

A METHOD FOR PRODUCING BIODIESEL

FIELD OF THE INVENTION

5 This invention relates to a method for producing biodiesel. More particularly, this invention relates to a method for producing biodiesel using air bubbling and adding an alcohol/catalyst mix at a controlled rate to an oil source to increase the production rate of the biodiesel.

10 BACKGROUND

Generally, a transesterification reaction of vegetable oil and animal fat is carried out in the presence of an alcohol and a catalyst. The reaction can be a base-catalyzed reaction or an acid-catalyzed reaction. In most cases, a base-catalyzed reaction is
15 employed as it is the most economical process for treating straight vegetable oils, requiring only a relatively low temperature and pressure for converting the vegetable oil and animal fat into biodiesel.

Conventional methods of biodiesel production utilize either a batch reactor or an ultra-
20 and high shear in-line reactor to produce biodiesel continuously, semi-continuously, and/or in batch-mode. A Batch reactor uses blade stirrers to mix the reactants while an ultra- or high shear in-line reactor uses static high shear mixing to mix the reactants. Batch process reactions are usually easy to control but are often cost ineffective due to ineffective mixing methods and long reaction times. Continuous
25 processes which use high shear reactors are able to reduce reaction time but they are usually very energy intensive, that in turn, increases the production cost.

Another known method used in the production of biodiesel is the ultrasonic reactor method. In this method, the ultrasonic waves cause intense mixing so that the
30 reaction can proceed at a much faster rate. Ultrasound transfers energy into fluid and creates violent vibrations, which form cavitation bubbles. As the bubbles burst, a sudden contraction of the fluid occurs, and the reactants are mixed in the area of the bubbles. This method can be a good choice for small producers who may only need one or two ultrasound probes per reactor vessel. However, using ultrasound in large-

scale processing may be challenging because many ultrasound probes would be needed to reach every area of the reactant mixture.

It is, therefore, desirable to provide a method for producing biodiesel in a cost effective manner and at a faster rate.

SUMMARY OF INVENTION

The above and other problems are solved and an advance in the art is made by a method for producing biodiesel in accordance with this invention. A method in accordance with this invention has the advantage of increasing production rate and reducing production costs by allowing an oil source and an alcohol to have localized reaction in the presence of a catalyst through the use of air bubbling and controlled rate of adding an alcohol/catalyst mix to the oil source. A second advantage of the method in accordance with this invention is that the method reduces the time required to separate the biodiesel and glycerol formed from the method by using air bubbling to aid in the phase separation process.

In accordance with an embodiment of this invention, the method comprises feeding an oil source into a reaction tank. The oil source is then heated, and air bubbles are introduced into the reaction tank at a predetermined rate. An alcohol/catalyst mix is added into the reaction tank at a controlled rate for transesterification reaction to take place. The main products produced by the method are biodiesel and glycerol. As the reaction progresses, the heavier glycerol settles at the bottom of the reaction tank forming a lower phase comprised of glycerol and an upper phase of biodiesel. The biodiesel is then recovered from the reaction tank and the glycerol is withdrawn from the reaction tank.

In some embodiments of this invention, the air bubbles are introduced into the reaction tank through an aerator. The air bubbles form a stream of individual air bubbles within the reaction mixture and the stream of individual air bubbles circulates inside the reaction tank.

In some other embodiments of this invention, the alcohol/catalyst mix is introduced into the reaction tank through an inlet provided at an upper end of the reaction tank.

The alcohol/catalyst mix is introduced at a point proximate the stream of individual air bubbles.

In further embodiments of this invention, the air bubbles are introduced into the reaction tank to separate the biodiesel and the glycerol.

In some other embodiments of this invention, the method further comprises adjusting the placement of the aerator such that the aerator is disposed above the lower phase comprised of glycerol.

BRIEF DESCRIPTION OF THE DRAWINGS

The above and other advantages and features of this invention are described in the following detailed description and are shown in the following drawings:

Figure 1 illustrating a planar view of an example of an aerator that can be used in the method in accordance with the invention;

Figure 2 illustrating a planar front view of an example of a reaction tank that can be used in the method in accordance with the invention;

Figure 3 illustrating a side view of the reaction tank as shown in Figure 2;

Figure 4 illustrating a back view of the reaction tank as shown in Figure 2; and

Figure 5 illustrating a top view of the reaction tank as shown in Figure 2.

DESCRIPTION OF THE INVENTION

The present invention relates to a method for producing alkyl ester, otherwise known as biodiesel, by reacting vegetable oils and/or animal fats consisting essentially of triglycerides with an alcohol in the presence of a catalyst.

