

Acacia farnesiana seed oil: a promising source of biodiesel production

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Abstract— The study aimed to appraise oil contents, optimize the transesterification process, characterized by an analytical and physicochemical process, and assess its energy agreeability with fossil fuels. *Acacia farnesiana* (AF) seed containing maximum 23% oil content and 0.4 % low free fatty acid (FFA) content was evaluated by Soxhlet extraction. The petroleum ether yielded highest oil extraction (23%). KOH exhibited the best conversion results of 96% Fatty acid methyl ester (FAMES). FAMES were prepared by transesterification using KOH and methanol. The oil to methanol ratio, reaction temperature/time, stirring strength, and catalyst loading were investigated, and the optimum biodiesel yield was 96%. Pure AF seed oil biodiesel (AFOB) had a kinematic viscosity @ 40 °C (cps); flash, pour and cloud points of 5.3 mm²/s, 158 °C, -28 °C and 7 °C, respectively. The S and P contents were 1.22 and 0.79 µg/g. The viscosity and density of AFOB were comparable to ASTM biodiesel standards. By using GC-MS spectrometry five fatty acid were detected comprising palmitic acid (6.85%), stearic acid (2.36%), oleic acid (12.13%), linoleic acid (46.85%), and α-linolenic acid (1.23%). The study concludes that AFOB can be an intriguing raw material for FAMES yield as an alternative fuel source.

Index Terms— Biodiesel, *Acacia farnesiana* seed, Non-edible oil, Alkaline-catalyzed hydrolysis, Physicochemical properties.

1 INTRODUCTION

With the running out of fossil fuels, the increasing demand for clean energy, it is essential to develop substitute hygienic and renewable energy sources (1). Energy plays a crucial role in the entire features of our community and economic life due to being a vibrant part in the existence and safety of any nation. Facing with speedy intake, increasing expenses, conservational difficulties, and lack of fossil fuels, the world is looking for alternative energy sources, especially biodiesel (BD) (2-5). Feedstock is the most vitally and fundamentally important for biodiesel enhancement. Biomass is the plant material derived from the reaction between CO₂ in the air, water and sunlight, via photosynthesis, to produce carbohydrates that convert solar energy to chemical energy (6, 7). About 170 billion tons of biomass is produced every year on earth. Biomass has always been a major source of energy for mankind and is presently estimated to contribute of the order 10–14% of the world's energy supply, and more than 60% in underdeveloped regions, more than 90% of the living energy of about 2.5 billion people in the world is biomass energy (6). Biomass energy has the advantages of easy combustion, less pollution and lower ash content. The disadvantages

are low calorific value and thermal efficiency, large volume and hard to transport. The thermal efficiency of direct combustion biomass is only 10 to 15%. Biomass energy can be efficiently utilized through energy conversion technology [6], such as converting biomass into liquid and gaseous fuels as biodiesel through chemical routes (7). The biodiesel fuels are oil esters (8) which have been an auspicious substitute source to fossil fuels because of its biodegradability, renewable nature, low emission lethal and good storing and transportation properties (9). In recent years, exploration and development of valuable energy plant seeds as biodiesel source has become a research hotspot. Amongst these substitute foundations, vegetable oils have gained substantial consideration because they can be obtained from renewable sources and produced internally, and are not as detrimental to the environment like petrol (10). There is a severe shortage of fuel in several emerging countries, so the energy disaster has become the day-to-day authenticity of most households (11). Raw materials from commercial sources come from palm, sunflower, coconut rapeseed, soybean, and flaxseed, etc. (12).

BD contains alkyl esters of long-chain fatty acids (13).

The oxygen content amount of BD is higher than that of petroleum diesel, which indicates that carbon emissions, particulate emissions, CO, aromatic hydrocarbons, sulfur, smoke, and noise are significantly reduced (14). BD is a recyclable, non-poisonous, decomposable, and environmentally friendly fuel (15, 16). Erucic acid is the main component of fatty acid composition (56-66%), which makes the oil unsuitable for human consumption (17, 18), but it leads to high stability and low melting point (19). Current research on renewable fuels focuses on the manufacture of biofuel from plant oils (20). By using cheap raw materials, the production charges of biodiesel can be concentrated. Biodiesel is synthesized via transesterification utilizing oil and alcohol (usual methanol) (21). Plant-based usually can reduce carbon monoxide emissions, aromatic hydrocarbons, ash, sediment, water, glycerides, and methanol (2). There are various methods for producing biodiesel, among which transesterification appears to be a modest and operative fuel production method (22). The transesterification reaction is a chemical reaction in the presence of a catalyst between triglycerides, and short-chain alcohols, which can generate monoesters, branched-chain, and long-chain triglyceride molecules that are converted into glycerol and monoesters (22). The three-step reaction forms monoglycerides and diglycerides as intermediates (23). Methanol is a frequently used short-chain alcohol besides ethanol, propanol and butanol. Methanol is used commercially to produce biodiesel due to its low-charge (24). First-class essential catalysts such as (KOH) are supplementary operative in the transesterification process (22). The reaction equations can represent the entire transesterification reaction in Fig.1.

Biodiesel mainly comes from edible oil, and there is little research on the yield of BD from inedible and low-cost oil. The cost of using appetizing oil as a biodiesel reserve accounts for approximately 60-70% of substantial raw costs (25). The high cost of biodiesel is the major barrier for its commercialization, and the main part (85-95%) of the total cost of biodiesel production is the cost of the raw material. To control this situation, it is important to explore non-edible oil resources which can be proved as cost effective as well as resolving the food concerns (25, 26). To use cheap raw materials is the only way to reduce the production cost of BD, such as mixed compounds comprising fatty acids, inedible oils, animal fats, recycled oils, refined plant waste oils and by-product oils (26). It makes sense to develop substitute bases of renewable oil, which not only added advance the commercial feasibility of biodiesel but also upsurge the probable amount of such fuels. The main obstacle in the commercialization is the high cost of biodiesel (27, 28). According to the researchers, (85-95%) is the cost of the raw material, which plays an essential role in the cost of the biodiesel (29). Consequent-

ly, there is a need to obtain biodiesel from lower charge fossil resources such as inedible oils. Using non-edible raw materials can easily produce biodiesel (22, 30, 31) while gaining multiple benefits, such as higher energy security, diversification of energy and agriculture, and accelerating development of rural areas, thereby increasing employment opportunities. Therefore, the growth of mainly inedible oil-bearing plants and the production of biofuels can promote the use of cultivated land by increasing the value of marginal land and limiting competition with food crops (32, 33). Besides, most inedible oil harvests cultivate glowing on the desert and can bear deficiency and scarcity circumstances without concentrated care (34). In this case, inedible oil crops can be created, such as *Jatropha curcas*, *Pongamia pinnata*, *Madhuca indica*, Soapnut seeds (*Sapindus mukorossi*), cotton seeds (*Gossypium*), *Terebinth* (*Pistacia terebinthus*), rubber seeds oil, Honne oil, rice bran oil and palm oil, etc. can be planted on a large area in a non-planted wasteland. In general, the effects of several manipulated variables on transesterification reactions have been studied, and the fuel characteristics of biodiesel formed from inedible oils have been resolute (22, 34). They are suitable sources for the production of biodiesel.

