

Article

Deacidification of *Pistacia chinensis* Oil as a Promising Non-Edible Feedstock for Biodiesel Production in China

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Abstract: *Pistacia chinensis* seed oil is proposed as a promising non-edible feedstock for biodiesel production. Different extraction methods were tested and compared to obtain crude oil from the seed of *Pistacia chinensis*, along with various deacidification measures of refined oil. The biodiesel was produced through catalysis of sodium hydroxide (NaOH) and potassium hydroxide (KOH). The results showed that the acid value of *Pistacia chinensis* oil was successfully reduced to 0.23 mg KOH/g when it was extracted using ethanol. Consequently, the biodiesel product gave a high yield beyond 96.0%. The transesterification catalysed by KOH was also more complete. Fourier transform infrared (FTIR) spectroscopy was used to monitor the transesterification reaction. Analyses by gas chromatography-mass spectrometry (GC-MS) and gas chromatography with a flame ionisation detector (GC-FID) certified that the *Pistacia chinensis* biodiesel mainly consisted of C₁₈ fatty acid methyl esters (81.07%) with a high percentage of methyl oleate. Furthermore, the measured fuel properties of the biodiesel met the required standards for fuel use. In conclusion, the *Pistacia chinensis* biodiesel is a qualified and feasible substitute for fossil diesel.

Keywords: biodiesel; *Pistacia chinensis* seed oil; deacidification; transesterification

1. Introduction

Biodiesel is a renewable, green alternative fuel. Compared with fossil fuels, it has many advantages such as environmental friendliness and better efficiency. Many countries throughout the World have attempted to expand the use of biodiesel along with their industrial development. However, the high cost of feedstocks required for biodiesel production, accounting for roughly 60% to 80% of the total cost, is a critical restraining factor in its universal commercialisation. [1]. Currently, raw materials for over 95% of commercially produced biodiesel come from edible oils, such as soybean, rapeseed and palm oil. Large-scale production can be undertaken by the agricultural industry [2], but the excessive use of edible oil to produce biodiesel may cause a series of problems in the World, especially in developing countries including China. The negative impacts of biodiesel production lie in the excessive consumption of crops originally grown to provide food originally and the encroachment on already limited arable lands. These problems lead to the rising prices of edible oils and economic imbalance. Researchers have attempted to search for cheaper non-edible oils as alternative feedstocks for use in biodiesel production. These include *Jatropha* oil [3,4], *Pongamia* oil [5], *Cornus wisoniana* oil [6] and *Cerbera* oil [7]. Recently, seed oils from Okra (*Hibiscus esculentus*), *Moringa oleifera* and Milo (*Thespesia populnea*), widely distributed in the tropical and subtropical regions, have also been used as suitable feedstocks for biodiesel production [8–10]. However, due to the importance of considering geographical location and climate in different countries in the development of potential biodiesel feedstocks, some oil-producing plants such as *Pistacia chinensis* are proposed for use in China [11]. This plant can grow in lands that are unsuitable for growing crops, making it suitable for the agricultural conditions in the country [12].

Pistacia chinensis is widely distributed throughout most provinces in China because it is very adaptable; moreover, its seeds have a high oil content of approximately 40% [13]. At present, studies on *Pistacia chinensis* are mainly being reported from China [14] and focus on the biological characteristics and agricultural techniques in propagation and cultivation [15,16], as well as the physical and chemical properties of seed oil [17,18]. There are few reports about using *Pistacia chinensis* as feedstock to produce biodiesel [19]. However, the high free fatty acid (FFA) content of *Pistacia chinensis* seed oil may be a major hindrance to the biodiesel production due to the use of dominant industrial basic catalysts [20]. It is well known that the higher FFA content of the feedstock greatly reduces biodiesel yield when using alkaline-catalysis technology. This reduction is attributed to the consumption of catalyst and the difficulties involved in separating and purifying the product [21]. Surprisingly, the acid values of *Pistacia chinensis* seed oils in China range from 9.7 mg/g to 79.7 mg/g due to their different provenances or oil extraction processes [18]. Most of the values, ranging from 10.0 mg/g to 20.0 mg/g, are still considerably high for biodiesel production. The content of FFAs in oil should be reduced to less than 0.5 wt% before an alkaline catalyst could be used for biodiesel production [22].

