimum* L.; Lin seed), 38% and 1.9% (*Ailanthus altissima*; heaven tree), and 35% and 2.1% (*Toona sinensis*; Juss.), respectively, (Figure 8 and Supplementary Materials Table S1).

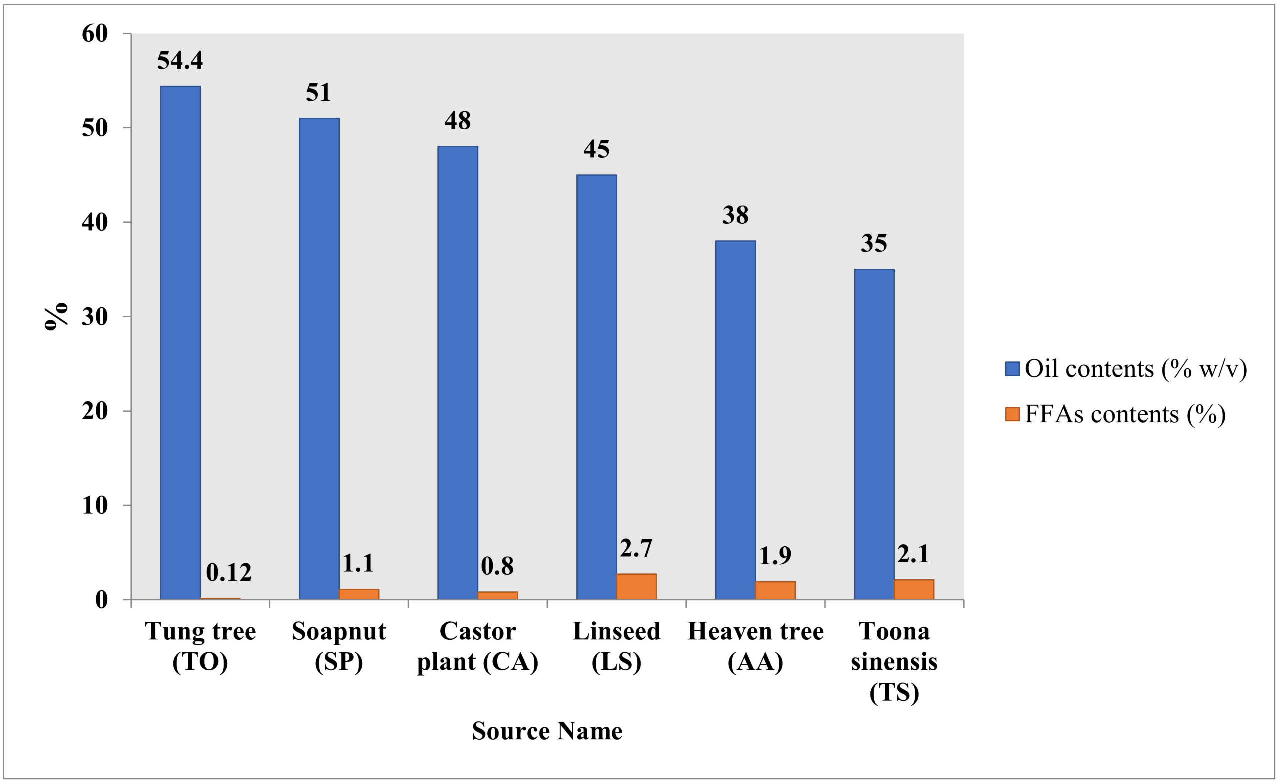


Figure 8. Seed oil content (SOC; *w*/*v*%) and FFAs (%) of the six plants. LSBD =*Linum usitatissimum* L; linseed oil biodiesel; TOBD =*Vernicia fordii*; tung oil biodiesel; AABD = Heaven tree; *Ailanthus altissima* oil biodiesel; TSBD = *Toona sinensis* (Juss.) oil biodiesel; CABD = *Ricinus communis;* castor oil biodiesel; and SPBD = *Sapindus mukorossi;* soapnut oil biodiesel.

4.1. Process of Variables/Optimization

4.1.1. Effect of Methanol to Oil Molar Ratio on FAMEs Yield

The results of the optimization process for all six plant sources are given in Table 2; the details of the process are given in Supplementary Materials (Table S2 AAOB; Table S3 CAOB; Table S4 LSOB; Table S5 SPOB; Table S6 TOOB; Table S7 TSOB). The types of alcohol and methanol to oil molar ratios also have a significant impact on biodiesel production. Short-chain alcohols are preferred for biodiesel production as these are cost-effective and possess greater efficiencies and higher reaction speeds. In the current study, the optimum of methanol to oil molar ratios were 6:1 for TOBD, AABD, LSBD, and CABD; however, these were 5:1 for TSBD and 7:1 for SPBD. Overall, we applied 4:1 to 7:1 methanol to oil molar ratios for all sources in the present study (Details in Supplementary Data). Moreover, methanol to oil molar ratios from 4:1 to 5:1 produced comparatively low FAME yield, and 6:1 was best as it gave an optimum yield, and an equilibrium reaction was established. However, beyond 7:1, the soap content was increased, and FAME content was decreased (Figure 9). The difficulty was further aggravated for separating glycerol due to high methanol solubility rate and reversible equilibrium reaction with the methyl esters to form mono-glycerides [53].

Table 2. Process of optimization of non-edible seed oil for biodiesel oil production.

Source Name/ Optimized Condition	Molar Ratio of Methanol to Oil	Temperature (°C)	Stirring Intensity (rpm)	Reaction Time (min)	Amount of Catalyst Used	Amount of Methanol CH_3OH (mL)	Percentage Yield of Various Products		
					KOH (g)		Biodiesel (% *v*/*v*)	Glycerol (% *v*/*v*)	Soap (% *v*/*v*)
TOBD	6:1	65	700	80	0.32	8.33	97.2	1.8	1
AABD	6:1	65	700	60	0.32	8.33	93.9	6	0.1
LSBD	6:1	65	700	80	0.32	8.33	98	2	0
TSBD	5:1	65	700	80	0.42	10	95.2	3.8	1
CABD	6:1	65	700	60	0.32	8.33	96	4	0
SPBD	7:1	65	700	60	0.32	8.33	96	3	1

