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Production and Evaluation of Ethylic Biodiesel from Mixed Non-Edible Castor and Bitter Almond Oils

E. T.B. Al-TIKRITY
Tikrit University

A.B. FADHIL
University of Mosul

K. K. IBRAHEEM
Tikrit University

Abstract: Selection of low price and non-edible feedstocks for biodiesel production is a considerable target to diminish the production cost of biodiesel. Accordingly, the current work is to inspect the production of fatty acid ethyl esters (ethylic biodiesel) from mixed non-edible oils particularly; castor oil seeds (CSO) and bitter almond oil (BAO). Various blends of the CSO: BAO (10:90 -50:50 % w/w) were prepared and evaluated in order to choose the optimal blend whose properties similar to those reported for conventional raw oils. The results indicate that the equivalent blend (50:50 % w/w) possess the optimal properties. Therefore, it was chosen as a raw feedstock for producing ethylic biodiesel through the standard base-catalyzed transesterification process by using KOH as a catalyst with ethanol. Experimental parameters affecting transesterification process, such as the type and the concentration of the alkali catalyst, ethanol to mixed oils molar ratio, the reaction temperature, and the reaction time were optimized. Biodiesel with maximum yield (98.88 wt. % with purity of 97.66 % w/w) was obtained by implementing 0.50 % KOH w/w of oil, 5:1 ethanol to oil molar ratio, 35 °C reaction temperature and 30 minutes time. The 1HNMR spectroscopy affirmed the transformation of the oils blend to ethylic biodiesel. The obtained ethylic biodiesel had density and kinematic viscosity values much lower than those of ethylic biodiesel produced from CSO individually. Furthermore, the properties of the resulting biodiesel were within the acceptable limits prescribed by ASTM D 6751. It was concluded that using such blend oils could be an easy approach to improve the properties of produced biodiesel from castor oil, and to reduce production cost.

Keywords: Mixed non-edible oils, Ethylic biodiesel, Transesterification, Fuel properties evaluation, Analysis of biodiesel

Introduction

Biodiesel (BD) is manufactured on industrial scale through alcoholysis reaction of various edible oils with methanol in the presence of alkaline catalyst [1]. One of the significant key in such production process is the type and the cost of the oil feedstock. In fact, 95% of commercial BD is currently produced from edible oils, which is reported to be 70-80% of the total manufacturing cost [2, 3]. European and United States of America are producing BD from rapeseed, soybean and sunflower oils feedstock, while coconut and palm tree oils are predominantly used in Asia [4].

Regardless of that, employing edible oils in making BD has been of major awareness due to the food versus fuel dispute [5, 6]

Utilization of non-edible oils, when compared with edible oils, has received high attention in developing countries because the indigence for edible oils as food, and they are far too expensive to be utilized as fuel [5].

- Selection and peer-review under responsibility of the Organizing Committee of the Conference

Consequently, exploring new innovations toward renewable and cheaper raw materials to produced low cost BD has become a new strategy for BD production feasibility.

Many inexpensive non-edible oils sources like Jatropha curcas [7,26], waste animal fats, waste cooking oil [8], and waste fish oil [9], castor oil [10], and oil from rubber seeds [11] are preferentially utilized for that purpose. This implementation arises from the reality that many of such oils are not suitable for human consumption, and could be used as successful alternative feedstock for production process.

However, castor trees (Ricinuscommunis), among many non-edible oil producer plant, are an important industrial non-food crop, it can be cultivated in wild land without special care, and can tolerate sever climate conditions [9, 10,12,13],. Its productivity for non-edible oil from their seeds (the castor seed oil, CSO) is cheap and high, as much as 51.0%. It was reported that the global production of CSO reached around 1.8 million tons per year, and that India, China and Brazil are the leading production countries [12, 13].

