EFFECT OF STIRRING SPEED AND REACTION TIME ON SUNFLOWER BIODIESEL PRODUCTION

H. M. S. Shoukry* and M. A. MOSALLAM**

ABSTRACT

Biodiesel was produced from sunflower oil using methyl alcohol at a ratio of 22.5% (v/v) to the oil and sodium hydroxide as a catalyst at a ratio 5.5 g per 1 liter oil. The production of biodiesel was carried out under four stirring speeds: 250, 500, 750 and 1000 rpm and for each stirring speed, five reaction times were used: 10, 20, 30, 40 and 50 min. the research concluded to that: 1- the optimum stirring speed for the production of biodiesel from sunflower oil is 750 rpm. 2- the optimum reaction times are 40 and 50 min. 3- the highest biodiesel production is 98% and was obtained at 50 min time of reaction for each of the two speeds of stirring: 500 and 750 rpm and at 10 min time of reaction under the speed 10 rpm. 5- the use of sodium hydroxide as a catalyst for the production of biodiesel from sunflower oil at a concentration of 3.5 g per liter of oil results in an excess soap in the produced biodiesel and a loss of biodiesel in the washing water.

INTRODUCTION

According to the environmental worries and the consumption of non-renewable natural energy resources, developing alternative resources of energy as a substitute of traditional fossil fuels has been risen. Biodiesel is among those alternatives and it is defined as an alternative fuel for diesel engines produced by chemically reacting a vegetable oil or animal fat with an alcohol, **El Diwani *et al*. 2009**. In preparation for a future petroleum fuel crisis, the United States recently made a commitment to triple bioenergy usage within 10 years. The European Union also has made a similar proposal, **Barbara *et al*. 2008.** The use of biodiesel has drawn attention in the last decade as it is a renewable, biodegradable, and nontoxic fuel, **Hossain *et al*. 2010**. The objective of this work is to study the effect of stirring speed and reaction time on the production of biodiesel from sunflower oil.

*Prof., Animal Production Dept., Fac. of Agr., Al-Azhar Univ., Cairo.
** Assoc. Prof., Agr. Eng. Dept., Fac. of Agr., Al-Azhar Univ., Cairo.

REVIEW OF LITERATURE

Biodiesel is made using the process of transesterification. Transesterification is the process of using an alcohol (e.g. methanol or ethanol) in the presence of a catalyst, such as sodium or potassium hydroxide, to chemically break the molecule of the raw renewable oil into methyl or ethyl esters of the renewable oil with glycerol as a by – product **(Transesterification, 2000).** Oil in sunflower seeds is high (40 – 45%) and sunflower plant was successfully cultivated in Egypt in the new reclamined areas and in soils of salinity which does not exceed 3000 ppm and sunflower can be cultivated under different weather conditions **(Kenanaonline, 2006).** **He *et al*., 2005,** studied six variables which affect the production of biodiesel from canola oil using a continuous-flow reactive distillation system. These variables included the feed methanol to triglycerides molar ratio, reaction time, catalyst concentration and reaction time. **Singh *et al*. 2006,** studied the production of biodiesel from canola oil at different catalyst concentrations, reaction temperatures, and methanol-to-oil molar ratios. They concluded that: the operating conditions for maximizing biodiesel yield were potassium methoxide as catalyst at 0.2 mol/mol (1.59%wt), reaction temperature of 50.C, and methanol-to-oil molar ratio of 4.5:1. Experimental verification gave 95.8% biodiesel yield from canola oil. **Dobromir *et al* 2007,** produced biodiesel from used sunflower oil, rapeseed oil and soybean oil. The reaction was carried out under the following conditions: 60 ^{o}C reaction temperature, 1 h reaction time, 1 – 10 % (v/v) sodium methoxide. The yield of biodiesel from each of the three used oils was: 85.5, 86.5 and 86 % respectively. The Total Acid Number “TAN” of the produced biodiesel ranged from 0.018 to 0.024 mgKOH/g. **Barbara *et al*. 2008,** used a mixtures of commercial oils for the production of biodiesel. The reactions were carried out under stirring speeds of 600 rpm, time of reaction of 30 min, temperature of 65 ^{o}C, catalyst (lithium hydroxide) of ratio 1.5 % (w/w) and methanol to oil molar ratio of 3: 1. The mixture of the oils: corn, cotton, sunflower, canola and coconut gave 97.89 % (w/w) biodiesel yield. **El Diwani *et al*. 2009**, produced biodiesel from jatropha oil under bench and pilot scales. They used methyl alcohol at ratio 10 % by volume to the oil and sodium hydroxide as a catalyst at ratio 1% by weight to the oil. The reaction was carried out under temperature of 65 ^oC for one hour. Percent of the produced biodiesel was 98 %. “TAN” for the biodiesl produced under bench and pilot scales were: 0.26 and 0.30 mgKOH/g respectively and the Calorific Values “C.V.” were 40.59 and 39.44 Mj/kg. **Hossain *et al*. 2010**, produced biodiesel from waste canola oil. Conditions of the reaction were: 1: 1 molar ratio of methanol to oil, temperature of reaction of 55 ^oC, stirring speed of 250 rpm, time of reaction of 2 h and 0.5 % sodium hydroxide. The yield of biodiesel was 49.5 % and TAN was 0.1 mgKOH/g. **Math *et al*. 2010**, produced biodiesel from a mixture of 75 % restaurant waste oil and 25% pig fat oil. They got 80% (v/v) yield of biodiesel. They mentioned that the optimum conditions for the reaction are: methanol percent of 40% (v/v), temperature of 65 ^oC, time of reaction of 90 min, sodium hydroxide of 0.3% (w/w) and 1.5 cm^3 sulfuric acid. **Hamed *et al*. 2006**, produced biodiesel from Salmon oil. They used sulfuric acid to reduce the acid value of the salmon oil from 12.04 to 3 mg KOH/g. the reaction carried out under a temperature of 52^oC and 600 rpm stirring speed. Biodiesel yield was 99 % (w/w) under methanol molar ratio of 9.2 and sodium hydroxide of 0.5% (w/w). To compare the effect of stirring speed of 1800 rpm with respect to ultrasonic irradiation in the production of biodiesel from triolein, Hoang ***et al. (2007)*** obtained 93% yield under 25^oC temperature, molar ratio of ethanol/triolein of 6/1, base catalyst concenteration of 1 wt% for both NaOH and KOH and reaction time of of less than 20 min.