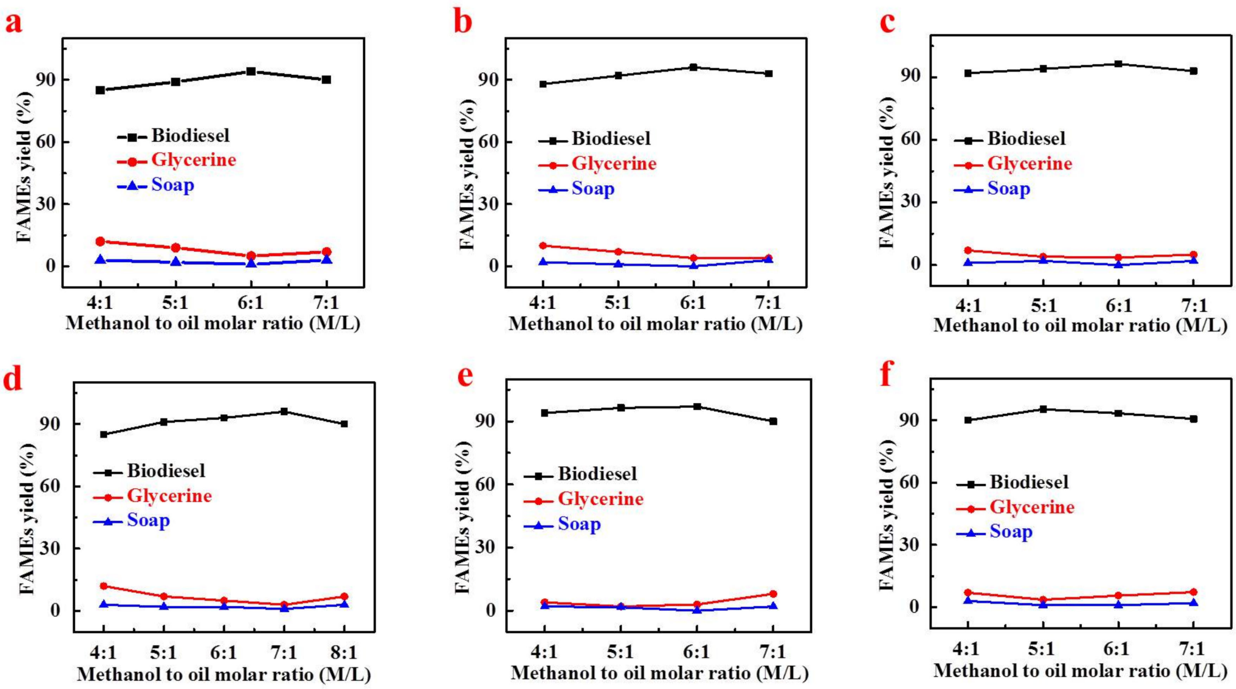


Figure 9. Effect of methanol to oil molar ratio on biodiesel yield (% *v*/*v*). (**a**) TOBD, (**b**) AABD, (**c**) LSBD, (**d**) TSBD, (**e**) CABD, and (**f**) SPBD.

4.1.2. Effect of Catalyst Concentration on FAMEs Yield

Biodiesel production largely depends upon the choice of appropriate, cost-effective, and environment-friendly catalysts based on the nature of oil [54], which greatly help in the transesterification of oil. In our study, among all the tested catalysts (Details are given in Supplementary Materials; (Table S2 AAOB; Table S3 CAOB; Table S4 LSOB; Table S5 SPOB; Table S6 TOOB; Table S7 TSOB), KOH was evaluated as effective in the context of FAME's yield. The optimum concentration of KOH was 0.32 (g) for TOBD, AABD, LSBD, CABD, and SPBD, respectively, whereas it was 0.42 (g) for TSBD. The percentage yield of biodiesel in our study was highest in the LSBD (98%), followed by the TOBD (97.2%), and CABD and SPBD (96% each), TSBD (95.2%), and AABD (93.9%), respectively (Figure 10).

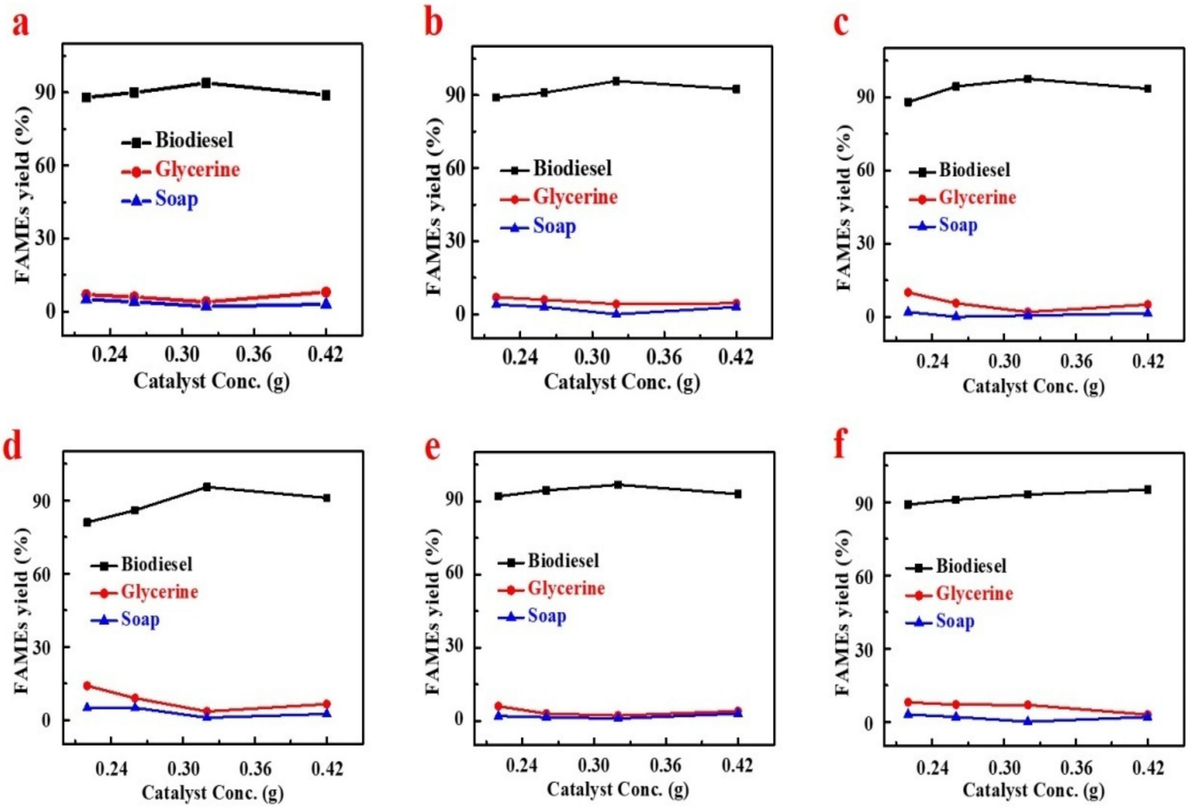


Figure 10. Effect of catalyst concentration on biodiesel yield (% *v*/*v*). (**a**) TOBD, (**b**) AABD, (**c**) LSBD, (**d**) TSBD, (**e**) CABD, and (**f**) SPBD.