Unfortunately, the CSO consists of about 87 - 95% of Ricinoleic acid. This acid percent impress undesired properties, as far as BD production is concerned, like high polarity, high viscosity, and high density. It was mentioned that the kinematic viscosity of CSO amounts of 226 mm2/s at 40°C, while those of most vegetable oils are in the range 27–54 mm2/s at the same temperature. Therefore, BD derived from CSO has much higher viscosity than those of conventional biodiesels. Accordingly, many researchers suggest mixing of BD obtained from CSO with lower viscosities diesel (biodiesels or petro diesel fuel) to be within the acceptable fuel engine properties. Whereas, the other proposed mixing of CSO with another oils or animal fats, in order to select the optimum oils blend that satisfy the suitable specifications to produce BD [14-17].

On the same concept, Bitter Almond seeds, which obtained from almond tree (Prunusdulcis var. amara), gives high percent of non-edible oil about 42% and has been applied to produce BD [18-20]. Nevertheless, little literature published about synthesis of BD from bitter almond oil (BAO) via base-catalyzed transesterification, but no works were reported for production of BD from mixing of BAO with other non-edible oils [18-20].

The present research work suggests production of ethylic BD from a mixture of CSO and BAO, through optimized ethanolysis transesterification reaction. Different blends of CSO and BAO were prepared and evaluated to select the optimal blend properties. The quality of the produce BD was analyzed by FTIR, and 1H NMR spectroscopy, and the fuel properties of the obtained ethylic BD were determined following the ASTM standards.

Experimental

Materials and Reagents

Castor seeds and bitter almond seeds were collected from Nineveh and Dohuk Governorates (north of Iraq), respectively. All chemicals were of analytical reagent grade, and used as received without any further purification. Absolute ethanol (99.9 %), methanol, potassium and sodium hydroxides (KOH, NaOH, and pellets), sodium methoxide (CH3ONa), sodium ethoxide (CH3CH2ONa), iodine, acetone, sodium sulphate (Na2SO4), and diethyl ether were purchased from BDH (UK). Chloroform, n-hexane, hydrochloric acid, and cyclohexane were purchased from Fluka (Germany).

Seed Preparation and Oil Extraction

Seeds of castor and bitter almond were cleaned and oven-dried at 50°C for 24h. After grinding of the dried seeds using an electrical grinder, the oil was extracted by n-hexane in a Soxhlet extractor for 10h. The extracted oils were separated from the solvent by distillation using a rotary evaporator. The obtained oils was dried over anhydrous sodium sulfate to eliminate the residual moisture, filtered and kept in a sealed container at 5°C for further assessment and use. The yield of the oil was calculated for each type seeds, on dry bases [22].

Analysis and Characterization of the Extracted Oils

The Fatty acids composition of both CSO and BAO were determined using gas chromatography (GC, Perkin Elmer, Auto system GLX, Shelton, U.S.A.) equipped with a Supelco SP™-2380 (30 m x 0.25 mm i.d., 0.25 µm film thickness) column equipped with flame ionization detector (FID)[22]. The average molecular weight (g/mol) of the oils was determined by a weighted average method utilizing the fatty acid profile. The iodine value of CSO and BAO was determined based on Hanus method. Properties of CSO and BAO were determined following ASTM standard methods.

Preparation of the Blended Oils

Different weight percent blends of CSO and BAO were prepared through mixing of CSO with (10, 20, 30, 40 and 50 % w/w of BAO). The prepared blends were thoroughly mixed by mechanical stirrer for 30 minutes to obtain homogeneous mixture. The physical and chemical properties of the blends oils were examined in order to select the acquired blend.

Transesterification of the Mixed CSO and BAO with Ethanol

The blend oils (100g) was placed in a three neck round-bottomed flask (500 mL), fitted with mechanical stirrer, condenser, thermostat, and sampling outlet, followed by addition(separately) a known amount of freshly prepared ethylic solutions of the catalyst salts, (NaOH, KOH, CH3ONa, and CH3CH2ONa). The mixture was, then, refluxed with stirring at 700 rpm for a specific reaction time. The reaction products were transferred into a separating funnel and left overnight to obtain two distinct phases. The lower layer (glycerin) was separated and discarded, while the upper layer (ethyl esters) was distilled under vacuum using a rotary evaporator to recover the excess alcohol, followed by washing with distilled water to remove the other impurities. Finally, the ethyl esters were dried over anhydrous sodium sulfate to dispose of any traces of leftover water. The ethyl esters yield was calculated as follow [11, 23]

$$\text{Biodiesel Yield (\% w/w)} = \frac{\text{Total weight of the purified ethyl ester X Ester content (\%)}}{\text{Total weight of the used blend oils}} \text{ X 100}$$