The first part of this paper focuses on the extraction and deacidification of oil from the seeds of *Pistacia chinensis* collected from Hebei Province, China. The second part focuses on the transesterification of the extracted oil into fatty acid methyl esters. Transesterification reactions were carried out using two homogeneous basic catalysts, namely, sodium hydroxide (NaOH) and potassium hydroxide (KOH). These are the most mature and widely used catalysts in biodiesel production [23].

The reactions were monitored using Fourier transform infrared (FTIR) spectroscopy. The biodiesel components were analysed qualitatively and quantitatively by gas chromatography-mass spectrometry (GC-MS) and gas chromatography with aflame ionisation detection (GC-FID). Finally, several physicochemical parameters were tested and compared with the ASTM D 6751 and EU 14214 standards to ensure biodiesel quality. The experimental results show that extraction by solvents, which is first reported when dealing with *Pistacia chinensis* crude oil, is an effective method in reducing FFA content. The results also show that *Pistacia chinensis* seed oil is a suitable and promising feedstock for biodiesel production in China.

2. Experimental

2.1. Materials and Apparatus

Crude oil of *Pistacia chinensis* was extracted from the seeds, which were purchased in Forestry Co. of Shexian County in Hebei Province, China, and was stored at 4 °C in the refrigerator. Analytical reagents included methanol (purity > 99.8%), sodium hydroxide (purity 99%), potassium hydroxide (purity 88%), *n*-hexane (purity 99%), petroleum ether (boiling point 30~60 °C), anhydrous sodium sulfate (purity 99%), etc. The analytic apparatus mainly includes Avatar 370 FTIR Spectrometer (Nicolet Co., Madison, WI USA), 6890N/5973N gas chromatography-mass spectrometer, 6890 Gas chromatography (Agilent Co., Wilmington, DE USA), DSY006B Petroleum Pour/Cloud/Solidification/Cold filter plugging tester, SYP1002 Petroleum Flash point tester (Fuli Co., Shanghai, China), and FDR-3601 Octane/Cetane number meter (Fulande Co., Changsha, China).

2.2. Oil Extraction and Biodiesel Production Procedures

The *Pistacia chinensis* seeds were dried at 40 °C in an oven to remove excess moisture before the extraction process. They were then ground into fine particles. These particles, were extracted with *n*-hexane as the solvent, and the crude seed oil was recovered using a rotary evaporator.

For the homogenous method, the mixture for producing biodiesel was prepared manually by adding 1.0 wt% sodium or potassium hydroxide-methanol solution to the preheated crude seed oil, whose main components are triacylglycerols. The reactions were carried out in 250 mL round-bottomed glass flasks placed in a water bath with temperature control. The reaction mixture was vigorously stirred with mechanical stirrer set at 800 rpm stirring rate and refluxed under condensation system for required time. After the reaction, the excessive methanol was evaporated by heating and glycerine in the lower layer was removed; then the upper layer, mainly containing the desired biodiesel product, whose main components are fatty acid methyl esters (FAME), was washed with water. The ester layer after separation was dehydrated with anhydrous sodium sulfate. At last, the refined biodiesel was obtained through filtration.

2.3. Analysis

2.3.1. Physicochemical Parameters of Crude Oil and Biodiesel

The density (d_4^{20}) of crude oil was measured based on GB/T 5526-1985. The acid value (AV) was determined according to GB/T 5530-2005. The saponification value (SV) was determined from GB/T 5534-2008. Then the relative molecular weight (M) of crude oil was calculated using the formula [24]:

$$M = \frac{56.1 \times 3 \times 1000}{SV - AV} \quad (1)$$

The biodiesel yield was calculated from the biodiesel and oil weights using the following:

$$\text{Yield \%} = \frac{\text{Weight of biodiesel produced}}{\text{Weight of oil used in reaction}} \times 100\% \quad (2)$$

The determined properties of the biodiesel produced included the following: density (ASTM D 5002), kinematic viscosity (ASTM D445), flash point (ASTM D 93), cold filter plugging point (ASTM D 6371), cloud point (ASTM D 2500), pour point (ASTM D 97), ash content (ASTM D 874), acid value (ASTM D 664) and cetane number (ASTM D 613). Triplicate determinations were performed for every experiment, and the data were reported as means \pm standard deviation.

