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Characterization and transesterification of Iranian bitter almond oil for biodiesel production

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ABSTRACT

In the present work the production of biodiesel using bitter almond oil (BAO) in a potassium hydroxide catalyzed transesterification reaction was investigated. The BAO was obtained from resources available in Iran and its physical and chemical properties including iodine value, acid value, density, kinematic viscosity, fatty acid composition and mean molecular weight were specified. The low acid value of BAO (0.24 mg KOH/g) indicated that the pretreatment of raw oil with acid was not required. The fatty acid content analysis confirmed that the contribution of unsaturated fatty acids in the BAO is high (84.7 wt.%). Effect of different parameters including methanol to oil molar ratio (3–11 mol/mol), potassium hydroxide concentration (0.1–1.7% w/w) and reaction temperature (30–70 °C) on the production of biodiesel were investigated. The results indicated that these parameters were important factors affecting the transesterification reaction. The fuel properties of biodiesel including iodine value, acid value, density, kinematic viscosity, saponification value, cetane number, flash point, cloud point, pour point and distillation characteristics were measured. The properties were compared with those of petroleum diesel, EN 14214 and ASTM 6751 biodiesel standards and an acceptable agreement was observed.

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1. Introduction

Fossil fuels as energy resources are of vital importance to any country. There are limited resources of these fuels and therefore investigation of alternative renewable fuels is a necessary and increasing need. One of the renewable energy sources is biodiesel, which consists of mono alkyl esters of long chain fatty acids [1]. In addition of being renewable, biodiesel has other advantages such as having fuel properties close to petroleum diesel [2], reducing regulated air pollutants emissions [3,4], reducing greenhouse emissions [5], together with being non-toxic, environmentally safe and biodegradable [6].

There are several ways to produce biodiesel. Among them, transesterification is the most common one [7,8]. The transesterification is a reaction of oil with an alcohol in the presence of suitable catalysts such as acid or alkaline catalysts [9,10]. Methanol is the most common alcohol because of its properties and its low cost [11]. Alkaline catalysts, such as potassium hydroxide are more effective in the transesterification process [12].

The high cost of biodiesel is the major barrier for its commercialization [13,14]. Researchers have found out that the main part (85–95%) of the total cost of biodiesel production is the cost of the raw material [15]. Therefore, production of biodiesel from low-cost

feedstock, e.g., non-edible oils is necessary. Furthermore, most of the non-edible oil crops grow well on waste lands, can tolerate drought and dry conditions and do not need intensive care [16]. Many studies have been conducted on biodiesel production from non-edible oils, such as *Jatropha curcas* oil [17–19], karanja seed oil [17,20], okra seed oil [21], rubber seed oil [22,23], castor oil [24], and mahua oil [25,26]. Generally, the effect of several operating variables on the transesterification reaction has been investigated and the fuel properties of produced biodiesels from non-edible oils have been determined, so that these oils were found to be appropriate viable sources for producing biodiesel. However, there are other non-edible oils that have not been investigated as a raw material for the production of biodiesel, so far. Bitter almond oil (BAO) is one of these oils.

The almond tree (*Prunus dulcis*), which belongs to the family of Rosaceae, is a native to the Mediterranean climate region of the Middle East. But it was spread into other regions of the world by humans, so that it is cultivated in Europe, Asia and Northern Africa, and more recently also in California. Italy, Spain, Morocco, France, Greece, and Iran are the main countries that produce almond. The almond tree grows to a height between 3 and 8 m, with a trunk of up to 30 cm in diameter. Many varieties of almonds can be broadly divided into two types, namely sweet almond (with white flowers) and bitter almond (with pink flowers) [27].

The yield of the bitter almond oil, which is obtained from seeds of almond tree through a pressing process, is about 40–45%. Bitter

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almond oil has a yellowish color and an acrid-burning taste. Bitter almond fruit contains amygdalin and an enzyme that decomposes it into glucose, benzaldehyde and hydrocyanic acid making the fruit non-edible [28].

In this work, the fatty acid composition and other physical and chemical properties of Iranian bitter almond oil were determined. The major purpose of the study was to investigate biodiesel production from the bitter almond oil by KOH-catalyzed transesterification. The effect of three important parameters including methanol to oil molar ratio, catalyst concentration and reaction temperature on the product yield, biodiesel purity and biodiesel yield were studied.