Analysis and Properties Evaluation of the Biodiesel

The 1HNMR spectra were used for analysis of the produced ethyl esters by using a Varian (Palo Alto, CA, USA) VNMR spectrometer operating at 300 MHz. Samples were diluted in 500 µL of deuterated chloroform (CDCl3). Properties of the produced ethyl ester were evaluated as per the ASTM standard procedures. The properties included density (ASTM D 5002), kinematic viscosity (ASTM D 445), flash point (D93), saponification value (ASTM D5555-95), the refractive index (D1747 – 09), the acid value (ASTM D664), the cloud, and pour points (ASTM D 2500). The Iodine value was measured according to Hanus method. The soap content was determined in accordance with AOCS Cc 17-95. The method proposed by Pisarello et al. [24] was used for determination of the total glycerin in the produced ethyl esters. Each property was measured in triplicate and the results were presented as the mean ± standard deviation (SD).

Results and Discussion

Properties and Analysis of the Oils

Solvent extraction of oils from castor and bitter almond seeds has given, on dry bases, 51.0 and 42.0%w/w, respectively, which are relatively high comparing to other non-edible vegetable seeds. Therefore, CSO or BAO can be exploited as potential feedstock for BD production. As mentioned earlier, the high viscosity and density of CSO may disclose unsuitable BD for engine fuel. On that subject, different blends of CSO and BAO (10/90 - 50/50 % w/w BAO/CSO) were prepared and evaluated so as to select the optimal blend properties which correspond to BD production. The results, in Table1, indicate that the density, viscosity, refractive index, and flash point of the CSO have markedly been reduced with increasing BAO content in the blends. This improvement in oil properties arise from the merits of BAO comparing to that of CSO.

Table1. Properties of CSO, BAO and their blends

Property	*CSO*	*BAO*	*Blend* 90+10	*Blend* 80+20	*Blend* 70+30	*Blend* 60+40	*Blend* 50+50
Density @ 15.6 g/mL	0.9658	0.9100	0.9397	0.9364	0.9275	0.9211	0.9193
Kinematic Viscosity@ 40°Cmm^2/s	197.43	24.12	131.42	101.21	86.66	75.33	54.77
Flash Point $^{\circ}$C	185	180	175	170	165	145	130
Acid Value mg KOH / g oil	0.10	0.46	2.61	2.21	1.85	1.66	1.47
Refractive index@20°C	1.4780	1.4674	1.4711	1.4688	1.4665	1.4651	1.4643
Pour Point °C	-<15	-7	-15.0	-14.0	-11.0	-11.0	-10.0
Sapon. Value mg KOH / g oil	188.47	175.23	170.20	174.80	180.06	217.21	219.80

Properties of the blend in comparison to other non-edible oils

Property	CSO+BAO (50/50%)	CJCO [a]	SMSO [b]
Density @ 15.6 g/mL	0.9193	0.9180	0.9141
Kinematic Viscosity@ 20°Cmm^2/s	54.77	35.40	42
Flash Point $^{\circ}$C	130	186.0	230
Acid Value mg KOH / g oil	1.47	11.0	13.6
Refractive index @ 20°C	1.4643	-	1.4700
Pour Point °C	-10.0	-	-8
Sapon. Value mg KOH / g oil	219.80	-	195.0

[a] Ref.[26]; [b] Ref.[24]