2.3.2. Biodiesel Chemical Composition

The FTIR spectra ranging from 400 cm^{-1} to 4000 cm^{-1} , were collected at 64 scans with a spectral resolution of 4 cm^{-1} . The spectra were analysed using the Omnic 6.1 data processing software. The components and composition of biodiesel were identified qualitatively and calculated quantitatively using GC-MS and GC-FID, respectively. GC-FID analyses were performed on an Agilent 6890 GC equipped with a FID. GC-MS analyses were performed on a same GC coupled to an Agilent 5973 quadrupole mass selective detector (MSD). Separation was achieved on a fused silica capillary column HP-5 ($30 \text{ m} \times 0.25 \text{ mm i.d.}$, $0.25 \mu\text{m}$ film thickness, stationary phase of 5% phenyl and 1% vinyl dimethyl polysiloxane). The GC operating conditions for both techniques were the same. Temperature increases were initiated from $100 \text{ }^\circ\text{C}$ to $280 \text{ }^\circ\text{C}$ at a rate of $4 \text{ }^\circ\text{C min}^{-1}$, with isothermal hold at $100 \text{ }^\circ\text{C}$ and $280 \text{ }^\circ\text{C}$ for 5 min, respectively. Hydrogen and helium were used as carrier gases for GC-FID and GC-MS, respectively. The mass spectrometer was operated in the electron impact mode at 70 eV ionisation energy and scanned from 30 Da to 600 Da. Data were acquired and processed using the instrumental software GC and MSD Chemstation.

3. Results and Discussion

3.1. Crude Oil Extraction and Parameter Determination

Four extraction methods were attempted to obtain oil from *Pistacia chinensis* seeds. They were carried out under respective optimum conditions according to single factor and orthogonal experiments (detailed data of some preliminary experiments are not provided to preserve the conciseness of this paper). Those processes included the following: (A) leaching at room temperature for 72 h, (B) simple

reflux extraction at 80 °C for 5 h, (C) Soxhlet extraction at 80 °C for 8 h, and (D) ultrasonic extraction for 30 min. The extracted crude oil was weighed, and the average oil content in the seed was calculated (Table 1). The results show that the Soxhlet extraction served as the most effective method in preparing crude oil, with an oil extraction rate reaching to 31.3%. This method is suitable for oil extraction in laboratories, whereas leaching may be used in large-scale industrial preparations because of its very limited energy consumption.

Table 1. Comparison of four extraction methods in preparing *Pistacia chinensis* seed oil.

Extraction method	Seed/g	Oil/g	Yield/%
Leaching	50	13.25	26.5
Reflux	30	7.50	25.0
Soxhlet	10	3.13	31.3
Ultrasonic	10	2.76	27.6

The physicochemical parameters of crude oil were determined using the methods stated in the Experimental. The density (d_4^{20}) was determined to be 0.8751 g/mL, whereas the average value of acid value (AV) was 33.40 mg KOH/g, which was much higher than those of most edible oils. The saponification value (SV) was 203.26 mg KOH/g, and the relative molecular weight (M) was calculated to be 990.8.

3.2. Deacidification of Refined Oil

In order to acquire qualified feedstock for biodiesel production under alkaline catalysis, crude oil has been refined through the degumming process consisting of the following steps: preheating, hydrating, centrifugalising and vacuum dehydrating. Then three methods were compared to check their respective effects on oil deacidification: alkali refining, pre-esterification catalysed by sulfuric acid and extraction by ethanol.