MATERIALS AND METHODS

A commercial sunflower oil (Slite trade) was used in these experiments. Methanol alcohol (99.8 concentration) was used for the production of biodiesel. The alcohol was used at a ratio, by volume, 22.5 % to the oil. Sodium hydroxide was used as a catalyst for the production of biodiesel at a ratio of 5.5 g per 1 liter oil (in preliminary experiments, a concentration of 3.5 g of sodium hydroxide per each liter of oil and 27.5 % by volume methanol alcohol to the oil, were tested for the production of biodiesel. The produced biodiesl has an excess quantity of soap and needed to be washed for more than 10 times and a noticeable quantity of the biodiesel drained out with the washing water – the produced biodiesel according to the previously mentioned concentrations, for the preliminary experiments, was analyzed for the Total Acid Number "TAN" and the Calorific Value "C.V."). An overhead mechanical stirrer (Wheaton instrument trade) of power of 120 W was used for the reaction. The stirrer has a speed range from 0 to 15000 rpm. The stirrer was adjusted to the required reaction stirring speed using a tachometer: Cole Parmer trade, Model 8203 – 10. biodiesel was produced under four stirring speeds: 250, 500, 750 and 1000 rpm. For each speed, five times of reaction were investigated. These times are: 10, 20, 30, 40 and 50 rpm (the choosed values of stirring speeds and reaction times are based on Preliminary experiments).

Steps of production of biodiesel were as follows: sodium hydroxide was dissolved in the methanol alcohol to form the sodiummethoxide using the stirrer. For each time under each tested speed, two replicates were tested. For each replicate, 200 cm^3 oil was added to 45 cm^3 sodiummethoxide and the mixture was stirred to perform the transesterification process. Then the sample was transferred into a 250 cm^3 measuring cylinder and left for separation for 3 h or more. The sample separates into two layers: the lower one which is glycerin and the upper one which is methyl ester of the sun flower oil (which includes soap, alcohol, impurities, etc). using a pipette, half of the produced methyl ester was transferred into a 250 cm^3 separating funnel. 25 cm^3 of distilled water was added into the funnel, then the funnel was shaked to mix the water and the ester. The sample was left for the separation of the washing water. After separation of the washing water, the water was drained out of the funnel and the washing process was repeated (5 – 7 times) till the washing water became clear. The washed biodiesel was then transferred into a 250 cm^3 measuring cylinder to determine its volume. The determined biodiesl volume was calculated as a percent, by volume with respect to the half of the volume of the oil used in the sample (100 cm^3). The produced biodiesel was analyzed for the Total Acid Number "TAN" and the Calorific Value "C.V." at the Egyptian Petroleum Research Institute. Also the produced biodiesel was tested for cleanliness according to **Daniel, 2010** : 3 cm^3 of the produced washed biodiesel were dissolved into 27 cm^3 methanol alcohol to note if there exists undissolved liquids or suspended impurities.