The reduction in the features of CSO, after blending with BAO, may attributed to a possible interaction between hydroxyl groups of the ricinloeic acid and the free fatty acids of BAO, or to the dilution effect. This was assured through the 1H NMR spectrum of the CSO and the blend which is depicted in Fig.1a and Fig.1b, respectively. The signals at (4.13 and 4.30 ppm) which are related to glyceride protons were separated in the CSO [25]. After blending with BAO, the signals have coalesced and become multiple as well as their integration increases. The signal at (2.32 ppm) which belongs to CH2-COO was found in the CSO and disappeared after blending with BAO [25]. The integration of the signal at (3.4 ppm) which correspond to the proton of the hydroxyl group of the ricinoliec acid was reduced from 0.58 to 0.34 ppm after blending of CSO with BAO [25]. Hence, the equivalent blend (50/50 %) was chosen for production of BD via base-catalyzed transesterification with ethanol.

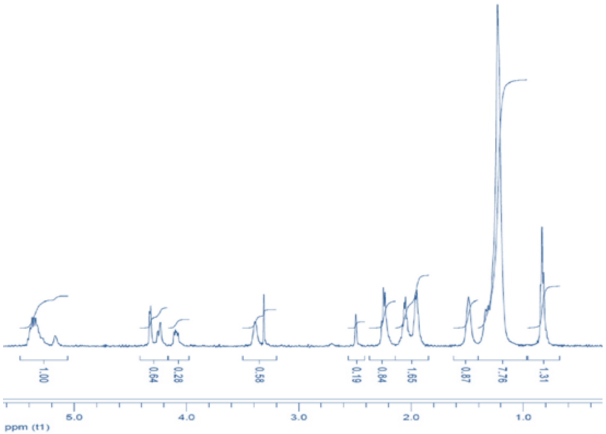

Fig.(1a). ^{1}HNMR of castor seed oil

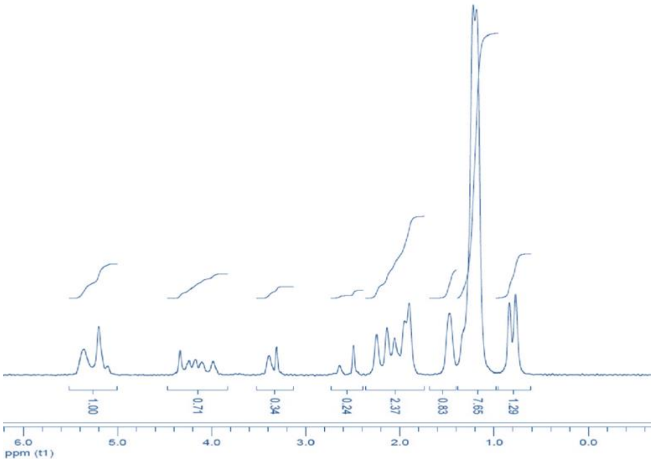

Fig.(1b). ^{1}HNMR of mixed castor oil and bitter almond oil

Biodiesel Production from the Blend of CSO and BAO

The properties of the 50:50 equivalent blends of CSO and BAO were found to be close to the feature of those conventional oils, Table 1; so, it was selected for BD synthesis. The lower acid value of the blend (0.03 mg KOH/g), in contrast to the other non-edible oils [20, 26, 27], makes it suitable feedstock for production of BD via direct optimized protocol base- catalyzed reaction. Therefore, parameters affecting the transesterification process, such as the catalyst type and concentration, ethanol to oil molar ratio, reaction temperature, reaction time, and rate of stirring were thoroughly investigated.

Influence of Catalyst type and Concentration

Effect of concentrations and catalyst type on transesterification reaction of the blend was investigated by trying various alkali catalysts (KOH, NaOH, CH3ONa and CH3CH2ONa) at different concentrations (0.25-1.50 % w/w of oil) as depicted in Fig.2. The other parameters were kept constant at 6:1ethanol: oil molar ratio, 70°C reaction temperature, 700 rpm agitation rate, and 60 minutes reaction time. It was noticed that the lower concentrations of the alkali catalysts (0.25 wt.%) led to an incomplete reaction and resulted in a low yield of BD. The yield increased and amount to maximum at 0.50 wt.% using any of the tested catalysts. Fig.2 also, exhibit that among the tested catalysts, KOH has given the highest yield of BD (92.12 wt.%) which was in accordance with many literatures published elsewhere [20-22, 28,29].