The vegetable oils can be one selected from a group comprising straight vegetable oil and waste vegetable oil. The straight vegetable oil may include, but not limited to, palm oil and soybean oil.

- 5 The alcohol can be one selected from a group comprising methanol, ethanol, propanol, and other monoalkyl alcohols. Preferably, methanol is used.

The catalyst can be acidic or basic. Suitable catalysts include alkali metal hydroxides such as sodium or potassium hydroxide, and acidic compositions such as sulfuric acid.

- 10 Preferably, sodium hydroxide is used.

In one embodiment of the invention, the vegetable oil or animal fat is first fed into a reaction tank. The oil or fat is then heated gradually from room temperature to a temperature of between 55°C to 65°C and this temperature is maintained throughout
15 the reaction. As the oil or fat is heated from room temperature, air bubbles are introduced into the reaction tank at a predetermined rate.

The air bubbles are introduced into the reaction tank by an aerator. Air flows from a pipe into the aerator inside the tank. The aerator is preferably located at bottom end
20 of the reaction tank. Other suitable locations may also be considered. The aerator has several small openings through which air flows into the reaction mixture in the reaction tank as air bubbles. The aerator may be of various shapes and sizes, such as a disc, tube and plate. The exact size and shape of the aerator being a design that may depend on various factors including the shapes and sizes of the reaction tank
25 used. In one exemplary embodiment of the invention, the aerator is a tube having plurality of openings that are arranged in a circular manner as depicted in Figure 1. One skilled in the art will recognize that other suitable configurations may be used without departing from invention. The exact configuration of the aerator is left as a design choice for those skilled in the art.

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The size of the openings of the aerator is dependent on the size of air bubbles to be introduced into the reaction mixture. Preferably, the size of the openings is in the range of 1.0 mm or less.

The number of openings provided on the aerator may vary according to the volume of the oil or fat added to the reaction tank and the size of the reaction tank. In all cases, the number of openings selected must be sufficient to cause an effective localized reaction to occur in the reaction tank.

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When the temperature of the oil or fat in the reaction tank reaches a temperature of between 55°C to 65°C, an alcohol/catalyst mix is added to the reaction tank at a controlled rate to form a reaction mixture for transesterification reaction to take place. The alcohol/catalyst mix is preferably prepared by dissolving the catalyst in alcohol

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using a standard agitator or mixer to form a corresponding alkoxide.

In one exemplary embodiment of the invention, the alcohol/catalyst mix is added to the oil or fat at a rate between 0.16 to 0.27 L/min when a volume between 2.20 and 3.00 L of the alcohol/catalyst mix is to be added to the oil or fat. The amount of

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alcohol/catalyst mix to be added to the oil or fat is dependent on the amount of the oil or fat used in the method. The amount of the alcohol/catalyst mix added per unit time is dependent on the total amount of the alcohol/catalyst mix required to carry out the transesterification reaction and the average time consumed for the transesterification reaction to take place.

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In accordance with the method of the invention, the rate at which the air bubbles are introduced into the reaction tank must be sufficient to allow effective mixing and effective transesterification reaction to take place. If the rate of introducing the air bubbles into the reaction tank is slow, there may not be enough air bubbles in the

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reaction mixture to allow effective mixing and reaction to take place. If the air bubbles are introduced into the reaction tank at a very fast rate, the air bubbles may become joined, forming elongated air bubbles within the reaction mixture. This will adversely affect the mixing and reaction conditions.

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The rate at which the air bubbles are introduced into the reaction tank must be adequate to allow the air bubbles to form a stream of individual air bubbles in the reaction mixture and to circulate inside the reaction tank. The individual air bubble allows oil or fat molecules to adhere to each individual air bubble. This encourages localized reactions of the oil or fat molecules with the alcohol in the presence of the

catalyst. The localized reactions allow transesterification to take place more effectively and this in turns reduces the production time.

When adding the alcohol/catalyst mix at a controlled rate to the reaction tank, the alcohol/catalyst mix is preferably added proximate the top surface of the oil or fat to ensure that the alcohol/catalyst mix will mix and react effectively with the whole volume of the oil or fat in the reaction tank. In addition, the alcohol/catalyst mix is preferably added at a point proximate the stream of the individual air bubbles to ensure that localized reaction take places between the oil or fat adhered to the individual air bubble and the alcohol/catalyst mix.

The reaction mixture can comprise varying amounts of oil or fat and alcohol. Preferably, the ratio of straight vegetable oil / alcohol; waste cooking oil / alcohol is 1:4.5, 1:5.0, 1:5.5 or 1:6.0 as shown in the Examples below. The ratio of the alcohol/catalyst mix is preferably in the range of 1:0.013 to 0.027.