2. Materials and methods

2.1. Materials

Crude bitter almond oil was obtained from Kimia Pack Co., a local company in Tehran, Iran. Various methyl esters used as standards in the chromatographic determination were purchased from Wako Pure Chemicals, Japan. All other chemicals and solvents that were supplied from Merck, Germany and used during biodiesel synthesis, purification and analysis were: methanol of 99.5% purity, potassium hydroxide pellets of 85% purity, manganese sulfate powder of 98% purity, *n*-hexane of 99% purity and hydrochloric acid of 37% purity.

2.2. Experimental procedure

The transesterification reaction was carried out in a 500 ml glass made batch reactor, equipped with a thermometer, condenser and magnetic stirring systems. The reactor containing of 50 g bitter almond oil was placed in a water bath heated by a hot plate. The BAO was agitated and heated up to an appropriate temperature. A defined amount of catalyst previously dissolved in methanol was added to the BAO, the reaction started and continued for 60 min.

After the reaction ended, heating and stirring were stopped and the reaction mixture was transferred into a decanter and left for 1 h to separate two distinct phases, *i.e.* ester phase (biodiesel) and glycerol phase. It took approximately 10 min to conduct this phase separation, but the biodiesel layer was translucent. After 1 h, the ester phase became transparent and the separation was completed. Further reaction may happen during the settling time, but the process is slow because of a low temperature, lack of stirring and presence of low amounts of catalyst and methanol. However, it is said that even longer settling time is favorable for the separation [8,29]. The glycerol phase formed in the lower layer, was decanted. The ester phase (biodiesel) was washed with 35 ml of hot distilled water, then with 35 ml of hydrochloric acid 0.5% to neutralize the remaining catalyst and to decompose the soaps formed during the transesterification reaction. Finally, it was washed with 35 ml of hot distilled water three times. The successive rinses removed impurities such as the residual catalyst, methanol, glycerol and soaps. The ester phase was then dried using manganese sulfate and filtered under vacuum conditions to eliminate manganese sulfate crystals. The final product was weighed for determination of product yield and then analyzed by GC to calculate biodiesel purity.

2.3. Analytical methods

Iodine values of the BAO and the biodiesel were determined according to AOCs Official Methods Cd 1c-85 and Cd 1-25 [30], respectively. Acid values of the BAO and the biodiesel were

obtained according to AOCs Official Methods Ca 5a-40 and Cd 3d-63 [30], respectively. The BAO and the biodiesel densities were measured using a DMA 38 density meter from Anton Paar, Germany. Kinematic viscosities of the BAO and the biodiesel were determined according to the ASTM D445 method [31]. Saponification value of the biodiesel was measured according to AOCs Official Method Cd 3-25 [30]. Other biodiesel properties including cetane number, flash point, cloud point, pour point and distillation characteristics were obtained according to the ASTM D976, ASTM D93, ASTM D2500, ASTM D97 and ASTM D86 methods [31], respectively.

Fatty acid composition of the BAO was obtained by gas chromatography according to AOCs Official Method Ce 2-66 [30]. The mean molecular weight of the BAO was calculated according to the fatty acid analysis and using following equation

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

where, MW_{oil} stands for molecular weight of the BAO, MW_i and x_i stand for molecular weight and mass fraction of the *i*th fatty acid, respectively.

The fatty acid methyl ester composition of the purified biodiesel samples was determined by gas chromatography using a GC PERI-CHROM PR2100 equipped with a capillary column (50 m × 0.201 mm × 0.50 μm) with HP-PONA stationary phase and flame ionization detector. Helium with a flow rate of 0.6 ml/min was used as carrier gas. The GC column temperature adjusted to 120 °C for 5 min, increased up to 225 °C with a rate of 4 °C/min and held at 225 °C for 20 min. The injector and detector temperatures were both set at 250 °C. For analysis of the biodiesel, 0.3 g of the purified biodiesel sample was dissolved in 5 ml of *n*-hexane and 1 μl of the resulting solution was injected into the GC.

In this research, the results were reported in terms of product yield, biodiesel purity and biodiesel yield which were calculated using Eqs. (2) and (3), respectively.

$$\text{Product yield} = \frac{m_{product}}{m_{BAO}} \times 100 \quad (2)$$

$$\text{Biodiesel yield} = \frac{m_{product} \times \text{purity of biodiesel}}{m_{BAO}} \quad (3)$$

where, $m_{product}$ stands for the mass of final product after purification process, m_{BAO} is the mass of the BAO used for each experiment (50 g) and the purity of biodiesel was obtained by GC analysis.