However, there are additional inedible oils that have not been deliberate as raw resources for biodiesel manufacturing. So far, *Acacia Farnesiana* oil (AFO) is one of them.

In this study, *Acacia farnesiana* (AF) is chosen as the inedible raw material to produce biodiesel. Its scientific name is *Acacia farnesiana*, Family Leguminosae (Mimosoideae), native to North America. The fruit (seed) is an elongated pod, 3 to 6 inches long, dry, and covered with hard skin, brown in color. The long-lasting fruit has a smooth appearance and contains seeds cherished by birds and other wildlife. An area of about one hectare area will produce 91,500 kg of seeds yield, and the efficiency of oil per hectare area is approximately 21,250 kg. Plant description is given in S1 (Supporting Material).

To study the optimal reaction conditions for the manufacture of FAMES from raw AF seed oil, we obtained the optimal transesterification conditions, i.e., oil to methanol molar ratio of 6:1, KOH (catalyst) concentration of 3.0 wt. %, reaction temperature of 65 °C, stirring rate of 700 rpm and 60 min of reaction time. This work explored the optimal reaction conditions for the production of biodiesel by AFO transesterification. The physico-chemical properties and composition of the FAME were investigated. It was acceptable for the standard of biodiesel qualities for BD100. Density, kinematic viscosity, cloud point, pour point, flash point, fire point, refractive index, cetane number, oxidation stability, saponification value, iodine value, acid value, specific gravity, ash content, and cold filter plugging point of the biodiesel products were came up to the biodiesel qualities. The values of fuel properties were

found to be comparable with mineral diesel. The synthesized esters were confirmed and characterized by the application of NMR (^1H & ^{13}C), Fourier Transform-Infra red spectroscopy, and Gas Chromatography-Massspectroscopy. For elemental concentrations, ICP (Inductively Coupled Plasma Spectroscopy) and EA (Elemental Analyzer) have been studied. It proved the feasibility of using AFO diesel in current diesel engines through system experiments, which does help to develop this renewable energy. The primarydetermination of this study is to evaluate whether AF seed oil can be used as a potential unconventional raw material for biodiesel pro-

duction. Due to the increasing demand for oils from the manufacture of biodiesel, the proposed research aim is to find oil from unconventional sources. This article describes the optimization of AF seed oil methyl ester (AF-MEs). The resulting AFMEs was characterized, the number of basic fuel properties was determined, and matched with ASTM D-6751 and EN 14214 principles. The biofuel produced from AF seed oil fulfills with the conditions verbalized by ASTM D6751 and EN14214 standards. Overall, a prospective inedible raw material for biodiesel making was discovered and scrutinized.

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2 MATERIALS AND METHODS

2.1 Materials

AF seeds were collected from three different places: Pakistan, Lakki Marwat (19.2%), Urumqi, China (16.7%), and Tianjin, China (23%). To remove the dust and dirt, the seeds were washed with purified water and dry to constant weight in an oven at 60 °C. Then dried the seed powder in a furnace for 24 hours at 60°C, and packed in a plastic tube, freeze and kept at -20°C until analysis. By using Soxhlet extractor, the oil was extracted employing solvent and secondly by electric oil press (FANGTAI SHIBAYOUFANG FL-S2017 China) and (FANGTAI SHIBAYOUFANG J508, China). Methanol (purity 99.9%), KOH, and Na₂SO₄ were analyzed. It was acquired from Merck (Germany) and can be used deprived of any cleansing.

2.2 Preparation of Feedstock

The dried AF seeds were ground in a heavy mill (XIANTAOPAI XTP-10000A, China) to pass through a 40 mesh screen. The seed oil extraction process was in a Soxhlet extractor using five different solvents petroleum ether, n-hexane, acetone, dichloromethane and ethyl acetate (60-110°C) for 8 hours. After removing impurities and solid particles by filtration, the oil and solvent were separated by a rotary evaporator.

2.3 Reagents and Chemicals

Methanol (reagent grade), phosphoric acid, sulphuric acid, KOH, NaOH, potassium methoxide, sodium methoxide, sodium sulphate, calcium oxide, calcium chloride, zirconium dioxide were obtained from Aladdin, Sigma and Sinopharm. Ltd. China. The chemicals are analytically pure (99.98%) and can be used without additional cleansing.

2.4 Oil Extraction

A sample of 20 g of seed powder was extracted using Soxhlet, and a large amount of seed oil was extracted by using a mechanical extractor (Table S1, Supporting Materials). The dried AF seed powder was placed in a Soxhlet extractor, and various solvents were used for oil extraction. After refluxing for 8 hours, the solvent was distilled off at 70°C. The solvent was removed by evaporator via a rotational evaporator (TOKYO RIKAKIKAI Co. Ltd N-1210B). Placed the residue in a desiccator and weighed to a constant value. Based on the ratio of the mass of oil obtained to

the mass of crushed seeds, the oil content in *A. farnesiana* seeds can be calculated (35). A large amount of oil was extracted from the seeds of AF by using mechanical pressing. This process is given in S2 (Supporting Material).

2.5 Transesterification of Oil

The transesterification reaction was conducted out in a 250 mL of flask prepared with a reflux condenser and a magnetic stirrer connected to a thermometer. Before producing biodiesel, the oil (15-70 g) was heated to 50°C, and then methanol (methanol/oil molar ratio of 6:1) and KOH (3.0wt. %) were added to obtain a solution. The mixture was added, and the combination was permissible to react for 1 hour (basic transesterification reaction). Fig. 1 shows the transesterification reaction. After standing for 24 hours, the separation occurred in the funnel, the upper layer (biodiesel) was composed for analysis, and the second lower layer (glycerine) was discarded. The residual catalyst containing methanol and soap is removed by continuous rinsing until the wash water is neutral. Anhydrous sodium sulphate was used to dry the washed over biodiesel, and then under concentrated compression can be filtered.

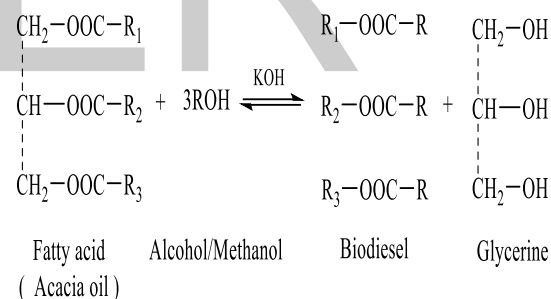


Fig. 1. Transesterification reaction of AF biodiesel.

2.6 Biodiesel Production

Biodiesel production catalyzed by KOH is also carried out in microwave reactors. After optimization, the reaction conditions of biodiesel were as follows: the reaction temperature was 65°C for 60 mins, the methanol to oil molar ratio was 6:1, and the KOH (catalyst) amount was 3.0 wt. % by weight. Also, through GC-MS, the chemical composition of biodiesel was analyzed.