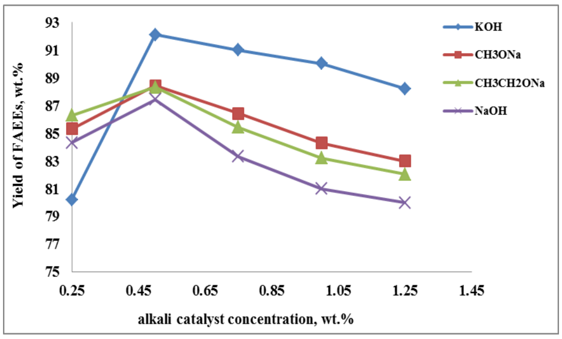

Figure 2. Effect of catalyst type and concentration on ethyl esters yield

The variation in the yield, among different alkali catalysts, could be attributed to their variation in the strength of alkalinity. Nevertheless, increasing the catalysts concentrations more than 0.50 wt.% decreased the ethyl ester yield due to the saponification reaction [28,29]. Therefore, KOH at a concentration of 0.50% w/w of oil was chosen as a type of catalyst and the optimal concentration, respectively.

Influence of Ethanol to Oils Blend Molar Ratio

Transesterification reaction is greatly influenced by alcohol to oil molar ratio. On this account, number of experiments were carried out using different ethanol to oil blends molar ratio in the range of 4:1–10:1, Fig.3, keeping the other factors fixed at 0.50 wt.% KOH, 70°C, 60 minutes, and 700 rpm. The results indicated that the maximum yield of ethyl ester (94.44 wt.%) was obtained at 5:1 ethanol to blend oil molar ratio. This finding was very low relative to other reported studies of ethanolysis of some non-edible oils. [22, 27, 30-35].

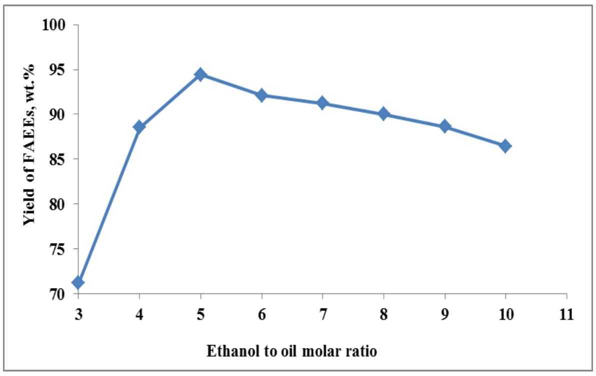

Figure 3. Effect of ethanol to oil molar ratio on ethyl esters yield

It can, also, behold that any further increase in alcohol to oil molar ratio reduced the yield of the ethyl esters due to the solubility of glycerin in the alcohol, pushes the equilibrium reaction to proceed backward to form mono-glycerides [33, 27]. Therefore, 5:1 was established as the optimal mixed alcohols to oil molar ratio.

Influence of Reaction Temperature

The reaction temperature plays a vital role on the transesterification reaction rate, yield conversion and eventually to the BD production cost. It is necessary to optimize the reaction temperature to obtain maximum yield with less energy consumption. It was reported that selecting the optimal reaction temperature for transesterification depends on the chemical composition of the raw oil feedstock, and the type of utilized alcohol [36]. Hence, different experiments were accomplished applying reaction temperature in the range of 25-75°C,whilst the other variables were fixed at 0.50% KOH w/w of oil, 5:1 ethyl alcohol to oils blend molar ratio,60 minutes reaction time and 700 rpm rate of stirring. Fig.4, display the transesterification of the oil mixture with ethanol at 25°C (room temperature) resulted in good yield of ethyl esters (90.02 wt.%), but further increase in the reaction temperature increased the ethyl ester yield. It fact, 35°C was the optimal reaction temperature because it gave highest yield of ethyl ester (96.55 wt. %). This finding is much less value than BD production from the other mixed non-edible oils and very important, as far as the cost of the production is concerned. [22, 31-33, 37, 38].