Alkali refining is a traditional method in reducing the acid value of crude oil. Alkali concentration and operating condition are related to the acid value of crude oil (Table 2) [24]. The stoichiometric alkali dosage is calculated using the following formula. Excessive alkali used in practice typically range from 0.05% to 0.25%:

$$G_{NaOH} = G_{oil} \times AV \times \frac{M_{NaOH}}{M_{KOH}} \times \frac{1}{1000} \quad (3)$$

Table 2. Alkali concentration and the operating temperature according to acid value of crude oil.

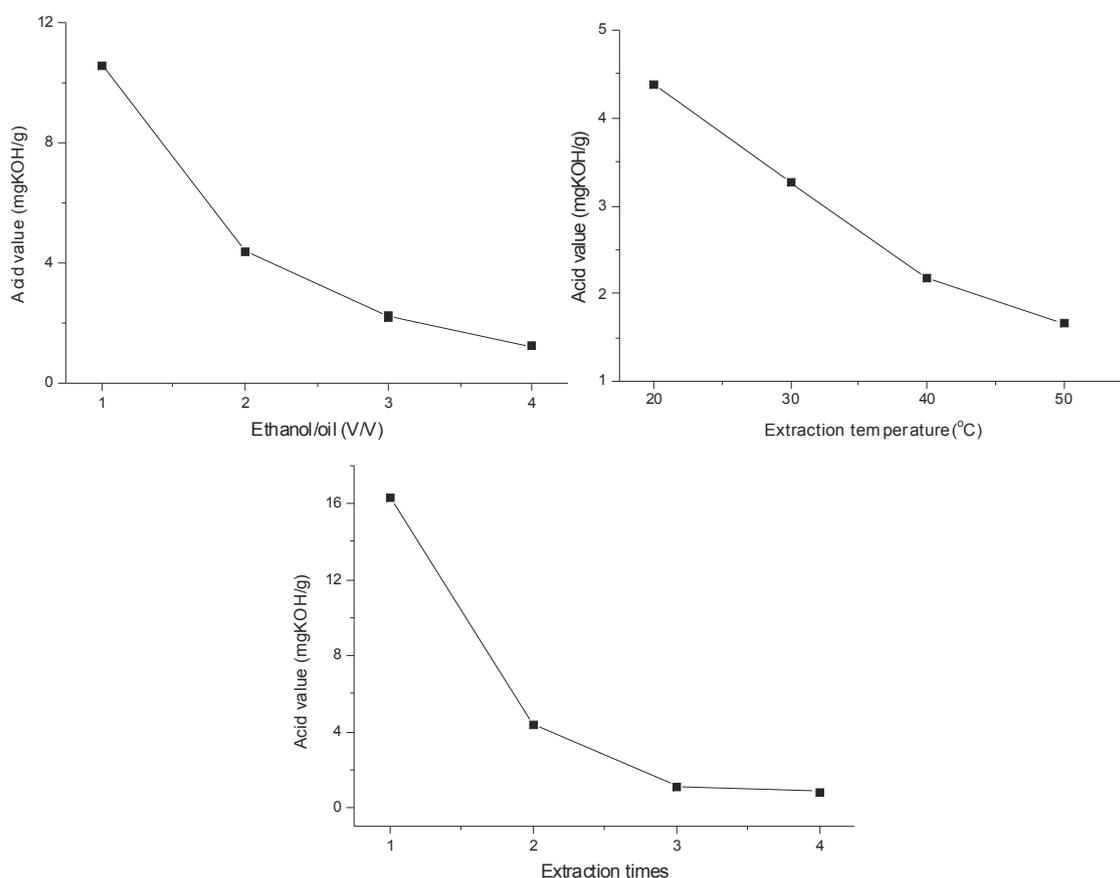
Acid value of crude oil	Alkali concentration/°Bé	Operating temperature/°C	
		Start	End
<5	4~6	75~80	90~95
5~7	12~14	50~55	60~65
7~9	16~24	25~30	45~50
>9	>24	20~30	20~30

Unexpectedly, the result showed that alkali refining was not a satisfactory method in dealing with *Pistacia chinensis* crude oil with so much high FFA content. Specifically, the acid value decreased to 2.10 mg KOH/g under the the following optimum conditions: 30.0 g crude oil, 0.2% extra sodium hydroxide 0.77 g, temperature increase from 20 °C to 30 °C, and stirring rate of 30 r/min.