2.7 FAMES Cleansing

After the separation of two layers, the biodiesel (upper layer) was cleaned by fractionation of the remaining methanol at 60°C. By accumulation 1-

2 drops of acetic acid, the catalyst was neutralized, and by continuous washing, through purified water, the remaining catalyst was detached. Further, by handling with anhydrous sodium sulfate (Na_2SO_4), the remaining water was removed and then filtered.

2.8 Properties Evaluation of Biodiesel Product

The key physiochemical characteristics of AF FAMES were evaluated by using the Standard technique under ASTM D-6751 (American Standard for Testing and Materials) and EN (European Norm).

2.9 Analytical Methods

Through the ASTM method, the synthesized AF FAMES properties were determined. Various analytical techniques have been applied to the process of identifying and monitoring FAMES using FTIR. Bruker Vertex 70 FT-IR spectrometer (Germany) was used to characterize *A. farnesiana* oil biodiesel in the range of 4000-400 cm^{-1} by FT-IR. The perseverance was 1 cm^{-1} and 15 scans.

NMR analysis was performed using a Bruker Avance III 400 NMR spectrometer (Germany) equipped with a 5 mm BBO probe of 7.05T. Deuterated chloroform (CDCl_3) was used as solvents, and tetramethylsilane (TMS) was used as internal standards (36). The recorded ^{13}C (75MHz) spectrum has a pulse duration of 30°, a cyclic delay of 1.89s, and a scans count of 160 times. The ^1H NMR (300 MHz) spectrums were recorded with a cyclic delay of 1.0 s, multiple scans of 8 times, and pulse duration of 30°.

The FAMES composition of *A. farnesiana* oil biodiesel was evaluated by gas chromatography model (QP2010SE, SHIMADZU, JAPAN). The detailed procedure is given in Table1 and S3 (Supporting Material).

Table 1 Gas chromatograph conditions.

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 ratio	Flow rate = 40:1
Temperature program	Initial temperature = 100 °C Rate of progression = 10 °C min^{-1} Final temperature = 210 °C, 20 min

2.10 Elemental analysis

The elements concentrations (Na, K, Ca, Mg) in biodiesel were determined by inductively coupled plasma optical emission spectrometry (Spectro-blue, Germany). The detailed procedure is given in S4 (Supporting Material).

The CHN and oxygen content of biodiesel is determined by the Vario EL CUBE (Germany) Elemental Analyzer (EA). This procedure is given in S5 (Supporting Material).

3. RESULTS AND DISCUSSION

Fig. 2 shows a photograph of an AF plant with flowers, seeds, and seed pods. To extract oil from AF seed powder, different types of solvents were used (Table 2). The particle size of the seeds, extraction temperature, an assemblage of seeds at the appropriate time, suitable seed pretreatment, the use of proper solutions, and environmental conditions are essential to obtain high oil content.

For extracting oil from AF seeds, the best solvent was Petroleum ether because it can reduce free fatty acid (FFA) content (0.4%) and provide a high yield of oil (23%). Similarly, when compared with other solvents, petroleum ether can be recovered and reused from the process and cheaper. The oil and FFA content obtained will vary and appear to be associated to the polarization of the extraction solvent, in the directive of petroleum ether, n-hexane, ethyl acetate, acetone, and dichloromethane. Large amounts of non-polar oils (triglycerides) can extract through less polar solvents and small

amounts of highly polar FFA, and vice versa. The oil content of the solvent n-hexane is 19.7% by weight (FFA = 1%). The oil content of ethyl acetate is as low as 18.8% by weight (FFA = 1.5%), while the oil content of more polar solvents (such as acetone and dichloromethane) are slightly lower (17.4-15.2%) (FFA = 1.3-1.7%). According to the extraction results, the use of petroleum ether and the capability to yield AF seed oil, AF seeds can be used as a useful source of alternative energy and biodiesel production.

In this work of AF crude oil, different catalysts were used to check which catalyst was more productive, and the highest conversion rates were obtained for FAMES (Fig. 3) and Table S2 (Supporting Material). The optimized KOH conversion rate can reach 96%, the NaOH conversion rate was 93.5%, CaO conversion rate was 92%, CH₃ONa conversion rate is 91.5%, the CH₃OK conversion rate was 91%, and ZrO₂ conversion rate is 90%. KOH gave the best FAMES conversion results, so to carry out further parameter studies; KOH was used throughout the experiment.

To determine the effect of catalyst types on the production yield of *Acacia farnesiana* oil methyl esters (AFOMEs). Six different experiments using KOH, NaOH, CH₃ONa, CH₃OK, CaO, and ZrO₂ at concentration of 2.0-4.0% (w/w) were tried. In all experiments, variables such as methanol/oil molar ratio (7:1), agitation intensity (700 rpm), and reaction temperature (65°C) were kept constant. The optimum yield of AFOMEs was obtained with KOH catalyst (Fig. 3). It was found that hydroxide-based catalysts (NaOH, KOH) produced greater esters yields than the corresponding methoxide catalyst (CH₃ONa, CH₃OK), indicating their better catalytic efficacy toward transesterification. These catalytic trends agree with some previous studies on methanolysis of *Pongamia pinnata* (37), *Brassica carinata* (38), and rapeseed oils (39), which revealed that KOH was the most efficient catalyst. However, a similar finding as in the present study was observed in on okra oil methyl esters (40). Some researcher such as, Vicente, Martinez, and Aracil (41) reported that the use of sodium methoxide is more appropriate for transesterification of oils. Such variations of catalysts activity might be ascribed to the varied nature of the feedstock vegetable oils as well as to the varying concentration of the catalysts used as different researchers used different oils and catalyst concentration for the experiments.

The biodiesel superiority can be appraised by physical and chemical methods, such as viscosity, density, flash point, cloud point, and pour point. Progressive analytical techniques, comprising GCMS, NMR, and FTIR, are widely used to assess the quality of biodiesel. The experimental results obtained in this work include the oil obtained from the extracted AF seed oil and the methyl ester yield of AF produced by the transesterification reaction with KOH catalyst; under the same reaction conditions, using KOH as the catalyst, the yield of methyl ester obtained by the transesterification reaction of AFoil and the fuel properties of AFoil were compared.



Fig. 2. (a-g) Optical plant photographs (flowers, seedpods, and seed).

Table 2 Solvent effect on FFAs and Oil contents

Solvent	Oil content (wt. %)	FFAs content (wt. %)
Petroleum ether	23	0.4
n-hexane	19.7	1
Acetone	17.4	1.3
Dichloromethane	15.2	1.7
Ethyl acetate	18.8	1.5

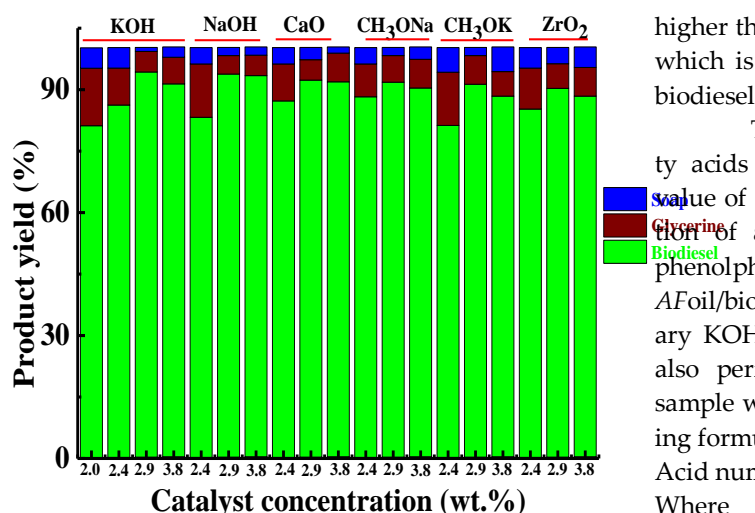


Fig. 3. Catalysts impact on conversion yield.