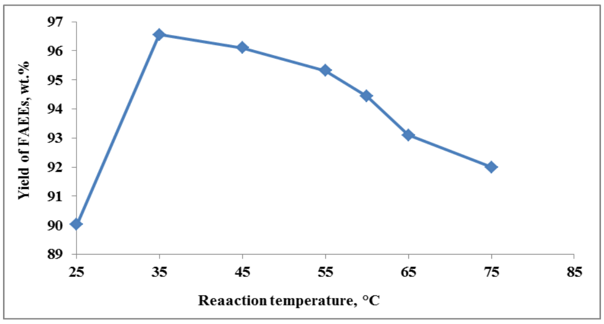

Figure 4. Effect of reaction temperature on ethyl esters yield

It was also noticed that heating more than 35°C favor the side reaction, i.e part of the esters may turned into soap (saponification reaction) leading to decreases the BD yield [35,36]. Hence, 35°C was chosen as an optimal reaction temperature.

Influence of Reaction Time

The influence of reaction duration on the ethyl ester yield was investigated by conducting the transesterification reaction at various durations (15, 30, 45, 60, 75 and 90 minutes), keeping the other parameters, for each experiment, at their optimal values Fig.5. The results exhibit that the highest ethyl ester yield (98.88 wt.%) was obtained at a time of 30 minutes which is much lower than those reported for the production of ethyl ester from various oil feedstocks in literature. [22, 31, 32, 35, 37, 39].

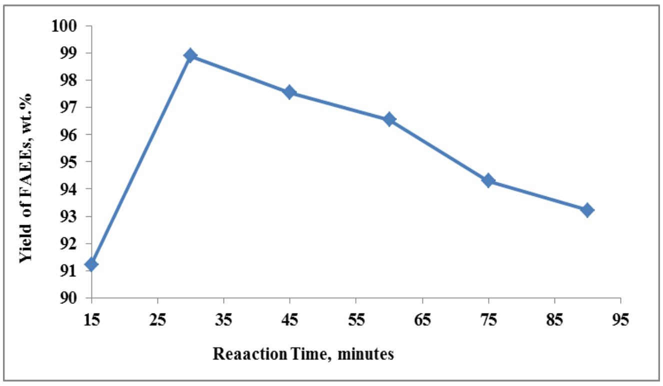

Figure 5. Effect of reaction time on ethyl esters yield

However, durations higher than 30 minutes resulted in lower ethyl ester yield due to the hydrolysis of some of the formed ethyl esters [31]. As a result, the optimal reaction duration was set as 30 minutes.

Effect of Stirring Rate

Transesterification process is induced by the stirring rate due to the mass transfer between the oil and the catalyst solution, and the reaction rate become faster as the mixing increases [27, 37]. Therefore, stirring speed was varied from 300 rpm to 900 rpm at intervals of 100 rpm, while the other parameters were kept at their optimal values. It was observed that phase change had occurred at different rate of stirring. At lower rate of stirring (300 rpm) a little phase changes formation was observed and the yield of the ethyl esters was at lowest level (88.56 % w/w) Fig.6. Whereas, the increase in the rate of stirring the phase change formation was quite clear and the BD yield increased to the maximum (98.88 %) at stirring rate of 700 rpm.