Pre-esterification can be also regarded as the first step performed in two-step biodiesel production using oil with high FFA content as feedstock [25]. With methanol, the high content FFA can be transformed into fatty acid methyl esters (FAMES) through the catalysis initiated by sulfuric acid. In the present study, the experiment was carried out under optimum conditions, *i.e.*, the molar ratio of methanol to oil was 1 to 10, the amount of the catalyst addition was 3.0 wt%, reaction time was 4 h, and the temperature was controlled at 60 °C. However, we found that the deacidification result of the acid value 2.24 mg KOH/g was not ideal. Thus, the two-step biodiesel production method commonly used in feedstocks with high acid value may not be suitable in processing *Pistacia chinensis* seed oil.

Finally, given the solubility of FFAs in polar solvents (e.g., isopropyl alcohol, ethanol, and methyl alcohol), an extraction method with these solvents was attempted to reduce FFA content. This method is seldom reported when processing crude oil for biodiesel production. Based on our experimental data, this extraction method showed an excellent deacidification effect, and had shorter operating time and lower cost compared with two former methods. To obtain the best experimental conditions, three aspects that influence the deacidification were studied: volume ratio of oil to ethanol, extraction temperature, number of repetitions (Figure 1).

Figure 1. Single factor experiments of *Pistacia chinensis* oil deacidification with ethanol.



An orthogonal experiment featuring the three levels and the four factors (the fourth was a blank factor added) was designed (Table 3a,b).

Table 3. (a) L_9 (3^4) orthogonal experimental program of *Pistacia chinensis* oil deacidification by ethanol; (b) Orthogonal experimental results of *Pistacia chinensis* oil deacidification by ethanol.

(a)					
Levels	The volume ratio of oil to ethanol (A)	Extraction temperature (B)	Extraction times (C)	Blank	
1	1:1	20 °C	1	1	
2	1:2	30 °C	2	2	
3	1:3	40 °C	3	3	

(b)					
Test No.	The volume ratio of oil to ethanol (A)	Extraction temperature (°C) (B)	Extraction times (C)	Blank	Acid value (mg KOH/g)
1	1:1	20	1	1	22.51
2	1:1	30	2	2	9.87
3	1:1	40	3	3	4.37
4	1:2	20	2	3	4.38
5	1:2	30	3	1	0.55
6	1:2	40	1	2	13.62
7	1:3	20	3	2	1.13
8	1:3	30	1	3	9.03
9	1:3	40	2	1	1.65
K_1	36.75	28.02	45.16	24.71	
K_2	18.55	19.45	15.90	24.62	
K_3	11.81	19.64	6.05	17.78	
\bar{K}_{13}	12.250	9.340	15.053	8.237	
\bar{K}_{23}	6.183	6.483	5.300	8.207	
\bar{K}_{33}	3.937	6.547	2.017	5.927	
R	24.94	8.57	39.11	6.93	
Priority factors	$C > A > B$				
Optimum solution	$K_{3A} < K_{2A} < K_{1A} \quad K_{2B} < K_{3B} < K_{1B} \quad K_{3C} < K_{2C} < K_{1C}$				
	$A_3B_2C_3$				

The experimental results revealed that the optimum parameter combination of *Pistacia chinensis* oil deacidification was $A_3B_2C_3$; this meant that the volume ratio of oil to ethanol was 1:3, extraction temperature was 30 °C, and the number of repetitions was 3. After performing three confirmatory experiments under optimum conditions, we also found that the average acid value was reduced to 0.23 ± 0.02 mg KOH/g. This value was much lower than that for alkali refining and pre-esterification under optimum conditions.