3.1. Properties of AF Oil Biodiesel

The ASTM technique is used to characterize the oil characterizations of AF oil biodiesel, namely density, kinematic viscosity, cloud point, pour point, flash point, acid value, and sulfur content. Table 3 lists several important physical and chemical properties and fuel characteristics of optimized AFOMEs/biodiesel produced approving to recognized biodiesel standards of ASTM D-6751 and EN 14214. The Current results are listed in Table 3. The density of AF oil biodiesel was measured at 15 °C was 0.83134 g cm⁻³, which follows well the required parameters for biodiesel.

The most critical characteristic of biodiesel is the viscosity because it affects the process of fuel immunization devices, especially at low heats, which distresses the flow of fuel. The poor atomization of the fuel spray occurs due to high viscosity. The kinematic viscosity of A. farnesiana oil biodiesel measured at 40°C was 5.32 cps, which is in the range of ASTM D6751 for biodiesel and ASTM D975 for petroleum diesel (Table 3).

Pour point (pp) is the heat rate at which the wax quantity extracted from the solution is appropriate to achieve fuel. Therefore, at such low limits of temperature, the fuel will flow, and the cloud point (cp) is the heat rate; when the fuels cold, the wax becomes visible on it. AF oil biodiesel has pp and cp of -28 and 7°C, respectively, which follow the diesel restrictions quantified in ASTM (Table 3). The handling, storage, and safety of fuels and flammable materials are the flashpoint considered parameters. The measured flashpoint (158°C) of AF oil biodiesel is

higher than that of petroleum diesel ASTM D975, which is equal to the value of ASTM D6751 of biodiesel (Table 3).

The acid value is an indicator of free fatty acids in petroleum and biodiesel. The acid value of oil and biodiesel is resolute by the titration of an acid-base aqueous solution. Using phenolphthalein as an indicator, the AFoil/biodiesel was titrated alongside a customary KOH aqueous solution. Blank titration was also performed with isopropanol. Titrate the sample with 1 mL of the sample. Use the following formula to calculate the acid value.

$$\text{Acid number} = (A-B) \times C / V$$

Where

A = Volume of KOH used for sample (mL)

B = Volume of KOH used for blank

C = Concentration of KOH

V = Volume of sample (mL)

The determined values of the acid number of AF oil biodiesel were found to be 0.32 mg KOH/g of sample, respectively (Table 3).

Table 3 Physio-chemical characterizations of AFOMEs and their comparison with standards and petrodiesel.

Studied parameters	EN 14214	ASTM D-6751	Petro-diesel	Experimental result	RSOB ⁽⁴²⁾	AAOB ⁽⁴³⁾	HMOB ⁽⁴⁴⁾
Density @ 15 °C (g/cm ³)	0.86–0.90	0.86–0.90	0.809	0.831	-	0.91	0.88
Kinematic viscosity@ 40 °C (cps)	3.5–5.0	1.9–6.0	1.3–4.1	5.32	4.5	36	4.1
Flashpoint, (°C)	Min. 120.0	Min. 130	60–80	158	120	-	153
Acid value, (mg KOH/g)	Max. 0.50	Max. 0.5	-	0.40	0.12	0.64	0.35
Saponification value (mg KOH/g)	-	-	-	174.8	-	-	-
Iodine value, (g I ₂ /100 mg)	Max. 120	Max. 120	-	142.5	-	132.11	-
Cloud point (°C)	-	-	-15 to 5	7	3	-	-
Pour point (°C)	-	-	-2.0	-28	-5	-	-9
Cetane number	Min. 51	Min. 47	49.7	52	-	-	51.2
Fire point (°C)	-	-	-	189	-	-	-
Oxidation stability (110 °C, h)	Min. 6	Min. 3	25.8	2.75	-	-	-
Ash content	-	-	-	0.002	-	-	0.01
Specific gravity	-	-	-	0.83134	0.85	-	-
Free fatty acid (%)	-	-	-	0.4	-	-	-
Cold filter plug point (°C)	Max.19	Max.19	-16	-25	-	-	-

References for comparative analysis: RSOB⁽⁴²⁾: *Rubber seed* oil biodiesel, AAOB⁽⁴³⁾: *Ailanthus altissima* oil biodiesel, and HMOB⁽⁴⁴⁾: *Hodgsonia macrocarpa* oil biodiesel

3.2. Optimization of Reaction Conditions on the Transesterification of AF Oil

We investigated different parameters of the transesterification process to obtain maximum biodiesel yields, such as methanol/oil molar ratio, catalyst concentration, agitation intensity, reaction time, and reaction temperature. The detailed optimization process is shown in Fig. 4, and Table S3 (Supporting Materials). FAMEs initial income was only 91.5%, but after optimization, it increased to 96%. We studied five different constraints, applied different conditions to them, and tested each parameter under three different states to confirm that this is the most appropriate condition to obtain the best results. The five configurations are as follows and discussed one by one.

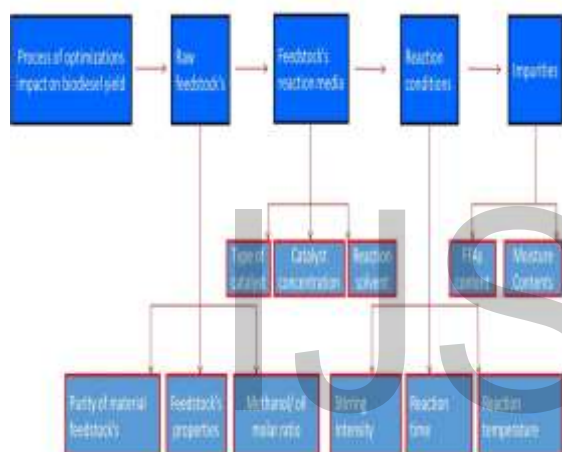


Fig. 4. Transesterification reaction of AF biodiesel.