3. Results and discussion

3.1. Properties of bitter almond oil

The physical and chemical properties of the applied bitter almond oil including the iodine value, acid value, density, kinematic viscosity, fatty acid composition and mean molecular weight is presented in and compared with other oils (Table 1). The iodine value of BAO was 81.60 mg I₂/g, which is lower than that for peanut seed oil. This value was not available for other oils. The acid value of the BAO was 0.24 mg KOH/g, which is lower than those reported for mahua oil and rapeseed oil but close to acid values of peanut seed, cottonseed, soybean and canola oils. No pretreatment with acid is required for oils with the acid value lower than 2 mg KOH/g [26]. The density of the BAO was relatively close to those measured for soybean, rapeseed and cottonseed oils. The kinematic viscosity of the BAO was quite alike to canola oil and relatively lower than rapeseed oil density but higher than mahua and peanut seed oils. However, it is far lower than the viscosities of soybean and cottonseed oils.

Table 1
Properties of the BAO in comparison with the other oils.

Property	BAO [this study]	Peanut seed oil [8,11]	Canola oil [13]	Cottonseed oil [20]	Rapeseed oil [20]	Soybean oil [20]	Mahua oil [21]
Iodine value (mg I ₂ /g)	81.60	123.22	NA ^a	NA	NA	NA	NA
Acid value (mg KOH/g)	0.24	0.20	<0.5	0.11	1.14	0.2	38.0
Density at 15 °C (g/cm ³)	0.921	0.888	NA	912	914	920	960
Viscosity at 40 °C (mm ² /s)	34.98	22.72	35.3	50	39.5	65	24.58
Fatty acid composition (wt.%)							
Myristic acid (C14:0)	0.1	NA	0.1	NA	NA	NA	NA
Palmitic acid (C16:0)	10.3	11.38	5.5	11.67	3.49	11.75	16.0–28.2
Stearic acid (C18:0)	3.9	2.39	2.2	0.89	0.85	3.15	20.0–25.1
Oleic acid (C18:1)	33.9	48.28	55	13.27	64.4	23.26	41.0–51.0
Linoleic acid (C18:2)	46.0	31.95	24	57.51	22.3	55.53	8.9–13.7
Linolenic acid (C18:3)	4.8	0.93	8.8	0	8.23	6.31	NA
Arachidic acid (C20:0)	0.2	1.32	0.7	NA	NA	NA	0.0–3.3
Molecular weight, M _w (g/mol)	866	NA	882	NA	NA	NA	NA

^a Not available.

The fatty acid composition analysis showed that the contribution of unsaturated fatty acids in the bitter almond oil was 84.7 wt.% containing of 33.9% oleic acid, 46.0% linoleic acid and 4.8% linolenic acid. Saturated fatty acids consisting of 14.5% of total fatty acids mainly composed of palmitic acid and stearic acid. The rest of the fatty acid composition (0.8%) which includes other components of small amounts was not determined. The high content of unsaturated fatty acids could cause a reduced cloud point, cetane number and stability of produced biodiesel due to increased content of unsaturated fatty acid methyl esters [22].

3.2. Effect of methanol to oil molar ratio on biodiesel production

Molar ratio of methanol to oil is one of the most important variables that affect the transesterification reaction [32–34]. The effect of this parameter on the product yield, biodiesel purity and biodiesel yield is shown in Fig. 1. Experiments were carried out using methanol to oil molar ratio in the range of 3:1–11:1, while other variables including KOH concentration and reaction temperature were set at 0.9% w/w and 50 °C, respectively. The stoichiometric molar ratio of alcohol to triglyceride is 3:1; but the transesterification is a reversible reaction and an extra amount of alcohol is commonly used in order to shift the equilibrium in benefit of alkyl ester generation. It was observed that with increasing the methanol to oil molar ratio from 3:1 to 7:1, the product yield increased from 91.8 to 97.2 while the biodiesel purity and yield increased from 74.6 to 92.7 and from 68.4 to 90.9, respectively. Therefore, the effect of methanol to oil molar ratio on the biodiesel purity and yield is more than its effect on the product yield. Further increase in the ratio had a little effect on the product yield. However, with increasing the molar ratio from 7:1 to 11:1, the biodiesel purity and yield

decreased. This reduction might be related to the quality of phase separation prior to the analysis. Using more excess amount of methanol causes difficulties in the separation of glycerol from biodiesel phase by increasing the solubility of glycerol. Remained glycerol in the biodiesel phase, causes the equilibrium reaction to proceed backward and the recombination of esters and glycerol into mono-glycerides so that the biodiesel purity and consequently the biodiesel yield decreases [34–36].