RESULTS AND DISCUSSION

Table (1) shows the produced biodiesel at each of the five tested times of reaction (10, 20, 30, 40 and 50 min) for each of the four tested stirring speeds (250, 500, 750 and 1000 rpm). Fig. (1) shows the relation between biodiesel production and time of reaction at each of the four tested stirring speeds.

Table (1): The produced biodiesel at each of the five tested times of reaction for each of the four tested stirring speeds.

Time (min)	**Stirring speed (rpm)**			
	250	**500**	**750**	**1000**
	Biodiesel production (% by volume)			
10	**50**	**70**	**86**	**98**
20	**85**	**90**	**96**	**92**
30	**93**	**95**	**96**	**92**
40	**95**	**95**	**96**	**92**
50	**96**	**98**	**98**	**90**

Table (1) and Fig. (1) show that:

1- For stirring speeds 250, 500 and 750 rpm, the production of biodiesel increases as the time of reaction increase.

2- For these three speeds, the production of biodiesel becomes nearer and at its utmost range (95 – 98%) at the times 40 and 50 min.

3- For the speed 100 rpm, the biodiesel production decreases as the time increase.

4- The highest biodiesel production recorded is 98% and occurs at 50 min for each of the speeds 500 and 750 rpm and at 10 min for the speed 1000 rpm.

5- The lowest biodiesel production (50%) is obtained at 10 min for the speed 250 rpm.

Fig. (2) shows the relation between biodiesel production and stirring speed at each of the five tested times of reaction. Table (1) and Fig. (2) show that:

1- For times of reaction: 20, 30, 40 and 50 min, the production of biodiesel increases as the speed of stirring increase till the speed 750 rpm Where the production begins to decrease. Speed 750 rpm is considered to be the optimum speed for producing biodiesel from sunflower oil.

2- At 10 min time of reaction, the production of biodiesel increases considerably as the speed of reaction increase.

Table (2) shows analysis of a sample of the produced biodiesel for the "TAN" and the "C.V." according to the Egyptian Petroleum Research Institute:

Table (2): Analysis of the produced biodiesel:

Calorific Value (cal/gm) ASTM D 240	Total acid number (mgKOH/g) ASTM D 664
9855.79	0.272

When dissolving 3 cm^3 of the produced biodiesel in 27 cm^3 of methanol alcohl (test of cleanliness according to **Daniel, 2010)**, the biodiesel dissolved at once completely in the alcohol and no impurities were left.

Table (3) shows the biodiesel produced in a preliminary experiment (at a concentration of 3.5 g of sodium hydroxide per each liter of oil and 27.5 % by volume methanol alcohol to the oil) under the four tested speeds at the corresponding given times of reaction:

Table (3): The produced biodiesel using sodium hydroxide at 3.5 g per liter oil.

Time (min)	**Stirring speed (rpm)**			
	250	**500**	**750**	**1000**
	Biodiesel production (% by volume)			
10	**-----**	**-----**	**-----**	**68**
20	**-----**	**42**	**64**	**-----**
30	**26**	**-----**	**-----**	**28**
40	**-----**	**-----**	**-----**	**-----**
50	**-----**	**60**	**-----**	**-----**

This produced biodiesel was analysed for the "TAN" and the "C.V." at the Egyptian Petroleum Research Institute. The analysis was according to table (4):

Table (4): Analysis of the produced biodiesel using sodium hydroxide at 3.5 g per liter oil.