3.3. Biodiesel Production and Analysis

Pistacia chinensis biodiesel was produced under homogeneous basic catalysis by NaOH and KOH. The optimum reaction conditions were as follows: 6:1 molar ratio of methanol to oil, addition of 1.0 wt% catalyst, 60 min reaction time, and 60 °C reaction temperature. In a former study conducted by the researchers using the same experimental conditions [20], the average biodiesel yield from the seed oil of *Pistacia chinensis* plant from an identical location was only 67.5%. The reason for the lower biodiesel yield in that study was the corresponding lower quality of the oil. Furthermore, the acid value of refined oil was reduced to 2.10 mg KOH/g through the process of degumming and alkali refining. When using the homogeneous alkaline catalysis, the higher FFA content of the feedstock generally reduce biodiesel yield due to the consumption of catalyst and the difficulties involved in separating and purifying of the product [21]. In contrast, all parallel experiments yielded more than 96.0% biodiesel, because the acid value of 0.23 mg KOH/g was converted into an FFA content of approximately 0.13 wt% after ethanol deacidification. This value is much less than 0.5 wt%, which is the upper limit in biodiesel production under basic catalysis.

Furthermore, it can be inferred that transesterification was thoroughly catalyzed by both NaOH and KOH. However, the biodiesel yield (97.06 ± 0.14) under KOH catalysis was slightly higher than that of NaOH catalysis (96.62 ± 0.30). From the chemometrics aspect, 1.4 wt% KOH offered an equivalent OH⁻ of 1 wt% NaOH, Meanwhile only 1 wt% KOH can give better catalysis effect than 1 wt% NaOH. This may be caused by the stronger alkalinity of KOH, which promoted the reaction more completely. Consequently, KOH was identified as a better catalyst candidate than NaOH in terms of chemical behaviour. This finding corresponds with the results of similar studies on transesterification reactions [26,27]. In addition, the use of KOH is more economically feasible because at the end of the reaction, the mixture can be neutralised with phosphoric acid to produce potassium phosphate—a type of fertiliser [28].

3.4. Biodiesel Analysis

The biodiesel produced from *Pistacia chinensis* was characterised using FTIR spectroscopy. FTIR spectra show characteristic FAME bands (Figure 2). In the functional group –CO–O–CH₃ of the FAME, which was generated during the transesterification, 2854 cm⁻¹ and 2925 cm⁻¹ were attributed to the stretching and asymmetric stretching vibrations of –CH₃; 1436 cm⁻¹ corresponded to the asymmetric bending vibration of –CH₃; 1196 cm⁻¹ was due to the stretching vibration from O–CH₃; and 1743 cm⁻¹ was assigned to the vibration of –C=O. These results reflect the conversion of triacylglycerols into methyl esters [29].

The main components of biodiesel from *Pistacia chinensis* were analysed qualitatively and quantitatively through GC-MS and GC-FID (Figure 3). The components included methyl palmitate, methyl linoleate, methyl oleate and methyl stearate (Table 4). The minor components included other kinds of FAMES and traces of monoacylglycerols. The proposed chemical structure of biodiesel is C₁₉H₃₆O₂ [24]. This suggestion means that C₁₈ FAMES –including high percentage of methyl oleate–should be dominant in the biodiesel. Therefore, this *Pistacia chinensis* biodiesel with 81.07% C₁₈ FAMES is a chemical qualified product as biofuel.

Figure 2. FTIR spectrum of *Pistacia chinensis* biodiesel.