3.3. Effect of KOH concentration

The catalyst concentration is another important variable that affects transesterification reaction [32–34] along with hydrolysis and saponification reactions [37]. The effect of potassium hydroxide on the product yield, biodiesel purity and biodiesel yield is shown in Fig. 2. Experiments were performed using KOH concentration in the range of 0.1–1.7% w/w (based on the weight of the BAO), while the methanol to oil molar ratio and reaction temperature were set at 7 mol/mol and 50 °C, respectively.

As shown in the figure, when a KOH concentration of 0.1% w/w was used, the biodiesel yield was 47.1% w/w. Insufficient amounts of potassium hydroxide resulted in incomplete conversion of oil into the esters so that the biodiesel purity was 47.9%. With increasing KOH concentration from 0.1 to 0.9% w/w, the product yield approximately remained constant but the biodiesel purity and yield increased. The biodiesel yield reached a maximum value of 90.8% w/w when the KOH concentration of 0.9% w/w was used. Further increase in potassium hydroxide concentration, resulted in decreased product yield, biodiesel purity and biodiesel yield. However, the effect of the excessive amount of catalyst on the biodiesel purity was less than that on the product yield and the biodiesel yield. When excess catalyst is used the amount of soaps

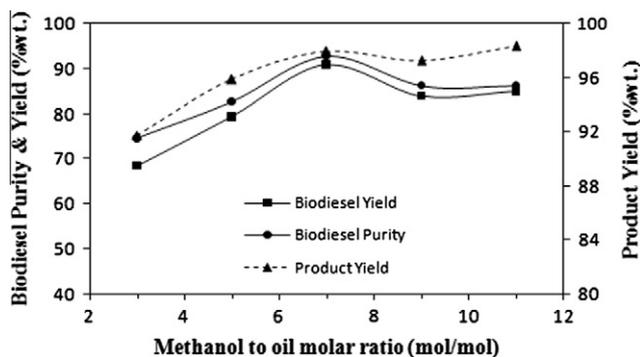


Fig. 1. Effect of methanol to oil molar ratio on the product yield and biodiesel yield (KOH concentration of 0.9% w/w and reaction temperature of 50 °C).

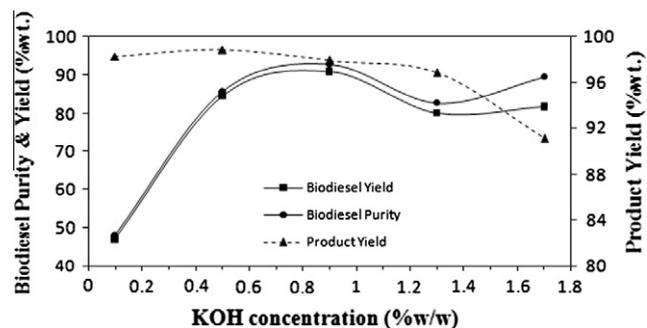


Fig. 2. Effect of KOH concentration on the product yield and biodiesel yield (methanol to oil molar ratio of 7 mol/mol and reaction temperature of 50 °C).

formed by saponification reaction increases. The soaps increase the methyl ester solubility in glycerol and produce emulsification of the ester and glycerol that makes difficulties in the separation of the esters [36,38,39]. Therefore, ester losses increase and consequently the product and biodiesel yield decreases. In this study, due to the low free fatty acid content (0.24 mg KOH/g), formed soap was not considerable in general. However, as mentioned above, utilization of excess catalyst increased the amount of formed soap.

3.4. Effect of reaction temperature

The effect of the reaction temperature on the product yield, biodiesel purity and biodiesel yield is represented in Fig. 3. Experiments were accomplished utilizing a reaction temperature in the range of 30–70 °C, while the methanol to oil molar ratio and potassium hydroxide concentration were set at 7 mol/mol and 0.9% w/w, respectively. When the reaction temperature increased from 30 to 50 °C, there was a little change in the product yield. However, with increasing the temperature from 50 to 70 °C the product yield decreased. Biodiesel purity and yield increased when the reaction temperature was raised from 30 to 50 °C. The maximum value of biodiesel yield was obtained at 50 °C. Further increase in temperature (from 50 to 70 °C) caused a decrease in both the biodiesel purity and yield. However, the reduction of the biodiesel yield was more significant than the biodiesel purity. At higher temperatures, the side saponification reaction of triglycerides by the alkali catalyst is much faster than the transesterification. In addition, despite using a condensation system in the experiments, a large number of bubbles produced by the vaporization of methanol, hindered the alcoholysis reaction and methanol losses increased at higher temperatures. Therefore, the product yield, biodiesel purity and consequently biodiesel yield decreased [17,37,39].