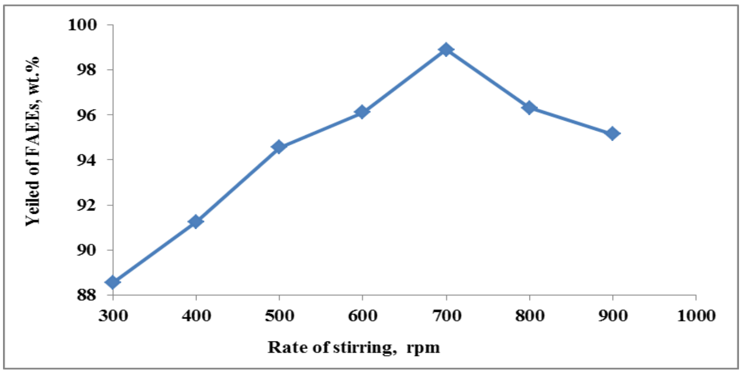

Figure 6. Effect of rate of stirring on ethyl esters yield

These results were attributed to the facts that increases the rate of stirring promotes homogeneity of the reactants, thence, raises the contact between the two immiscible phases (the blend oils and catalyst) to produce higher BD. Nevertheless, at high rate of stirring more than700 rpm the yield was decreased because of the hydrolysis of some BD causing soap formation. These findings were in good agreement with other authors [32, 40].

Fuel Properties Evaluation

The properties of the ethylic BD obtained from the said blend are summarized in Table 2 together with the standards specified in the ASTM D 6751 and the published values of several biodiesels in literature. For comparison, ethyl esters from CSO and BAO were prepared, separately, by applying the optimal conditions as recorded previously. Following the ASTM D 6751 standards, the obtained BD has a fatty acid ethyl esters content of (97.66±1. % w/w) which was equivalent to the limit specified in that standards (96.50 % w/w). It can be seen from Table 2 that the density and viscosity of the CSO ethyl ester were much higher than those documented for other ethylic biodiesels in literature [33, 38]. Accordingly, BD from the CSO is not suitable as a successful alternative to petro diesel unless these characteristics values are reduced to the acceptable limits. Blending of the CSO and BAO impose an ethylic BD with density and viscosity values much lower than those of the ethylic BD from CSO only, and close to those published for other ethylic biodiesels in literature [32,35]. This improvement in the produced BD is related to the effect of BAO increment which its ethylic BD has much lower density and viscosity than that obtained from CSO. The cloud and pour points of the ethyl ester produced from the mixture are suitable to be used in the cold weather conditions. Moreover, other merits like flash point, refractive index, the acid value, the total content of free glycerin and soaps in the obtained ethylic BD were much lower than those established in the literature [32, 35] and the standard of the ASTM D6751 Table 2. However, the variation in properties values of the produced ethyl ester in comparison to other non-edible oils could be attributed to many factors such as; different chemical composition of the oil feedstock, the applied conditions for the transesterification and the ester content in the biodiesels. The aforementioned achievements, in general, were persuadable feedback for completion of the ethanolysis of the blend. Thus, it may recommend that blending of CSO with BAO is a manner to reduce CSO properties to become highly suitable feedstock raw materials for BD production.

Table 2. Properties of ethylic biodiesel from mixed CSO and BAO in comparison to other ethylic esters

Property	*Ethylic BD from CSO*	Ethylic BD from BAO	*Ethylic BD from the oils blend*	*Ethylic BD from Used frying oil* [c]	*Ethylic BD from Sunflower oil* [d]	*ASTM D 6751*
Yield (wt.%)	94.55	86.55	98.88±1.50	-	-	-
Ester content (w/w %)	92.32	81.51	97.66±1. 0	93.20	98.90	96.50
Density @15.6 °C g/mL	0.9202	0.8677	0.8702± 0.0012	0.8883	0.8810	0.9000
Kinematic viscosity @ 40 °C mm^2/s	11.22	4.07	5.18± 0.11	5.81	4.71	3.0 – 6.0
Flash point °C	145	125	±1.0130	188	163	130.0
Acid value mg KOH/g	0.08	0.11	0.03± 0.01	0.46	0.24	0.50
Refractive index @ 20°C	1.4622	1.4531	1.4511± 0.0001	-	-	-
Cloud point °C	-3.0	5.0	-1± 0.50	9.0	-2.0	-
Pour point °C	<-15	-9.0	< -10	-1.0	-10.0	-
Total glycerin content (wt.%)	0.08	0.10	0.04± 0.01	0.25	0.15	0.24
Free glycerin content (wt. %)	0.0039	0.0031	0.0033± 0.001	0.14	0.0	0.02
Soaps (ppm)	0.62	0.62	0.34± 0.02	-	-	5.0