As the transesterification reaction progresses, biodiesel is formed. The products of the reaction include not only biodiesel, but also by-products such as glycerol, unreacted oil or fat, catalyst, unreacted alcohol, and trace amounts of water. All of the by-products are removed, though the order of removal is process-dependent.

In one embodiment of the invention, glycerol is separated from the biodiesel by using natural gravitational separation method. This gravitational separation method allows separation of individual phases of the mixture. Glycerol, which is much denser than the biodiesel, settles at the bottom of the reaction tank forming a lower phase, allowing it to be separated from the biodiesel. Biodiesel, which is less dense than glycerol, forms an upper phase of the reaction mixture.

In conventional gravitational separation method, phase separation can be observed within several hours of settling or overnight settling. In the phase separation in accordance with the method of this invention, phase separation can be observed within a relatively shorter period of time, for example, 1 to 3 hours based on the same volume of reactants used. This is attributed to the use of air bubbling in the phase separation step. The air bubbling which is initiated at the beginning of the process continued until substantially all the glycerol is separated and settled at the bottom of

the reaction tank. The air bubbles increase the rate at which the biodiesel and the glycerol separate. In one exemplary embodiment of the invention, phase separation has been observed to take between 1 to 3 hours to complete, based on a total volume of approximately 13.5L of the oil or fat and alcohol/catalyst mix used, from the time the alcohol/catalyst mix is added to the oil or fat.

As the glycerol separates from the reaction mixture and settles at the bottom of the reaction tank, the level of glycerol at the bottom of the reaction tank increases. Once the level of the glycerol reaches a level above the aerator, the aerator may be adjusted to a level higher than the level of the glycerol settled at the bottom of the reaction tank. The adjustment of the level of the aerator in the reaction tank reduces the aerator disturbance of the settled glycerol by the air bubbles injected into the reaction tank.

In one preferred embodiment, the reaction mixture is allowed to settle in the reaction tank for phase separation of the biodiesel and the glycerol. In another embodiment of the invention, the reaction mixture can be transferred to a separate settling tank, which may also be equipped with an aerator, for phase separation of the biodiesel and the glycerol. The glycerol, which is separated from the biodiesel, is then withdrawn from the reaction tank and the biodiesel is recovered. The recovered biodiesel can undergo further cleaning or purification processes, such as filtration and/or washing, to remove impurities if necessary. Any known methods of cleaning or purification processes can be employed.

In the method in accordance with the invention, the steps of heating the oil or fat and introducing the air bubbles into the reaction may occur simultaneously before the alcohol/catalyst mix is added to the reaction tank. Alternatively, these steps may occur one after another, that is, the oil or fat may be heated shortly after the air bubbles are introduced into the reaction tank or the oil or fat may be heated before the air bubbles are introduced into the reaction tank, before the alcohol/catalyst mix is added to the reaction tank.

In another embodiment of the invention, the steps of heating the oil or fat, introducing the air bubbles into the reaction tank and adding the alcohol/catalyst mix to the oil or fat at a controlled rate all occur simultaneously. In this embodiment, the rate at which

the alcohol/catalyst mix is added to the reaction tank may vary according to the temperature of the oil or fat in the reaction tank. The rate of adding the alcohol/catalyst mix may increase as the temperature of the oil or fat in the reaction tank increases from room temperature to between 55°C to 65°C. A convection current created by the heat and the agitation caused by the air bubbling allow the oil or fat and the alcohol/catalyst mix to mix and react effectively.

The method in accordance with the invention accelerates transesterification reaction by controlling the rate of adding the alcohol/catalyst mix to the oil or fat and introducing air bubbles into the reaction mixture to enable localized reactions to take place. This allows complete chemical reaction of the mixture to be reached in a shorter period of time and increases the conversion rate of the oil or fat into alkyl esters (or biodiesel).

The method of the invention is a semi-continuous process in that glycerol is separated from the reaction mixture as the reaction progresses. The glycerol is withdrawn from the reaction tank after the glycerol is separated from the reaction mixture. The biodiesel is recovered before the next batch of reaction mixture is added to the reaction tank. Alternatively, the glycerol produced by the method can be continuously withdrawn from the reaction tank as the reaction progresses. In this configuration, there will not be a need to adjust the level of the aerator during the phase separation process as the level of the settled glycerol in the reaction tank will always be lower than the level where the aerator is placed.