4.1.3. Effect of Temperature and Stirring Intensity on Fames Yield

Both temperature and stirring have a remarkable influence on mass transfer during transesterification of biodiesel production [55]. The optimum temperature (65 °C) and stirring intensity (700 rpm) were the same for all the sources in the present study. Details are given in Supplementary Materials (Table S2 AAOB; Table S3 CAOB; Table S4 LSOB; Table S5 SPOB; Table S6 TOOB; Table S7 TSOB). As the boiling point of methanol is 64.7 °C, so 65 °C was normal, as, beyond this temperature, it gets broken and produces a negative impact on biodiesel production. The stirring intensity at 500–600 rpm gave a low FAME yield as it required more shaking. However, at 700 rpm it gave the optimum yield. We found that beyond 700 rpm, it did not produce good results due to the binding of oil and methanol solution and extra shaking, which resulted in a negative impact on FAME yield (Figures 11 and 12).

Generally, biodiesel production generates about 10% (*w*/*w*) glycerol as the major byproduct. The excessive production of glycerol will pose challenges to the refined glycerol market [56]. In our study, the percentage yield of glycerol was highest in the AABD (6%), followed by the lowest in TOBD (1.8%). This highlights that glycerol outcome was at optimized levels in our study, and the plant sources reliably fall in a feasible range of biodiesel production (Figures 11 and 12). However, the glycerol produced as a byproduct can be used as animal feed rations and other value-added chemicals [56].

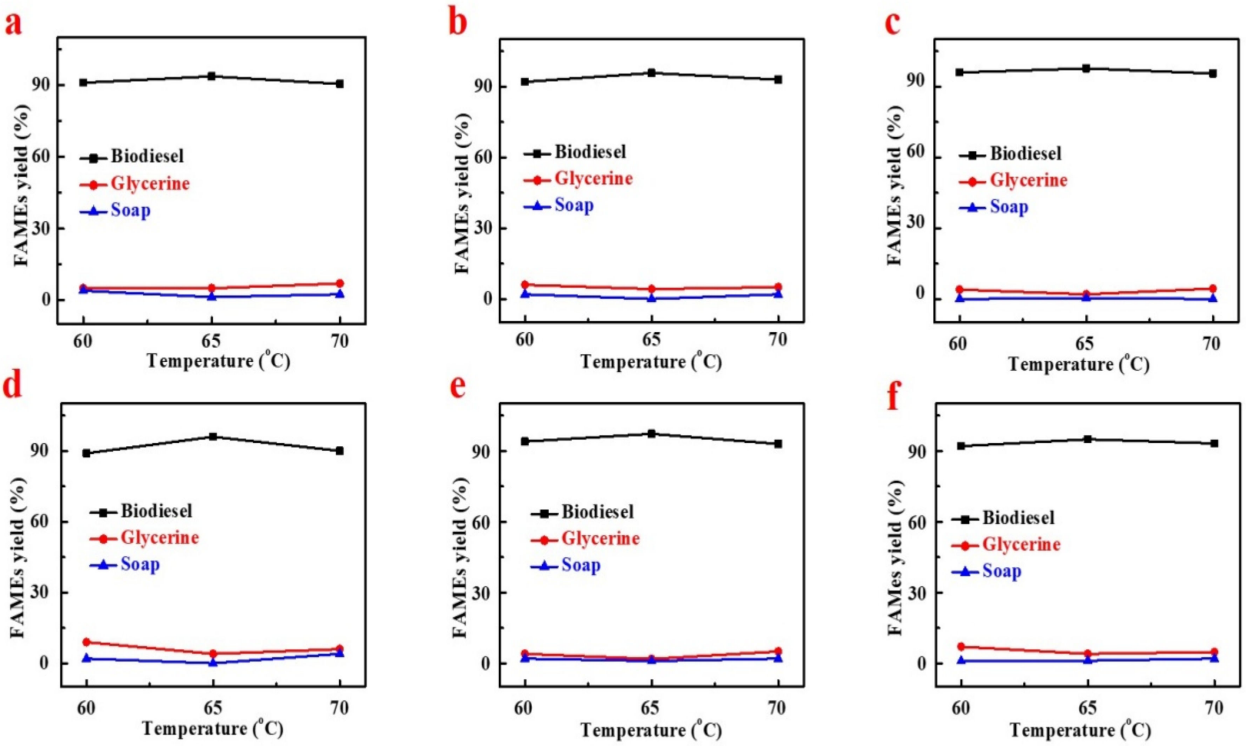


Figure 11. Effect of reaction temperature on biodiesel yield (% v/v). (**a**) TOBD, (**b**) AABD, (**c**) LSBD, (**d**) TSBD, (**e**) CABD, and (**f**) SPBD.