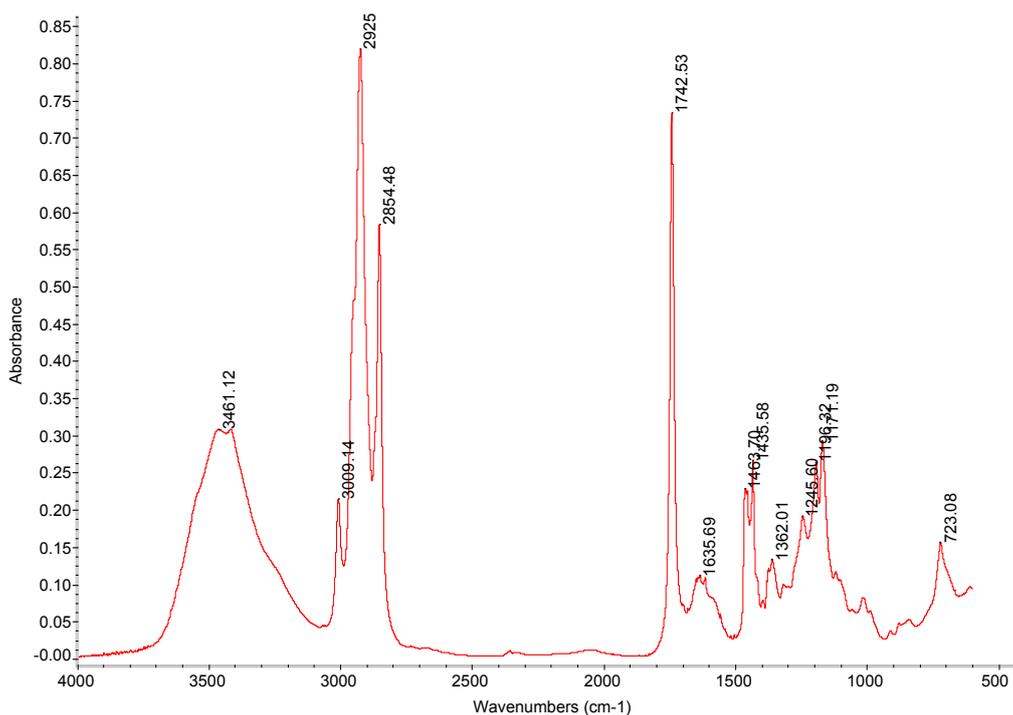


Figure 3. GC-MS (TIC) traces of *Pistacia chinensis* biodiesel.

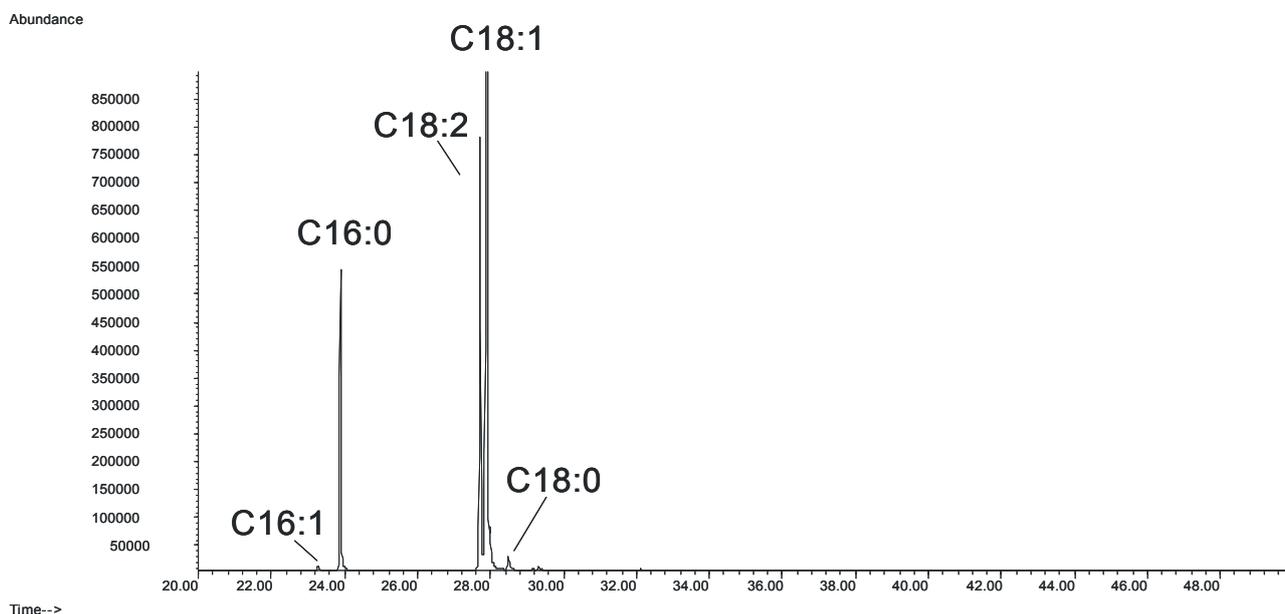


Table 4. Composition (%) of biodiesel of *Pistacia Chinensis*.