Calorific Value (cal/gm) ASTM D 240	Total acid number (mgKOH/g) ASTM D 664
8883.73	0.113

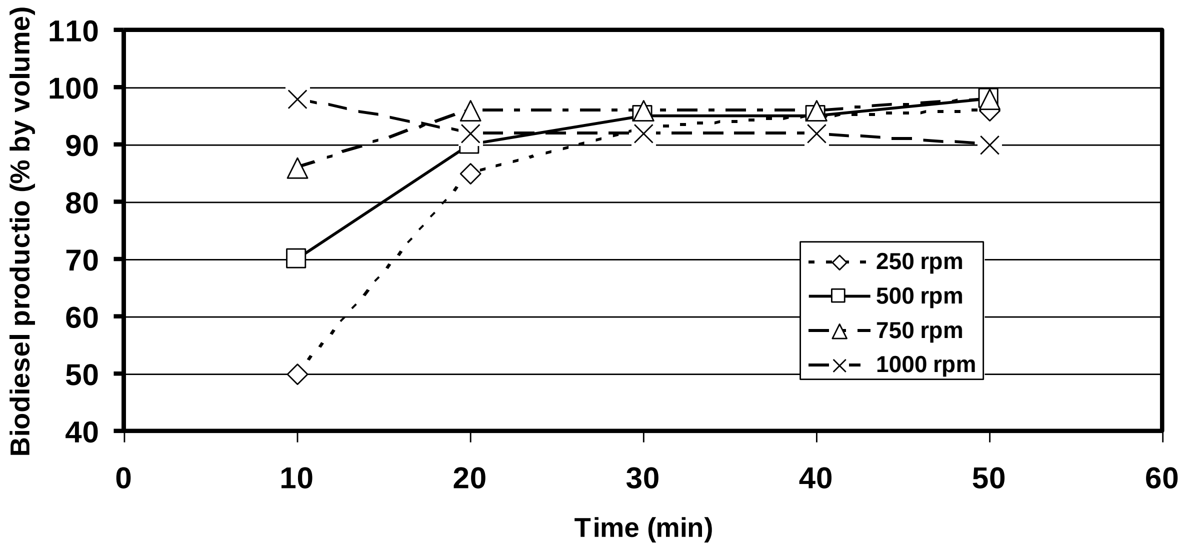

Fig. (1): The relationship between biodiesel production (% by volume) and time (min) at each of the four tested stirring speeds.

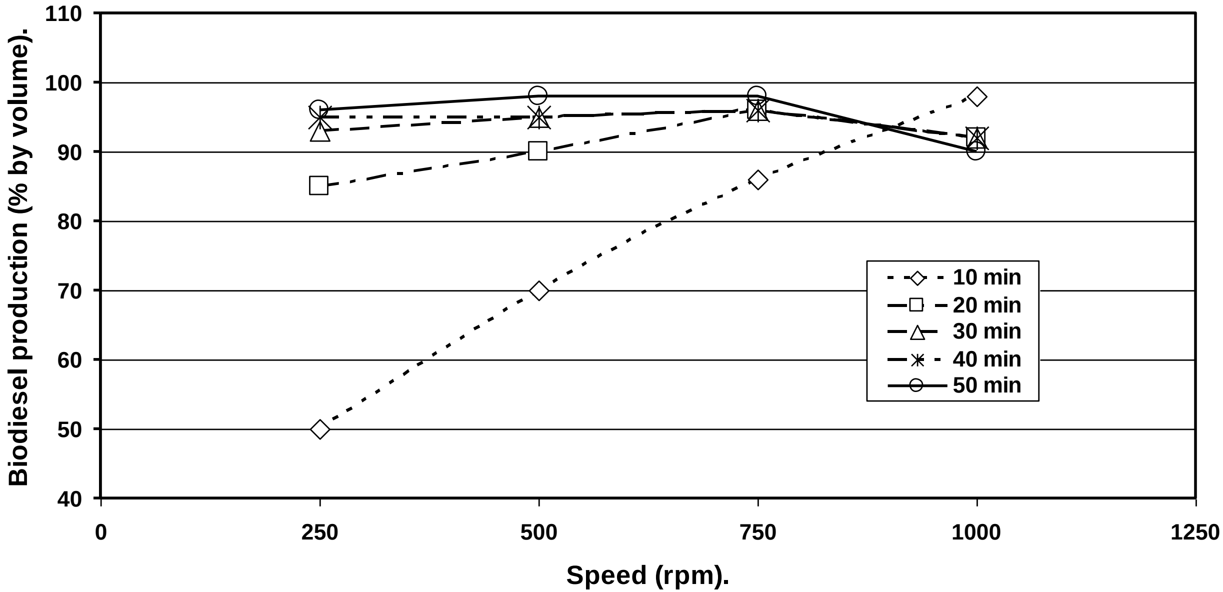

Fig. (2): The Relationship between between biodiesel poduction (% by volume) and stirring speed (rpm) at the tested five times.