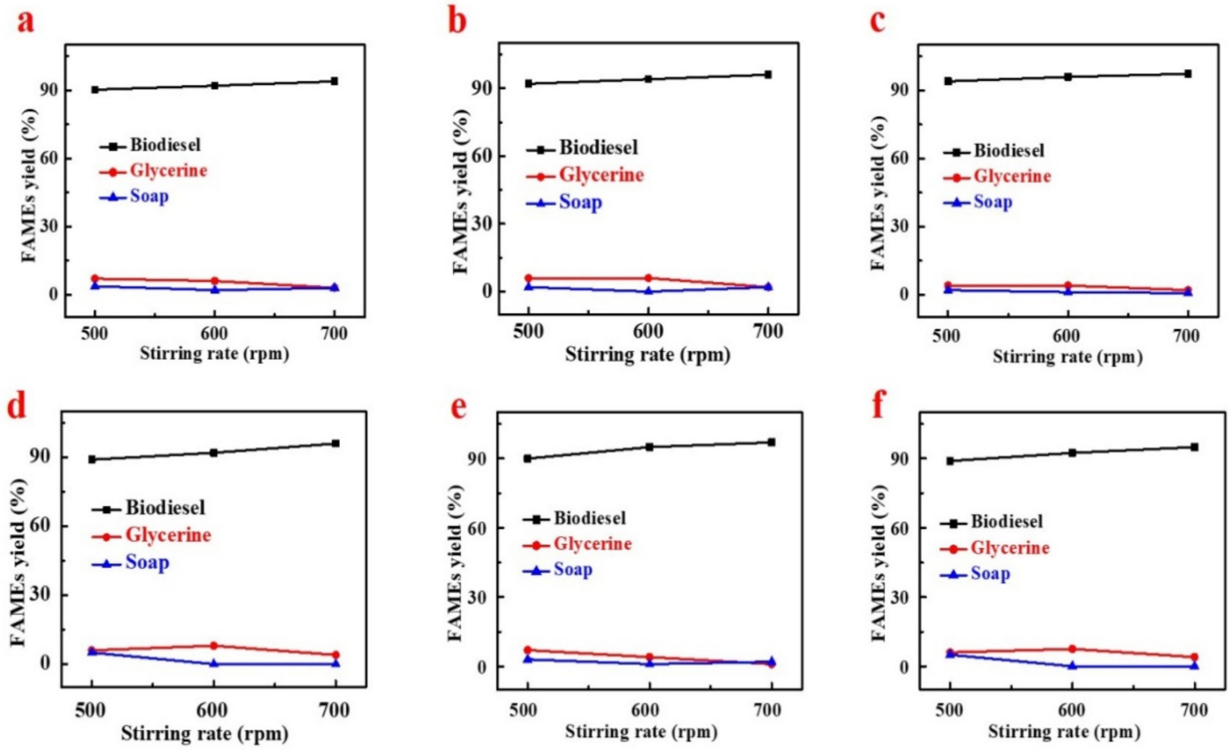


Figure 12. Effect of Stirring intensity on biodiesel yield (% v/v). (**a**) TOBD, (**b**) AABD, (**c**) LSBD, (**d**) TSBD, (**e**) CABD, and (**f**) SPBD.

Biodiesel contains soap as one of the impurities which may create issues in engine operation as well fuel storage [57]. We found that the soap percent content was 0 for

LSBD and CABD, 0.1 for AABD, and 1 each for TOBD, TSBD, and SPBD, respectively. This highlights that the biodiesel we obtained from six plant sources had minimum soap content which supports the notion of their testing and fuel usability at promising levels (Figures 11 and 12).

4.1.4. Effect of Reaction Time on FAMEs Yield

The heterogeneous and homogeneous catalysis need about 4 and 1 h, respectively, as the reaction time to achieve the maximum biodiesel yield [58]. In our study, the optimum reaction time was 80 min for TOBD, LSBD, and TSBD; however, it was 60 min for AABD, CABD, and SPBD, respectively. Details are given in Supplementary Materials (Table S2 AAOB; Table S3 CAOB; Table S4 LSOB; Table S5 SPOB; Table S6 TOOB; Table S7 TSOB). In fact, the reaction time ranges from 60 to 80 min based on the sources for clear separation of biodiesel, glycerol, and soap. In addition, if we allowed more setting time to the final biodiesel product, the possibility of reversible reactions occurred, and the resultant FAME's yield was compromised (Figure 13).

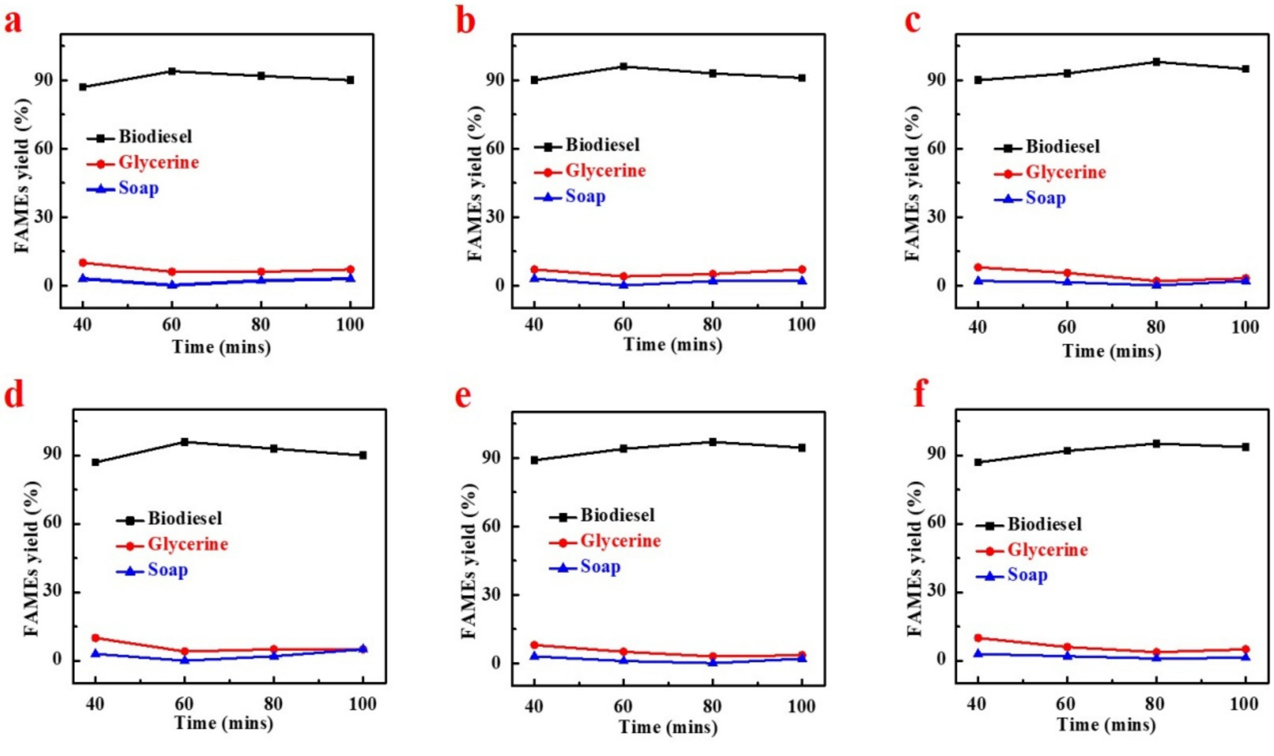


Figure 13. Effect of reaction time on biodiesel yield (% *v*/*v*). (**a**) TOBD, (**b**) AABD, (**c**) LSBD, (**d**) TSBD, (**e**) CABD, and (**f**) SPBD.