3.5. Biodiesel properties

Physical and chemical properties of the produced biodiesel including iodine value, acid value, density, kinematic viscosity, saponification value, cetane number, flash point, cloud point, pour point and distillation characteristics are presented in Table 2. The properties were compared to the petroleum diesel, EN 14214 and ASTM 6751 biodiesel standards. The comparison of these properties with petroleum diesel showed that density and kinematic viscosity values of the biodiesel is relatively close to petroleum diesel. Flash point of the biodiesel was 170 °C, which is a high value that has the benefit of higher safety than the petroleum diesel for transport purposes. However, the biodiesel's cloud and pour points were considerably higher than petro-diesel. Therefore, the biodiesel is less suitable to be used in winter time. As shown in the table,

Table 2
Physical and chemical properties of BAO biodiesel.

Property	BAO biodiesel [this study]	Petroleum diesel [35]	EN 14214	ASTM 6751-02
Iodine value (mg I ₂ /g)	117.29		<120	
Acid value (mg KOH/g)	0.44		<0.50	<0.80
Density at 15 °C (g/cm ³)	0.887	0.847	0.86–0.90	0.87–0.90
Viscosity at 40 °C (mm ² /s)	4.68	2.39	3.5–5.0	1.9–6.0
Saponification value	185.35			
Cetane number	44.6	46	>51	>47
Flash point (°C)	173	85	>120	>130
Cloud point (°C)	10	–19		–3 to 12
Pour point (°C)	–3	–23		–15 to 10
Distillation (°C)				
10%	320			
20%	322			
30%	324			
40%	325			
50%	326	258		
60%	327			

the properties of produced biodiesel, except cetane number, conformed to EN 14214 and ASTM 6751 standards for biodiesel. However, cetane number was lower than the standard limits. This value for cetane number is reasonable due to fatty acid composition of the BAO, which is mostly consisted of 10.3 wt.% palmitic acid, 33.9 wt.% oleic acid and 46.0 wt.% linoleic acid. The cetane numbers of methyl palmitate, methyl oleate and methyl linoleate are 85.9, 59.3 and 38.2, respectively [40]. As mentioned previously in Section 3.1, a high content of unsaturated fatty acids could reduce the cetane number.

4. Conclusion

The characterization of the Iranian bitter almond oil confirmed that its chemical and physical properties are comparable to that of some other vegetable oils. The acid value of the BAO was 0.24 mg KOH/g, which indicated that the pretreatment of raw oil with acid was not required. The fatty acid composition of the BAO confirmed that the contribution of unsaturated fatty acids in the bitter almond oil is high (84.7 wt.%). The bitter almond oil was transesterified using methanol and potassium hydroxide catalyst for production of biodiesel. Effects of methanol to oil molar ratio, potassium hydroxide concentration and reaction temperature on the product yield, biodiesel purity and biodiesel yield were studied. The results indicated that these parameters were important factors affecting the transesterification reaction. A high product yield (97.9 wt.%) was achieved with a high biodiesel yield (90.8 wt.%) at methanol to oil molar ratio of 7.0 mol/mol, potassium hydroxide concentration of 0.9 w/w and temperature of 50 °C. The measured properties of the biodiesel, except cetane number, were conformed to EN 14214 and ASTM 6751 standards. The reduced cetane number of the produced biodiesel can be solved by blending it with petro-diesel.

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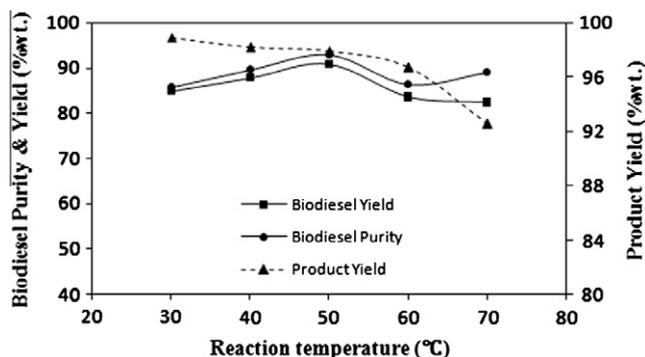


Fig. 3. Effect of reaction temperature on the product yield and biodiesel yield (methanol to oil molar ratio of 7 mol/mol and KOH concentration of 0.9% w/w).

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