3.2.1. Influence of Methanol to Oil ratio on FAMEs Yield

One of the most critical variables that affect FAMEs performance is the molar ratio of methanol to oil. The main goal of an ideal set of process variables is to maximize product output and minimize soap formation. The effect of the molar ratio of methanol to oil (4:1, 5:1, 6:1, and 7:1) on the yield of biodiesel is shown in Fig.5. It was observed that the molar ratio of methanol to oil increased from 4:1 to 6:1, the production of biodiesel increased significantly (96%), and additional increases in the molar ratio (7:1) resulted in a slight decrease in the production of biodiesel (90%). Besides, to separate the glycerin from biodiesel is very difficult. Because of high solubility, the higher alcohol molar ratio interferes with the separation of glycerol. Therefore, the excessive glycerol in the biodiesel phase permits the stabil-

ity reaction to proceed backward and recombine with the methyl ester to form monoglycerides (45). The results show that excessive methanol use has no substantial positive impact on FAMEs production. The separation of esters and glycerin is complicated. Generally, acid-catalyzed methods require higher molar ratios than base-catalyzed methods. It can be seen that the primary variables affecting the yield of esters are the types of alcohol and triglycerides. Generally, short-chain alcohols such as methanol, ethanol, propanol, and butanol can be used in the transesterification reaction to obtain a high ester yield.

One explanation of this result is that at molar ratios of methanol to oil greater than optimum level (6:1), the separation of esters layer from glycerol is difficult. The excess methanol hinders gravity decantation so that the visible yield of AFOMEs decreases because a portion of the glycerol and other contaminants remain in the biodiesel phase (46). In another study, Goff et al. (47) reported that the molar ratio more than 6:1 had no substantial effect on the production of esters but further methanol addition caused the problematical ester recovery and also increased process cost. Methanol/oil molar ratio >6:1 might depicted a dilution effect, whereas, for molar ratio <6:1 can cause insufficient mixing of the reactants in the biphasic transesterification reaction system, which might lead to lower ester yields. Our present study results are in agreement with those of Meher, Vidya, and Dharmagadda (37), Usta (48), and Rashid and Anwar (39) who reported best esters yields utilizing the molar relation around 6:1 during the methanolysis of *Pongamia pinnata*, tobacco, and rapeseed oils, respectively.

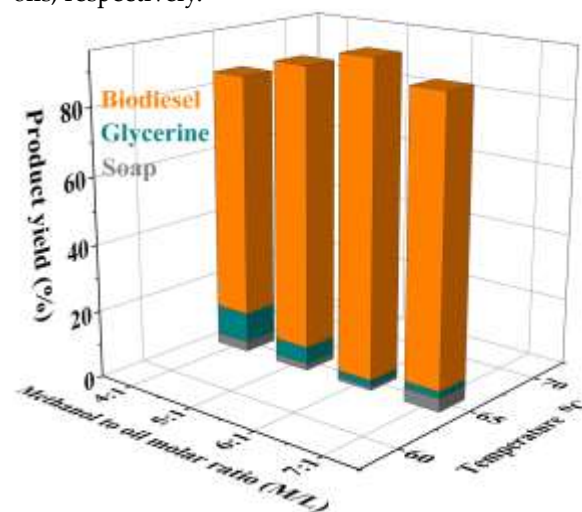


Fig. 5. Effect of molar ratio on biodiesel yield.

3.2.2. Influence of Catalyst Mediation on Biodiesel Yield

Various catalysts have been used for the transesterification of triglycerides, such as alkali, acid, enzyme, or heterogeneous catalysts. Although basic catalysts such as sodium methoxide (NaOMe) and potassium methoxide (KOMe) are more effective (49). The effectiveness of this attention on the transesterification of AF oil was evaluated, and the catalyst was found to exhibit the best catalyst concentration and activity. Therefore, we have applied and used six different catalysts (KOH, NaOH, CaO, OCH₃Na, CH₃OK, and ZrO₂) to check which catalyst is more effective. Based on the maximum/optimal conversion rate (96%), KOH continues to conduct further parameter research and optimization. In this study, potassium hydroxide, the most commonly used catalyst, was optimized based on maximum conversion yield. The screening data of the tested catalysts were shown in Fig.6. The reaction product is expressed as a percentage of FAMES in the reaction. The concentration of the substance is an important parameter in this study. The catalyst (KOH) meditation selected in this study was 2.0, 2.5, 3.0, 3.5 and 4.0 wt. % (based on the mass of crude oil). Throughout the transesterification reaction, the operating conditions for producing biodiesel from AF oil were: reaction temperature 65°C, reaction time 60 min, molar ratio of oil to methanol of 6:1, and stirring rate of 700 rpm. The results show that the effective KOH concentration needed for transesterification of AF oil was 3.0 wt. % (94%). It was observed that if the KOH concentration is reduced below or above the optimal value, the production of biodiesel will not increase significantly; on the contrary, the formation of glycerol and emulsion will increase. This may be due to the free acid content in the oil. As the catalyst concentration increased to 4.0 wt. %, the yield of methyl esters decreased to 91%. This is the result of (50, 51), which reports that the formation of soap in the presence of a large amount of catalyst increases the viscosity of the reactants, resulting in a decrease in the FAMES yield. This decline is understandable because the addition of an excessive amount of alkaline catalyst leads to the formation of an emulsion through increasing viscosity and complicates the recovery of the methyl esters (52, 53). In fact, this restricts the glycerine separation and as a result, saponification consumes the base catalyst and reduces product yields (40).

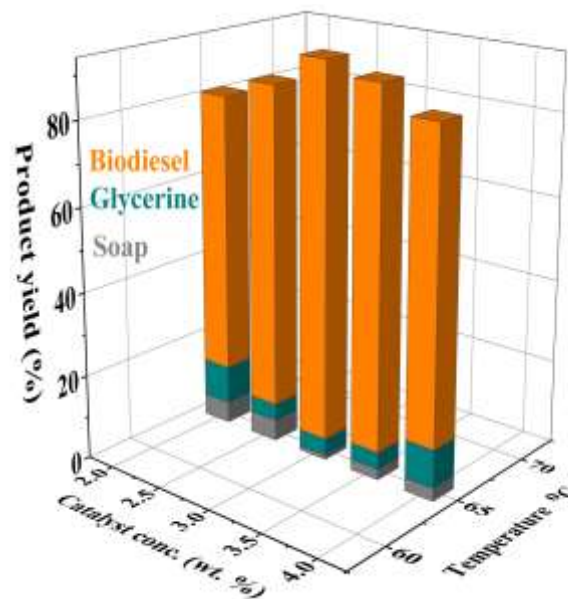


Fig. 6. Effect of catalyst concentration on biodiesel yield.

3.2.3. Effect of Reaction Temperature On Biodiesel Production

The reaction rate is very temperature-dependent. However, even at room temperature for a long enough time, the reaction is almost complete (49). Generally, the response is conceded out around the boiling point of methanol (60°C-70°C) and atmospheric pressure (54). Depending on the oil used, the transesterification reaction takes place at different temperatures. In the base-catalyzed transesterification reaction, the researchers maintained a series of steps in the temperature range of 65°C to 75°C. The boiling point of methanol is 64.9°C. Temperatures above this temperature will burn alcohol, resulting in a decrease in yield. To transesterify the refined AF oil to methanol (6:1) and 3.0 wt% KOH, the reaction was studied at four different temperatures of 60, 65, 70 and 75 °C. As shown in Fig.7, it was found that as the temperature increased from 60°C to 65°C, biodiesel production increased. The maximum yield (96%) was obtained at 65°C. According to reports, a further increase in temperature (70°C and 75 °C) will impair the conversion rate (90% and 85%) (55). Temperature affects the reaction rate and ester yield (56).