4.2. FTIR Analysis of Non-Edible Seed Oil Sources

To identify the functional groups and the bands corresponding to various stretching and bending vibrations in six biodiesel samples, the FT-IR spectroscopy of the mid-infrared region was used, as presented in Figure 14 and the Supplementary Materials Table S8. The two resilient ester representative absorption bands were detected from carbonyl (νC=O) around 1750–1730 cm^{-1} and C-O at 1300–1000 cm cm^{-1} [59]. The stretching vibrations and bending vibrations (ρCH_2) of CH_3, CH_2, and CH appeared at 2980–2950, 2950–2850, 3050–3000 cm^{-1}, and at 1475–1350, 1350–1150, 722 cm^{-1}, correspondingly [60]. The absorption peaks of the sample were detected in all biodiesel samples to be 3464, 3007, 2927, 2854, 1743, 1641, 1435, 1361, 1170, 1016, and 723 cm^{-1}, respectively. The peaks presence in all biodiesel FAMEs at 1430 and 1167 cm^{-1} specifies the conversion of crude oil to biodiesel. The strong absorption peak at 2852–2859 cm^{-1} and 2920–2927 cm^{-1} is just because of the alkane group of C-H stretching vibration. The C-H bending vibration appeared at 1430–1466 cm^{-1} due to strong absorption. All of the single bands symbolize saturated functional groups. The C=O stretching frequency peak at 1741.30 cm^{-1} is due to strong absorption, which is composed of an unsaturated functional group and is called an ester. Further, due to the C-O stretching vibration of the ester, the strong band appeared at 1017–1093 cm^{-1} and 1161–1174 cm^{-1}.

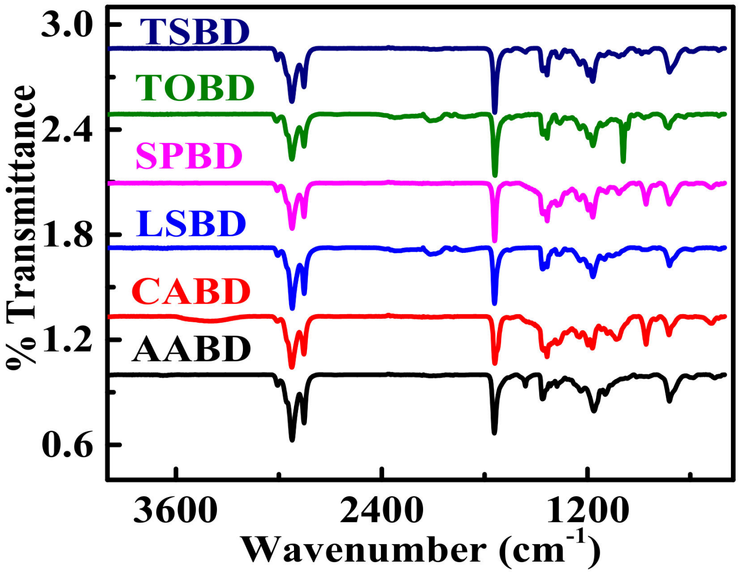


Figure 14. Non edible seed oil FAMEs FTIR analysis.

4.3. NMR Analysis of Non-Edible Seed Oil Sources FAMEs

The nuclear magnetic resonance (NMR) comprising ^{1}HNMR (Figure 15; Supplementary Materials Table S9); ^{13}C NMR spectrum (Figure 16; Supplementary Materials Table S10) were used for the characterization of the FAMEs of all six plant sources.

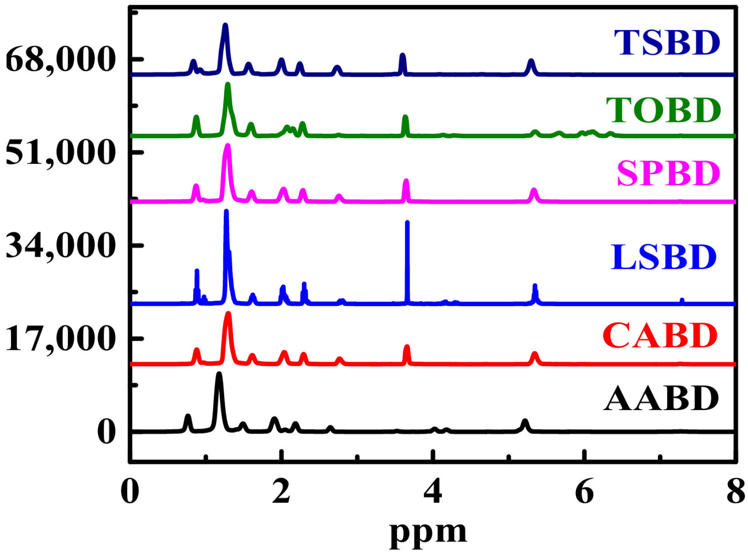


Figure 15. Non edible seed oil FAMEs ^{1}H NMR analysis.