The reaction tank used in the method in accordance with the embodiments of this invention may be of any suitable design. In one exemplary embodiment of the invention, the reaction tank takes the form as depicted in Figures 2 to 5. The reaction tank 10 may comprise a reaction chamber 12, an inlet 16 for introducing the alcohol/catalyst mix into the reaction chamber 12 at a controlled rate and an aerator (not shown) disposed at the bottom of the tank for introducing air bubbles into the reaction chamber 12.

The reaction tank may also be one that is partitioned into several compartments. Each compartment is provided with an aerator disposed at the bottom end of the compartment for introducing air bubbles into the compartment and an injection jet disposed at the top open end of the compartment for introducing the alcohol/catalyst

mix into the compartment at a controlled rate. The air bubbling and the injection of the alcohol/catalyst mix into the reaction tank operate in the same manner as described above.

- 5 The use of air bubbling in the method of this invention allows air bubbles to surround the oil molecules (triglycerides) and thereby enables the preferential reaction. The air bubbles are capable of inhibiting water and free fatty acid molecules from getting close to one another to compete for the catalyst.
- 10 The method of this invention increases the efficiency of converting vegetable oil or animal fat into alkyl ester (or biodiesel) and also reduces the processing time (as both the reaction time and settling time are reduced), costs and energy.

15 The invention will be further described in the following example embodiments of the invention, which do not limit the scope of the invention as set forth in claims.

EXAMPLE 1

- 20 11 L of straight vegetable oil (SVO) was added to the reaction tank. The SVO was heated to a temperature between 55°C and 65°C, and the temperature was maintained at this level throughout the reaction. While the SVO was heated, air bubbles were introduced into the reaction tank through an aerator provided at the bottom of the reaction tank. An alcohol/catalyst mix was added to the SVO at a controlled rate to form a reaction mixture while the SVO continued to be agitated by
- 25 the air bubbling and heat. The alcohol/catalyst mix was pre-prepared by dissolving approximately 39.5774 g of sodium hydroxide in approximately 2.226 L of methanol to form an alcohol/catalyst mix containing sodium methoxide and methanol. As the alcohol/catalyst mix was added to the SVO, transesterification reaction began. The reaction continued with continuous introduction of the air bubbles into the reaction
- 30 mixture. As the reaction progresses, glycerol and biodiesel were produced. The glycerol settled at the bottom of the reaction tank, forming a lower phase comprised of glycerol and an upper phase comprised of biodiesel. The phase separation process and the air bubbling continued until all the alcohol/catalyst mix was added to the SVO and the reaction was completed. The total time taken for the biodiesel to separate

from the reaction mixture was approximately 1 hour 25 minutes, from the time the alcohol/catalyst mix was added to the SVO.

Other ratios of the SVO and methanol were also tested and the results are as shown in Tables 1 and 2.

TABLE 1

Volume of reactants (L)	SVO to Methanol Molar Ratio			
	4.5:1	5.0 : 1	5.5 : 1	6.0 : 1
SVO	11	11	11	11
Methanol	2.226	2.473	2.720	2.968
Sodium hydroxide	0.01858	0.01858	0.01858	0.01858
Total volume	13.24	13.49	13.74	13.99

TABLE 2

Results	SVO to Methanol Molar Ratio			
	4.5:1	5.0 : 1	5.5 : 1	6.0 : 1
Time taken for biodiesel to separate (with aeration)	1 hr 25 mins	1 hr 4 mins	1 hr 15 mins	1 hr 22 mins

EXAMPLE 2

The same amount of SVO was added to the reaction tank and the SVO was heated to the same temperature as described in Example 1. While the SVO was heated, the SVO was mixed using a stirrer. The same amount of alcohol/catalyst mix was then added to the SVO at the same controlled rate while the SVO continued to be agitated by the stirrer and heat. The reaction continued with continuous stirring until all the alcohol/catalyst mix was added to the SVO and the reaction was completed. As the reaction progresses, glycerol and biodiesel were produced. The glycerol settled at the bottom of the reaction tank, forming a lower phase comprised of glycerol and an upper phase comprised of biodiesel. In this example, the total time taken for the biodiesel to separate from the reaction mixture was approximately 8 hours (overnight), from the time the alcohol/catalyst mix was added to the SVO. Other ratios of SVO and methanol were also tested and the results are as shown in Table 3.