Components	Methyl palmitate	Methyl linoleate	Methyl oleate	Methyl stearate
Formula	C16:0	C18:2	C18:1	C18:0
Composition (%)	18.93 ± 0.28	27.42 ± 0.40	52.32 ± 1.15	1.33 ± 0.16

With the aim to test whether or not the *Pistacia chinensis* biodiesel meets the required standards for fuel use. In line with this, several key fuel properties of the refined product have been identified. These properties are given in Table 5. The cetane number reflects the ignition quality of diesel fuel, which

strongly affects engine performance in terms of combustion and exhaust emissions among others. The greater the cetane number, the more ignitable the fuel is [8]. The cetane number (52.2) of *Pistacia chinensis* biodiesel meets the minimum requirements mandated in both the ASTM D6751 and EN 14214 biodiesel standards. The kinematic viscosity affects the atomisation when a fuel is injected into the combustion chamber; this process may lead to the formation of soot and engine deposits [8]. The kinematic viscosity values of methyl oleate, methyl linoleate, and methyl palmitate are 4.51, 3.65 and 4.38 mm² s⁻¹ at 40 °C, respectively [30]. The kinematic viscosity (5.24 mm² s⁻¹) of *Pistacia chinensis* biodiesel falls within the specified range in ASTM D6751. However, the value is slightly higher than that stated in the EN 14214 biodiesel standards, which may be due to the high percentage of methyl oleate. Although no limits are specified in both standards, the relatively low values of cloud point (1 °C), pour point (-2 °C), and cold filter plugging point (0 °C) of the biodiesel produced in the present study, compared with those of biodiesel from other non-edible feedstocks [8–10], show that *Pistacia chinensis* biodiesel has good cold flow properties. The higher flash point value of the product (166 °C) also reduces the risk of fires, which is one of the superior properties of biodiesel. Other fuel properties of this *Pistacia chinensis* biodiesel include density (887 kg m⁻³), ash content (0.013%) and acid value (0.35 mg KOH g⁻¹). These values are also comparable with those of diesel according to ASTM D6751 and EN 14214 standards (Table 5).

Table 5. Property comparison of *Pistacia chinensis* biodiesel with ASTM D6751 and EN 14214 standards.

Fuel property	Biodiesel	ASTM D6751	EN 14214
Density (15 °C), kg m ⁻³	887 ± 9.6 *	-	860–900
Kinematic viscosity (mm ² s ⁻¹ ; 40 °C)	5.24 ± 0.05	1.9–6.0	3.5–5.0
Flash point (°C)	166 ± 2.0	130 min	120 min
Cloud point (°C)	1.00 ± 0.12	Report	-
Pour point (°C)	-2.00 ± 0.15	-	-
Cold filter plugging point (°C)	0.00 ± 0.10	-	-
Ash content (%)	0.013 ± 0.001	0.02 max	0.02 max
Acid value (mg KOH g ⁻¹)	0.35 ± 0.03	0.50 max	0.50 max
Cetane number	52.2 ± 1.30	47 min	51 min

* Values are mean ± SD of triplicate determination.

4. Conclusions

The *Pistacia chinensis* oil-bearing plant is widely distributed in China, and represents an ideal and promising feedstock for biodiesel production. However, the high free fatty acids content of its crude oil may be a handicap in achieving widespread industrial applications. After comparing three deacidification methods, we find that extraction with ethanol is the most effective. In this method, the acid value of refined oil was successfully reduced to 0.23 mg KOH g⁻¹. Using this qualified oil, biodiesel with high yield (>96.0%) has been produced under catalysis by potassium and sodium hydroxides. KOH demonstrated better catalysing effect than NaOH. Finally, the characteristics of the FTIR spectra indicated the complete transesterification. The analyses of GC-MS and GC-FID likewise verified the chemical structural feasibility of *Pistacia chinensis* biodiesel as a substitute for fossil diesel. Likewise, several major fuel properties of the biodiesel have been measured, and these met the required standards for fuel use.

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