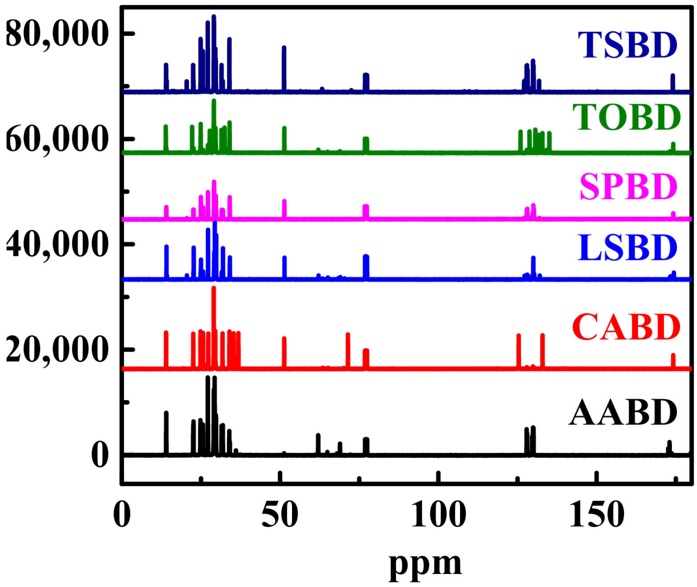


Figure 16. ^{13}CNMR analysis of Non-edible seed oil FAMEs.

4.3.1. ^{1}H NMR Analysis of Non-Edible Seed Oil Sources FAMEs

In the ^{1}H NMR spectra, the terminal methyl proton (C-CH_3) appearance signals were detected between 0.76–0.88 and 0.92–0.98 ppm, and the aliphatic chain (-(CH_2) n-) associated signals were observed between 1.19–1.29 and 1.47 ppm. The β-methylene ester (CH_2-C-CO_2Me) bands' appearance signals were detected around 1.55–1.62 ppm. Correspondingly, the methylene proton peaks near the base (-CH2-C=C-) appeared between 2.00–2.07 ppm attached to the allylic group. The methylene proton peaks signal were attributed between 2.19 and 2.30 ppm existing near the carbonyl group proton (-CH2-COOMe), the existence of a methylene proton (-C=C-CH2-C=C-) between the allylic groups associated between 2.74–2.80 ppm, and the single sharp peak at 3.52–3.66 ppm was the expressed ester bond (CH_3COO-CH) linked CH_3 group. The proton (-CH=CH-) from the glycerol moiety appeared between 5.23–5.34 ppm.

4.3.2. ^{13}C NMR Analysis of Non-Edible Seed Oil Sources Fames

In the ^{13}C NMR spectrums, a signal which showed the occurrence of an ester carbonyl carbon (-COO-) was observed at 174.14–174.46 ppm. The band's signals were observed in the spectrum between 126.68–128.00 and 130.14–130.45 ppm indicating the existence of unsaturation in the methyl ester. The occurrence of ester (C-O) methoxy carbon was observed at 51.35 ppm due to the long carbon chain methylene carbon of the fatty acid methyl ester. The band's occurrence detected between 22.20–34.10 ppm, and the terminal carbon of the methyl group peaks was observed at 13.80–14.12 ppm, respectively (Figure 16).

4.4. GC-MS Analysis of Non-Edible Seed Oil Sources FAMEs

The biodiesel obtained from crude oil of six plant sources and modified by methyl ester was evaluated by the gas chromatography and mass spectrometry (GC-MS) (Figure 17). The peaks were identified by the NIST-14 library matching software. After assessment, every single peak was matched with fatty acid methyl ester [61]. The retention time (min) and position of the determined peaks are presented in Table 3. The palmitic acid in TS, TO, SP, LS, CA, and AA plant sources was detected in retention times (min) of 15.115, 9.537, 15.235, 9.545, 15.213, and 15.115, respectively. The stearic acid in TS, TO, SP, LS, CA, and AA plant sources was detected in retention times (min) of 18.288, 14.255, 19.863, 14.248, 19.840, and 19.637, respectively. The oleic acid in TS, TO, SP, LS, CA, and AA plant sources was detected in retention times (min) of 18.947, 15.050, 20.853, 15.042, 20.583, and 20.387, respectively. The linoleic acid in TS, TO, SP, LS, CA, and AA plant sources was detected in retention times (min) of 22.098, 16.760, 22.397, 16.797, 22.262, and 22.022, respectively. The α-linolenic acid in TS, SP, LS, and CA plant sources was detected in retention times (min) of 24.708, 24.985, 19.565, and 24.940, respectively. The arachidic acid was only detected in SP and LS with retention times of 27.820 and 22.587, respectively. The gondoic acid in TO, SP, LS, and CA plant sources was detected in retention times (min) of 23.938, 29.065, 23.922, 29.058, respectively. The behenic acid was detected in AA with a retention time (min) of 18.647.

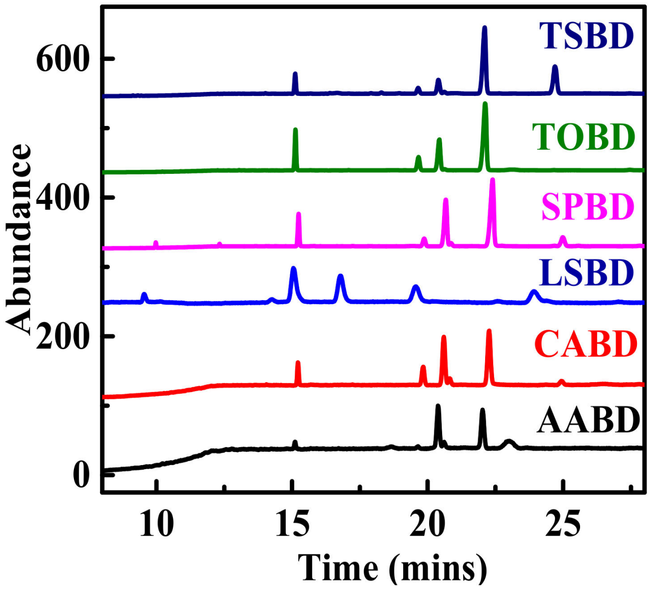
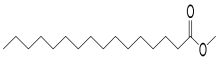
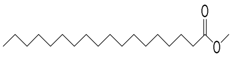
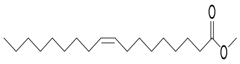
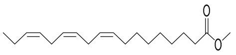
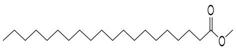
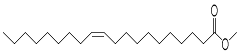


Figure 17. GC-MS study (Analysis) of Non-edible seed oil FAMEs.

Table 3. Gas chromatography and mass spectrometry (GC-MS) of six non-edible plant sources. The retention time in min is shown within brackets ().