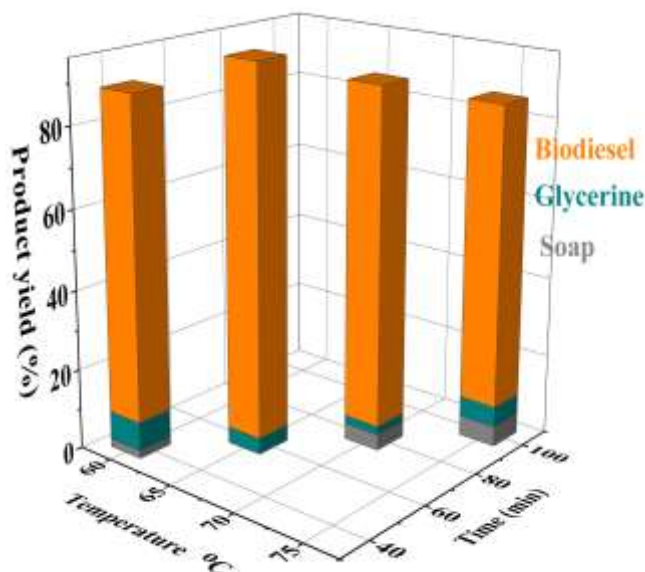


Fig. 7. Effect of temperature on biodiesel yield.

3.2.4. Reaction time influence on FAMES production

With reaction time, the conversion rate of methyl ester increases. The transesterification reaction is conceded out at the optimal reaction temperature (65°C) for different periods of 40 to 100 mins. Fig.8 shows the effect of this factor/reaction time on the performance of FAMES, indicating that the fatty acid methyl ester yield is lower (87%) within 40 mins after the start of the reaction, and the maximum result (95%) is reached within 60 mins. When the reaction time increased, the yield decreased slightly within 80 mins and decreased to (93%) and (90%) within 100 mins. This is inconsistent with literature data, indicating that due to the transesterification (hydrolysis) reverse reaction, longer reaction time will result in a lower yield, which usually results in the formation of more fatty acid soaps (57, 58).

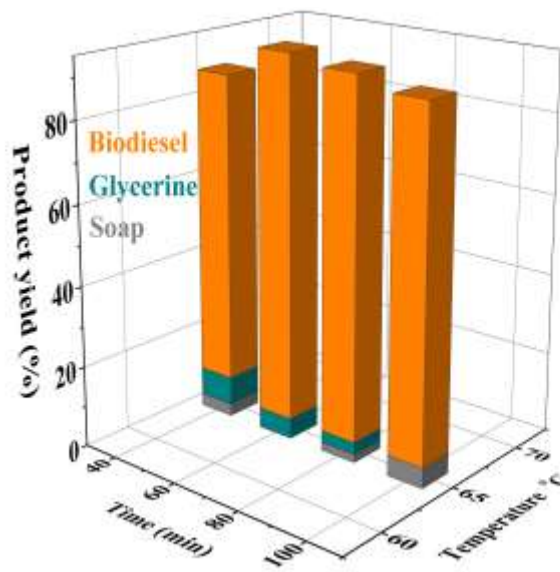


Fig. 8. Effect of time on biodiesel yield.

3.2.5. Influence of agitation rate on FAMES yield

The agitation rate is essential for the transesterification reaction because the grease is not miscible with the potassium hydroxide methanol solution. The reaction can be controlled by dissemination because the weak diffusion between the phases will cause the speed to decrease. Once the two parts are mixed and the response begins, no stirring is required. In four conditions/experiments, the effects of stirring on AFOMEs were studied at different stirring speeds (500, 600, 700 and 800 rpm). In all operations, the oil/methanol molar ratio was 6:1, the reaction temperature was 65°C, and the (KOH) catalyst concentration was 3.0 wt.%. As shown in Fig.9, the direct relationship between the stirring rate and AFOMEs production was clarified. That is, as the stirring speed increases, an increase in productivity is observed. Therefore, the 700 rpm mixing speed provides AFOMEs with the best AF oil conversion rate (96%). This is inconsistent with previous studies (59, 60, 61), which concluded that increasing the stirring speed will promote the homogenization of the reactants, consequential in sophisticated yields of methyl esters. Further increase in stirring rate (800 rpm) will affect negatively the conversion yield (88%).

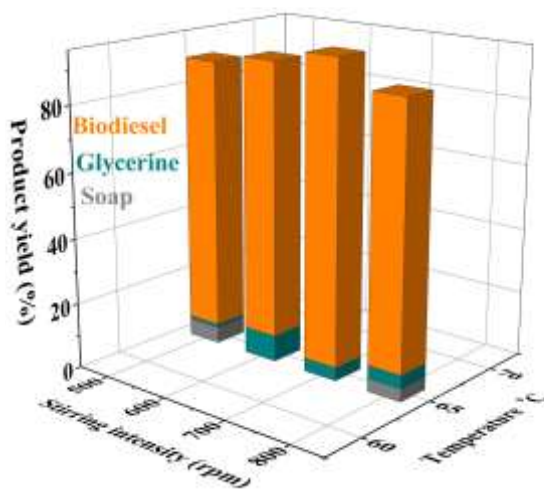


Fig. 9. Effect of stirring intensity on biodiesel yield.

3.3. FTIR Analysis

FT-IR spectroscopy is used to characterize and confirm the biodiesel produced by AFO (Acacia farnesiana oil). Esters have two characteristic impact absorption bands, which are generated by the stretching of methoxycarbonyl groups and the stretching of CO (62). The methoxycarbonyl group in A. farnesiana oil biodiesel shows the influential area at 1743 cm^{-1} . The FT-IR spectrum of A. farnesiana oil biodiesel is shown in Fig. 10 and Table S4 (Supporting Material). The small change in carbonyl frequency is due to the electron-donating effect of the methyl and C=O groups present in biodiesel. FT-IR spectroscopy shows that the characteristic band of fatty acid methyl ester corresponds to asymmetric and symmetric CH_3 stretching vibration ($-\text{CO}-\text{O}-\text{CH}_3$), the range is $3000\text{--}2854\text{ cm}^{-1}$, and the methyl group stretching band appears at 2926 cm^{-1} . However, the methylene stretching band appears in the asymmetric CH_3 deformation vibration at 2854 cm^{-1} and 1434 cm^{-1} respectively, the bending wave of the methyl group appears at 1434 and 1360 cm^{-1} , and the rocking (bending) of the methylene group appears at of 723 cm^{-1} . Due to the presence of the CO stretching vibration in biodiesel shows two asymmetric coupling vibrations at 1170 cm^{-1} v C-C(=O)-O and 1016 cm^{-1} , while 1170 cm^{-1} corresponds to CH_3 rocking vibration. And $1244\text{--}1015\text{ cm}^{-1}$ corresponds to the stretching vibration of the C-O ester group. This result reflects the conversion of triglycerides to methyl esters (63).