[c] Ref.[35]; [d] Ref.[37]

Analysis of the Mixed Castor and Bitter Almond Ethyl Esters

1H NMR spectroscopy was utilized by many researchers for monitoring transesterification reaction as well as to determine the conversion of oils to their fatty acid alkyl esters [23, 38]. Hence, the obtained ethyl ester was analyzed using this technique to assure the conversion of the mixed CSO and BAO into ethylic BD. The spectrum in Fig.7 shows the disappearance of the characteristic peaks of mono-, di- and triacylglycerol hydrogens at 4.25 ppm, and the appearance of another characteristic peak at 4.11 ppm, belongs to the ethyl group of the corresponding produced ester.

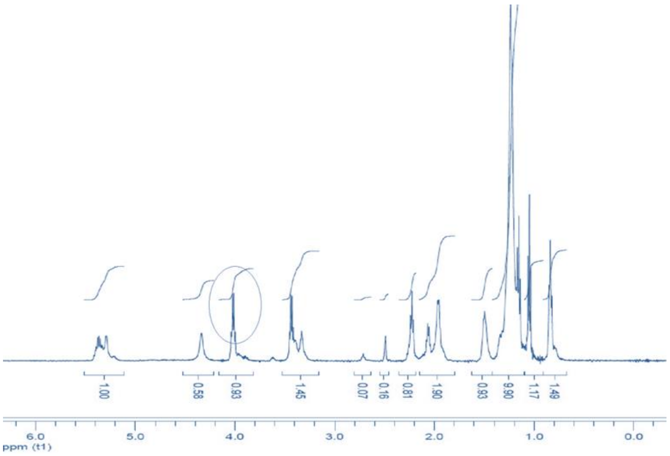

Figure 7. The ^{1}H NMR spectra of biodiesel obtained from mixed castor oil and bitter almond oil

The triplet peak at 2.26 ppm in the spectrum was attributed to α-CH2 protons, whereas the observed peak at 0.85 ppm refers to the terminal methyl protons. The strong signal at 1.26 ppm in the spectrum belongs to the methylene protons of carbon chain, while that observed at 1.58 ppm attributes to the β-carbonyl methylene protons. Finally, peak related to oliffinic hydrogen was appeared at 5.3 ppm [23, 37, 38]. This finding demonstrate the merits of transesterification process to convert low cost non-edible oils mixture into ethyl ester BD, and may considered a successful manner to alter the fuel properties of the parent feedstock into a more valuable fuel.

Conclusion

Fatty acid ethyl ester (ethylic biodiesel) was successfully prepared from mixed non-edible oils, CSO and BAO, with ethanol in the presence of an alkali catalyst. The results revealed that 50/50 v/v CSO: BAO was the acquired mixture for the preparation. Base-catalyzed transesterification of the mixture with ethanol was optimized, and the highest yield (98.88 wt.% and a purity of 97.66 % w/w) by implement 0.60 % KOH w/w, 5:1 ethanol to oils blend molar ratio, 35 °C reaction temperature, 30 minutes of reaction, and 700 rpm rate of stirring. The 1H NMR spectroscopy confirmed the conversion of the blend to their fatty acid ethyl esters. The measured properties of the biodiesel were in accordance to ASTM D 6751 standards indicating suitability of the obtained fuel for diesel engines. It was concluded that exploited the mixed oils system can reduces the required reaction temperature to give maximum conversion, which in turns reduces the cost of the biodiesel production.

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Author Information

E. T.B. Al-Tikrity
Department of chemistry, College of Science, Tikrit University, Salahaldeen, Iraq
Contact E-mail: emaad_bakir@yahoo.com

A.B. Fadhil
Chemistry Researches Laboratory, Department of Chemistry, College of Science, University of Mosul, Mosul, Iraq

K. K. Ibraheem
Department of chemistry, College of Science,
Tikrit University, Salahaldeen, Iraq