Fatty Acids/Exp. Results	TSBD	TOBD	SPBD	LSBD	CABD	AABD	Carbon and Double Bonds	Chemical Name	Chemical Structure	Molecular Weight
Palmitic acid	PA (15.115)	PA (9.537)	PA (15.235)	PA (9.545)	PA (15.213)	PA (15.115)	C16:0	Hexadecanoic acid, methyl ester		270
Stearic acid	SA (18.288)	SA (14.255)	SA (19.863)	SA (14.248)	SA (19.840)	SA (19.637)	C18:0	methyl stearate		298
Oleic acid	OA (18.947)	OA (15.050)	OA (20.853)	OA (15.042)	OA (20.583)	OA (20.387)	C18:1	9-Octadecenoic acid (Z(-, methyl ester		296
Linoleic acid	LA (22.098)	LA (16.760)	LA (22.397)	LA (16.797)	LA (22.262)	LA (22.022)	C18:2	9, 12-Octadecadienoic acid (Z, Z)-, methyl ester		294
α-Linolenic acid	LiA (24.708)	–	LiA (24.985)	LiA (19.565)	LiA (24.940)	–	C18:3	α-Linolenic acid		292
Arachidic acid	–	–	AA (27.820)	AA (22.587)	–	–	C20:0	Eicosanoic acid, methyl ester		326
Gondoic acid	–	GA (23.938)	GA (29.065)	GA (23.922)	GA (29.058)	–	C20:1	CiS- 11-Eicosenoic acid, methyl ester		324
Behenic acid	–	–	–	–	–	BA (18.647)	C22:0	Docosanoic acid/ Methyl behenate		354

4.5. ICP-OES and Elemental Analyzer Study of Non-Edible Seed Oil Sources FAMEs

Metal elements in biodiesel pose problems viz. engine degradation, operational issues, and cause of environmental pollution [62]. The percent element concentrations of C in TOBD, AABD, SPBD, TSBD, CABD, and LSBD were 76.47, 74.88, 76.44, 75.83, 71.71, and 76.25, respectively, which were comparable and less than the petro-diesel concentration (86.5). The percent element concentrations of H in TOBD, AABD, SPBD, TSBD, CABD, and LSBD were 11.65, 13.24, 12.13, 12.42, 11.43, 13.01, and 13.5, respectively, which were comparable petro-diesel (13.5). The percent element concentrations of N in TOBD, AABD, SPBD, TSBD, CABD, and LSBD were 1.96, 1.49, 0.0, 1.72, 0.0, and 1.41, respectively. The percent element concentrations of O in TOBD, AABD, SPBD, TSBD, CABD, and LSBD were 9.92, 10.39, 11.43, 10.03, 11.43, respectively and 0 for petro-diesel, Table 4, and Supplementary Materials Figure S1. Figure 18 displays the ICP-OES result of six non-edible seed oil FAMEs in comparison with petro-diesel. This highlights that element concentrations are within the normal range and are compatible with petro-diesel.

Table 4. Non-edible seed oil FAMEs EA (elemental analysis) study for C, H, N, and O.

Properties	TOBD %	AABD %	SPBD %	TSBD %	CABD %	LSBD %	Petro-Diesel
C	76.47	74.88	76.44	75.83	71.71	76.25	86.5
H	11.65	13.24	12.13	12.42	11.43	13.01	13.5
N	1.96	1.49	ND	1.72	ND	1.41	-
O	9.92	10.39	11.43	10.03	11.43	9.33	0

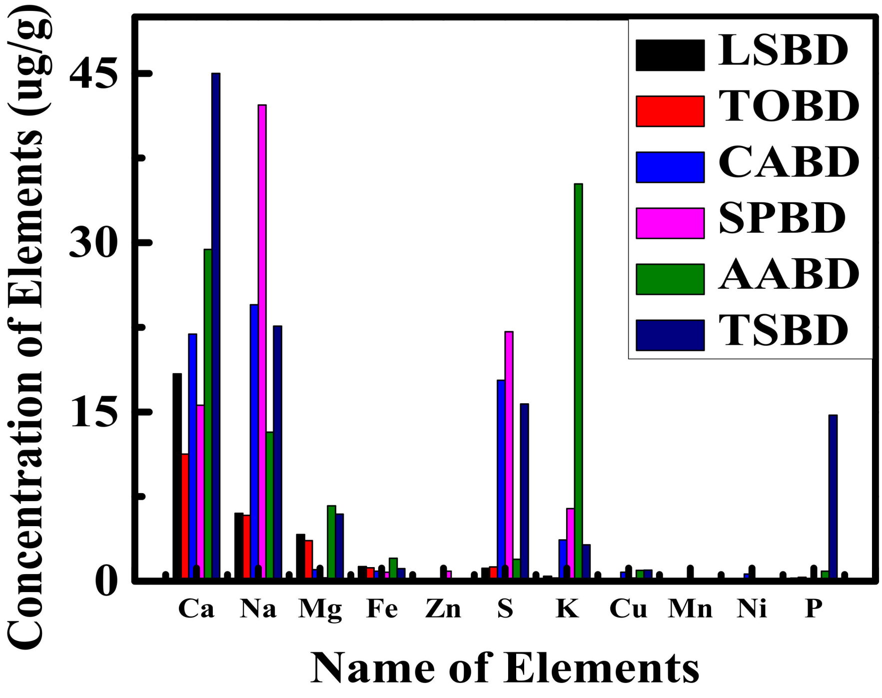


Figure 18. Non edible seed oil FAMEs ICP-OES study.

4.6. Physiochemical Characterization of Non-Edible Seed Oil Sources Fames

The physicochemical characterizations of the FAME obtained from six non-edible plant sources are given in Table 5. The percent oil content was highest in the TOBD (54.4), followed by the SPBD (51), CABD (48), LSBD (45), AABD (38), and TSBD (35). The density was highest in the TSBD (0.995), LSBD (0.932), SPBD (0.929), CABD (0.924), TOBD (0.8858), and AABD (0.873), which was comparable to EN 142214 (0.86–0.90) and ASTM D6751 (0.86–0.90). The kinematic viscosity was highest in CABD (11.13), followed by SPBD (5.0), AABD (4.74), TOBD (4.60), LSBD (3.34), and TSBD (2.32), which was compatible with EN