SUMMARY

Commercial sunflower oil (Slite trade) was used for the production of biodiesel. For the formation of the oil ester (the biodiesel), the methyl alcohol was used at a ratio of 22.5% (v/v) to the oil and sodium hydroxide was used as a catalyst for the production of the methyl ester of the oil at a ratio 5.5 g per 1 liter oil.

A mechanical stirrer was used for the production of the methyl ester of the oil. The production of biodiesel was carried out under four stirring speeds: 250, 500, 750 and 1000 rpm and for each stirring speed, five reaction times were used: 10, 20, 30, 40 and 50 min.

The produced biodiesel was analysed for the total acid number and the calorific value at the Egyptian Petroleum Research Institute.

Research results showed the following:

1- The optimum stirring speed for the production of biodiesel from sunflower oil is 750 rpm.

2- The optimum reaction times are 40 and 50 min.

3- The highest biodiesel production is 98% and was obtained at 50 min time of reaction for each of the two speeds of stirring: 500 and 750 rpm and at 10 min time of reaction under the speed 10 rpm.

4- The Calorific Value of the produced biodiesel is 9855.79 and the total acid number is 0.272 mgKOH/g.

5- The use of sodium hydroxide as a catalyst for the production of biodiesel from sunflower oil at a concentration of 3.5 g per liter of oil results in an excess soap in the produced biodiesel and a loss of biodiesel in the washing water.

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<u>الملخص العربى</u>

أثر سرعة التقليب و زمن التفاعل على إنتاج الديزل الحيوى من زيت عباد الشمس

هشام محمد صالح شكرى* و محمود أحمد مسلم**

استخدم زيت عباد شمس تجارى (ماركة سلايت) لإنتاج الديزل الحيوى. لتكوين استر الزيت (الديزل الحيوى). استخدم كحول الميثيل بنسبة ٢٢,٥ % (حجم/حجم) للزيت و استخدم هيدروكسيد الصوديوم كعامل مساعد لإنتاج ميثيل استر الزيت بنسبة ٥,٥ جم لكل ١ لتر زيت.

استخدم قلاب ميكانيكى لإنتاج ميثيل استر زيت عباد الشمس. تم انتاج الديزل الحيوى تحت أربع سرعات تقليب: ٢٥٠، ٥٠٠، ٧٥٠ و ١٠٠٠ ل/د و لكل سرعة تقليب استخدمت خمسة أزمنة تفاعل: ١٠، ٢٠، ٣٠، ٤٠ و ٥٠ د.

تم تحليل الديزل الحيوى الناتج لرقم الأحماض الكلى و للقيمة الحرارية بمعهد بحوث البترول المصرى.

بينت نتائج البحث الآتى:

١- أفضل سرعة تقليب لإنتاج الديزل الحيوى من زيت عباد الشمس هى ٧٥٠ ل/د.

٢- أفضل أزمنة التفاعل هى ٤٠ و ٥٠ د.

٣- أعلى انتاج للديزل الحيوى هو ٩٨ % وتم الحصول عليه عند زمن ٥٠ د لكل من سرعتى التقليب ٥٠٠ و ٧٥٠ ل/د وعند زمن ١٠ د للسرعة ١٠٠٠ ل/د.

٤- القيمة الحرارية للديزل الحيوى الناتج هى ٩٨٥٥,٧٩ كالورى/جم و عدد الأحماض الكلى ٠,٢٧٢ ملج ص أ يد /ج.

٥- استخدام هيدروكسيد الصوديوم كعامل مساعد لإنتاج الديزل الحيوى من زيت عباد الشمس بتركيز ٣,٥ ج لكل لتر زيت يترتب عليه كثرة الصابون بالديزل الناتج و فقد جزء غير قليل منه فى ماء الغسيل.

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* أستاذ – قسم الإنتاج الحيوانى – كلية الزراعة – جامعة الأزهر – القاهرة.

** أستاذ مساعد – قسم الهندسة الزراعية – كلية الزراعة – جامعة الأزهر – القاهرة.