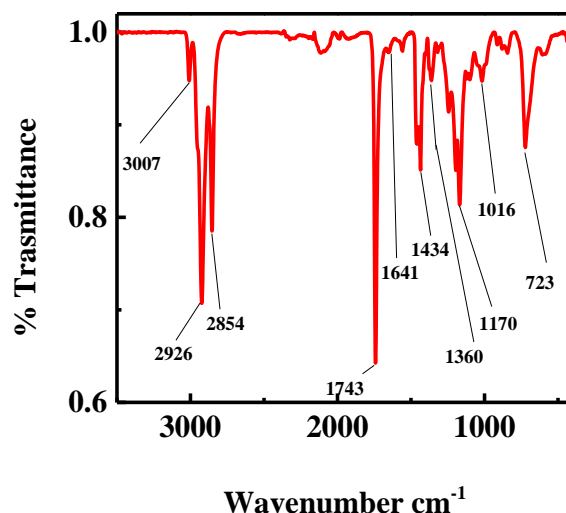


Fig. 10. AFOMEs FTIR analysis.

3.4. NMR analysis

The ^1H and ^{13}C NMR spectra of AFMEs were also recorded and shown in (Fig.11, 12), Tables S5 and S6 (Supporting Material).

The ^1H NMR spectrum (Fig. 11) was consistent with the range of vegetable oil methyl esters with similar composition, which can be further confirmed by calculating the fatty acid spectrum using the peak integral value [64]. Therefore, observed a signal at 5.33 (CH CH), 3.66 (COOCH_3), 2.77 (CH CH CH_2 CH CH), 2.29 ($\text{CH}_3\text{OOC CH}_2$), 2.03 (CH_2 CH CH), 1.61 ($\text{CH}_3\text{OOC CH}_2$ CH $_2$), 1.27 (CH_2) and 0.88 (CH_2 CH $_3$) ppm.

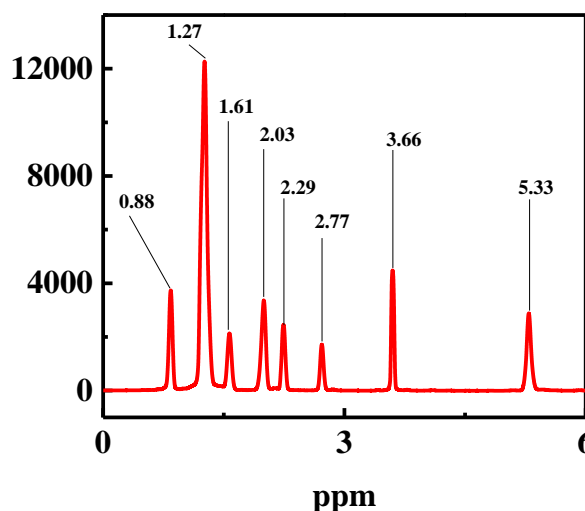


Fig. 11. ^1H NMR analysis of AFOMEs.

The signal in the ^{13}C NMR spectrums (Fig.12) was observed at 174.23 (COOCH_3). 130.1 And 127.89 (CH CH); 51.35 (COOCH_3); 34.1 (var-

ious CH₂); 22.55 (CH₂ CH₃); 14.07 (CH₂ CH₃) ppm.

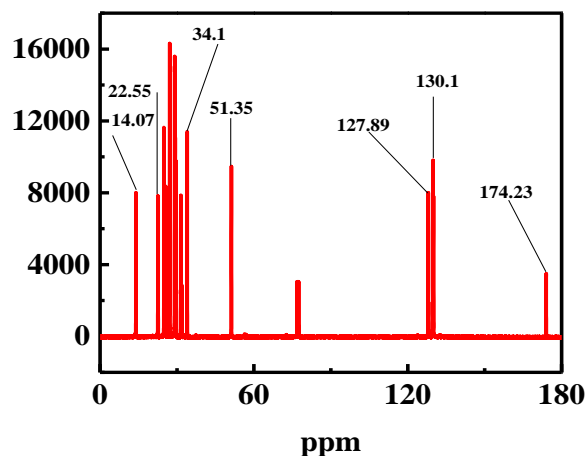


Fig. 12. ¹³C NMR analysis of AFOMEs.

3.5. Gas chromatography and mass spectrometry analysis

The chemical composition of the prepared AF oil biodiesel was accomplished by GC-MS chromatography. Five main peaks were detected by the GC spectrum of AF oil biodiesel (Fig.13). Each peak is identified from library matching software (NIST. 14) and corresponds to fatty acid methyl ester. The identified FAMES and its retention

time are shown in Table 4. First, the FAMES were identified by retention time data and then verified by mass spectrometry analysis. The chain length, the number of double bonds, and their positions are determined by mass spectrometry. Mass spectrometry was obtained by electron collision (EI) ion source (65).

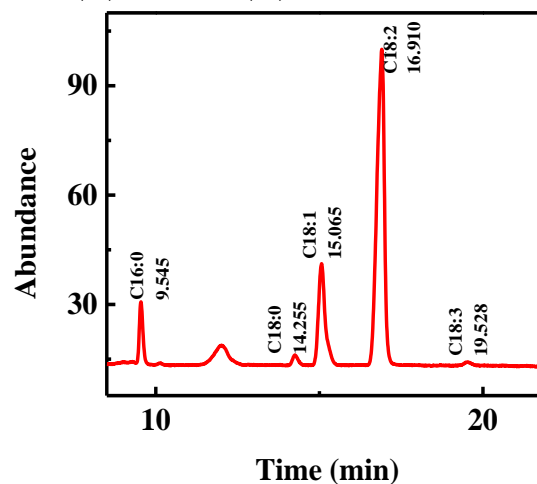
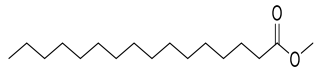
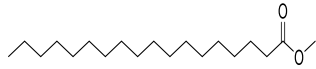
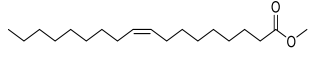
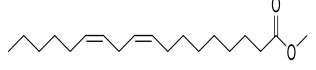
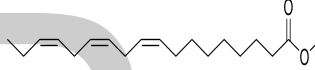


Fig. 13. GCMS study (Analysis) of AFOMEs.

Table 4 Fatty acid composition (GCMS analysis) of AFOMEs.