142214 (3.5–5.0) and ASTM D6751 (1.9–6.0), except CABD. The flashpoint was highest in TOBD (194), followed by the LSBD (172), CABD (170), AABD (169), TSBD (165), and SPBD (160), which were comparable to EN 142214 (minimum 120) and ASTM D6751 (minimum 130). The pour point was highest in the LSBD (−3), followed by AABD (−4), TOBD (−6), SPBD and TSBD (−7 each), and CABD (−10), which was comparable to EN 142214 (−15 to 16) and ASTM D6751 (−5 to 5). The cetane index was highest in CABD (54.53), followed by LSBD (54), TOBD (52), SPBD (51), AABD (49), and TSBD (48), which was comparable to EN 142214 (-51) and ASTM D6751 (−47). The sulfur content was highest in the LSBD (0.0093), followed by CABD (0.0082), TSBD (0.0042), SPBD (0.0032), and AABD (0.0021), but these values were comparable to EN 142214 (<0.01) and ASTM D6751 (<0.01). The free fatty acid contents were highest in the LSBD (2.7), followed by TSBD (2.1), AABD (1.9), SPDB (1.1), CABD (0.8), and TOBD (0.12), which was comparable to EN 142214 (maximum 0.50) and ASTM D6751 (<1).

Table 5. Physico-chemical characterizations of FAME obtained from six non-edible plant sources.

Properties	LSBD	AABD	SPBD	CABD	TOBD	TSBD	EN 14214	ASTM D6751
Oil Content (%)	45	38	51	48	54.4	35	-	-
Density at 15 °C (g/cm^3)	0.9323	0.873	0.929	0.924	0.8858	0.995	0.86–0.90	0.86–0.90
Kinematic Viscosity at 40 °C (cSt)	3.34	4.74	5.0	11.13	4.60	2.32	3.5–5.0	1.9–6.0
Flash Point (°C)	172	169	160	170	194	165	Min. 120	Min. 130
Pour Point (°C)	−3	−4	−7	−10	−6	−7	−15 to 16	−5 to 5
Cetane Index	54	49	51	54.53	52	48	−51	−47
Sulphur Content (%)	0.0093	0.0021	0.0032	0.0082	0.0031	0.0042	<0.01	<0.01
Free Fatty Acid, FFA (%)	2.7	1.9	1.1	0.80	0.12	2.1	Max. 0.50	<1
Calorific Value (MJ/kg)	41.85	37	39	41	39	-	-	-
Water Content (% by vol.)	0.05	0.09	0.03	0.31	0.02	0.06	-	-
Cloud Point (°C)	−6	2	−9	−3	−2	−5	−3 to 12	−3 to 12
Total Acid Number (mg KOH/g)	0.75	0.37	0.59	1.19	0.42	0.83	-	-
Carbon residue (%, *w*/*w*)		0.051	0.031	0.011	0.0150	0.021	-	-
Ash content (%)		0.01	0.002	0.001	0.02	0.01	-	-
Fire point (°C)	182	178	165	177	180	169	-	-

The percent calorific values were highest in TSBD (51), followed by LSBD (41.85), CABD (41), SPBD and TOBD (39 each), and AABD (37).The percent water contents by volume were highest in CABD (0.31), followed by AABD (0.09), TSBD (0.06), LSBD (0.05), SPBD (0.03), and TOBD (0.02). The cloud points were highest in the AABD (2), followed by TOBD (−2), CABD (−3), TSBD (−5), LSBD (−6), and SPBD (−9), which were compatible with EN 142214 (−3 to 12) and ASTM D6751 (−3 to 12). Total acid number was highest in CABD (1.19), followed by TSBD (0.83), LSBD (0.75), SPBD (0.59), TOBD (0.42), and AABD (0.37). The highest carbon residue percent was found in AABD (0.051), followed by SPBD (0.031), TSBD (0.021), TOBD (0.0150), and CABD (0.011). The percent ash content was highest in TOBD (0.02), followed by the AABD and TSBD (0.01 each), SPBD (0.002), and CABD (0.001). The highest fire point was observed in LSBD (182), followed by TOBD

(180), AABD (178), CABD (177), TSBD (169), and SPBD (165). This highlights that the physico-chemical properties of all sources in our study were compatible with the ASTM D6751 and EN 142214.

5. Conclusions

The non-edible plant seed oil, obtained from *Sapindus mukorossi* (Soapnut, SP), *Vernicia fordii* (Tung, TO), *Ricinus communis* (Castor, CA), *Toona sinensis* (Juss. TS), *Ailanthus altissima* (Heaven tree, AA), and Linum usitatissimum L. (Lin seed, LS) from China, provided promising biodiesel yield. The transesterification produced good quality biodiesel production possessing compatible combustion properties with that of diesel. The biodiesel obtained has compatible combustion properties with that of diesel. The seed oil contents obtained were TO-54.4% followed by SP-51%, CA-48%, LS-45%, AA-38%, and TS-35%, respectively. These plants thus can be cultivated on barren lands in order to enhance the feedstock and can be used as an alternate renewable, cost-effective, and environmentally friendly energy source for biodiesel production, either standalone or blended with petro-diesel.

Supplementary Materials: The following are available online at https://www.mdpi.com/article/10.3390/pr9050840/s1, Figure S1: Shows non-edible seed oil BD EA (Elemental analysis) for C, H, N, and O, Figure S2: Transesterification reaction, Table S1: Oil, FFAs contents and Product yields, Table S2: AAOB Process of optimization, Table S3: CAOB process of optimization, Table S4: LSOB process of optimization, Table S5: SPOB process of optimization, Table S6: TOOB process of optimization, Table S7: TSOB process of optimization, Table S8: FTIR data presenting various functional groups in Non-edible seed oil FAMEs, Table S9: 1H NMR spectroscopic data showing the chemical composition of various methyl esters (Methoxy proton) in non-edible seed oil, biodiesel (FAMEs), Table S10: 13C NMR spectroscopic data showing the chemical shift values corresponding to various structural features in non-edible seed oil (Methoxy carbon) FAMEs, Table S11: Shows Non edible plant seed oil BD ICP-OES detail elements concentration (ug/g) in comparison with petro-diesel.

Author Contributions: I.U.K. carried out the experiment and wrote the first draft of the manuscript. J.C. and Z.Y. supervised the research and revised the manuscript. H.C. helped in seed collection and revised the manuscript. All authors have read and agreed to the published version of the manuscript.

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Ethical Statement: This is to certify that study has been undertaken by keeping in preview and adherence to research ethics.

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