Fatty acids	Retention time	Number of carbons and double bond	Fatty acid (Weight %age)	Chemical name	Chemical structure	Molecular weight
Palmitic acid	9.545	C16:0	6.85	Hexadecanoic acid, methyl ester		270
Stearic acid	14.248	C18:0	2.36	Octadecanoic acid		298
Oleic acid	15.042	C18:1	12.13	9-Octadecenoic acid (Z) - , Methyl Ester		296
Linoleic acid	16.797	C18:2	46.85	9, 12-Octadecadienoic Acid (Z, Z)-, methyl ester		294
α -Linolenic acid	19.565	C18:3	1.23	9, 12, 15-Octadecatrienoic acid		278

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3.6. Elemental Analysis

Several elements in biodiesel can cause problems; that is why they are objectionable, such as stimulating biodiesel humiliation, engine performance degradation, operability problems, common complications (such as ecological pollution), and subsequent disease physical hazards (66). The main elements in the product from the biodiesel production mechanism are sodium (Na) and potassium (K), and their presence and concentration need to be controlled so that their content is below the limit of biodiesel. The highest acceptable concentration of Na and K in biodiesel was 5 mg kg⁻¹, while P and S is 10 mg kg⁻¹ (67). Extraordinary stages of Na & K causes soap creation. Further washing is required to reduce the sodium and potassium content below the specified in the standard. Supplementary essentials such as Ca, Mg and P are present in raw supplies used for biodiesel making. Phosphorus (P) comes from the raw materials in the product; its control amount in biodiesel is also essential. The element concentration of AF oil biodiesel (AFOBD) was compared with petroleum diesel, and the results confirmed that the element concentration of AFOBD was relatively lower than that of petroleum diesel. Elements in biodiesel, such as potassium (K), sodium (Na), magnesium (Mg), and calcium (Ca), are directed to the fuel injection nozzles to promote drainage, wear of pistons and piston rings, and locomotive passing filter plugs precipitation (68). Table S7 (Supporting Material) shows the detailed AFOB element concentration results, and the AFOBD K, Na, Mg and Ca concentrations are (4.9, 25.2, 1.26, 10.3 µg/g), which is lower than the level of petroleum diesel (213.3, 868.3, 35.6 and 21.4 µg/g). However, the maximum acceptable concentration of sulfur (S) and phosphorus (P) in biodiesel is 10 mg kg⁻¹, while Na and K are 5 mg kg⁻¹ (Fig.14). The presence of other elements such as Ca, Mg, and P comes from the raw materials used in biodiesel production. If the Na concentration is high enough, it will help to form soap while reducing the yield of FAMES. In this case, if the content of the above elements is too high, further cleaning is required to reduce the content below the prescribed standard.

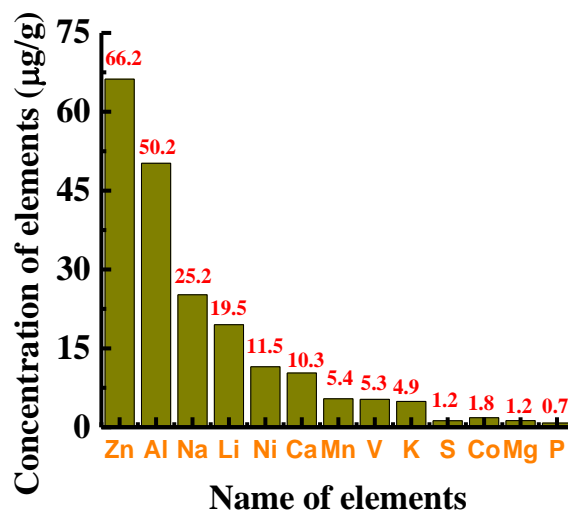


Fig. 14. AFOB FAMES ICP-OES study.

The C, H, N, and O documented meditations are declared in (Fig. 8b) and Table S8 (Supporting Materials). A significant difference between biodiesel and petroleum diesel is the presence of oxygen. Biodiesel contains oxygen, while diesel does not. The presence of oxygen can increase the combustion atmosphere, reduce the ignition delay time, and make the fuel burned more fully, thereby reducing the emissions of CO, PM, and other exhaust gases. Many researchers (69, 70) found that if the oxygen content in biodiesel is high, it can effectively reduce PM emissions in diesel engines. Fig. 8b shows that the oxygen content of AF BD100 is 8.11%, but the oxygen content in biodiesel is usually about 10% (71, 72). The higher hydrogen content in biodiesel is attractive for its use as a fuel (13.34%) (73). A high H/C ratio means that the hydrogen molecules in the fuel are more advanced because hydrogen has the highest burning rate of all fuels (gases or liquids). Increasing the hydrogen portion of the fuel mixture indicates faster and cleaner fuel burn. Since the heating value of hydrogen is higher than that of carbon, the ratio of H to carbon atoms increases, so the heating value usually increases.

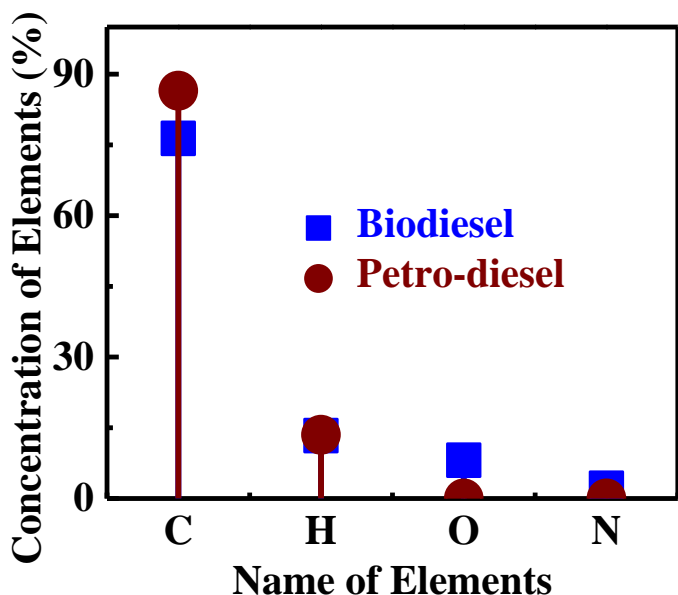


Fig. 15. AFO FAMES EA analysis.

4. CONCLUSIONS

A comprehensive study of AFO was conducted for the yield of biodiesel. The overall results confirm that AFO can be used as a resource for biodiesel manufacturing. AF has a variety of wild and cultivated resources, and the crude AF seed oil was used and transesterified by using KOH as an alkaline catalyst and methanol to form biodiesel. The highest biodiesel yield conversion of 96% was obtained through optimized protocol during the alkaline-catalyzed transesterification of reaction temperature 65 °C with a molar ratio (methanol to oil) of 6:1 of KOH (2.9 wt.%) at a mixing rate of 700 rpm and time of 60 mins. Most features of AFSOME fit well to ASTM D-6751, EN 14214, and high speed diesel, especially viscosity (5.32 mm²/s at 40 °C), flashpoint (158 °C) and Cetane number (52) are excellent. In general, AFSOMEs can be used as a potential substitute for non-edible oil source biodiesel production. The fuel properties of AF oil methyl ester compared to the recognized biodiesel standard, indicating that the transesterification of the oil improves its performance and makes it similar to petroleum diesel. Since China's environment and soil are suitable for AF planting, with all of these characteristics in mind, it can be concluded to overcome future energy crises and achieve the production on a larger scale. Overall, AF is a potential low-cost and high-quality raw material for biodiesel production in China and other subtropical regions.

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AUTHORS' 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. Chen Hang helped in seed collection and revised the manuscript. SAHS edited, formatted the manuscript and worked on bibliography.

DECLARATION OF INTEREST STATEMENT

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 paper.

ETHICAL STATEMENT

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

SUPPORTING DATA

The supporting data is provided.

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