TABLE 3

Results	SVO to Methanol Molar Ratio			
	4.5:1	5.0 : 1	5.5 : 1	6.0 : 1
Time taken for biodiesel to separate (without aeration)	overnight	overnight	overnight	overnight

EXAMPLE 3

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The same method as described in Example 1 was used in this Example except that 11 L of waste cooking oil (WCO), about 2.079 L of methanol and about 55.02 g (or 0.2583 L) of sodium hydroxide were used instead. The total time taken for the biodiesel to separate from the reaction mixture was approximately 2 hours 15 minutes, from the time the alcohol/catalyst mix was added to the WCO. Other ratios of WCO and methanol were also tested and the results are as shown in Tables 4 and 5.

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TABLE 4

Volume of reactants (L)	WCO to Methanol Molar Ratio			
	4.5:1	5.0 : 1	5.5 : 1	6.0 : 1
WCO	11	11	11	11
Methanol	2.079	2.310	2.542	2.773
Sodium hydroxide	0.2583	0.2583	0.2583	0.2583
Total volume	13.1	13.6	13.8	14.0

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TABLE 5

Results	WCO to Methanol Molar Ratio			
	4.5:1	5.0 : 1	5.5 : 1	6.0 : 1
Time taken for biodiesel to separate (with aeration)	2 hrs 15 mins	2 hrs 8 mins	1 hr 57 mins	1 hr 45 mins

EXAMPLE 4

The same method as described in Example 2 was used in this Example except that 11 L of waste cooking oil (WCO), about 2.079 L of methanol and about 55.02 g (or 0.2583 L) of sodium hydroxide were used instead. The WCO and the alcohol/catalyst mix

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were mixed using a stirrer. In this example, the total time taken for the biodiesel to separate from the reaction mixture was approximately 8 hours (overnight), from the time the alcohol/catalyst mix was added to the WCO. Other ratios of WCO and methanol were also tested and the results are as shown in Table 6.

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TABLE 6

Results	WCO to Methanol Molar Ratio			
	4.5:1	5.0 : 1	5.5 : 1	6.0 : 1
Time taken for biodiesel to separate (without aeration)	overnight	overnight	overnight	overnight

The above is a description of the subject matter the inventor regards as the invention and is believed that others can and will design alternative systems that include this invention based on the above disclosure.

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Claims

1. A method for producing biodiesel, the method comprising:
feeding an oil source into a reaction tank;
5 subjecting the oil source to heat;
introducing air bubbles into the reaction tank at a predetermined rate;
introducing an alcohol/catalyst mix into the reaction tank at a predetermined
rate to form a reaction mixture;
permitting the reaction mixture to separate into an upper phase of biodiesel and
10 a lower phase comprised of glycerol; and
recovering the biodiesel from the reaction tank.
2. The method according to claim 1, wherein the air bubbles are introduced into
the reaction tank through an aerator disposed at lower end of the reaction tank.
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3. The method according to claim 1, wherein the air bubbles introduced into the
reaction tank form a stream of individual air bubbles.
4. The method of claim 3 wherein the stream of individual air bubbles circulates
20 inside the reaction tank.
5. The method according to claim 3 wherein the alcohol/catalyst mix is introduced
into the reaction tank at a point proximate the stream of individual air bubbles.
- 25 6. The method according to claim 1, wherein the alcohol/catalyst mix is introduced
into the reaction tank at a rate dependent on the temperature of the oil source.
7. The method according to claim 1, wherein the alcohol/catalyst mix is introduced
into the reaction tank through an inlet provided at an upper end of the reaction tank.
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8. The method according to claim 1, wherein the steps of subjecting the oil source
to heat and introducing the air bubbles into the reaction tank at the predetermined rate
occur simultaneously.

9. The method according to claim 1, wherein the steps of introducing air bubbles into the reaction tank at the predetermined rate and introducing the alcohol/catalyst mix into the reaction tank at the predetermined rate occur simultaneously.
- 5 10. The method according to claim 1, wherein the steps of subjecting the oil source to heat, introducing the air bubbles into the reaction tank at the predetermined rate and introducing the alcohol/catalyst mix into the reaction tank at the predetermined rate occur simultaneously.
- 10 11. The method according to claim 2, further comprising:
adjusting the placement of the aerator such that the aerator is disposed above the lower phase comprised of glycerol.
12. The method according to claim 1, wherein the air bubbles are introduced into
15 the reaction tank continuously until the biodiesel is recovered in step (vi).
13. The method according to claim 1, wherein the recovering of the biodiesel from the reaction tank includes washing and drying of the biodiesel to remove any remaining catalyst, alcohol, glycerol and excess water.
- 20 14. The method according to claim 1, wherein the oil source is selected from the group comprising vegetable oils, waste vegetable oils and animal fats.
15. The method according to claim 1, wherein the catalyst is selected from the
25 group comprising sodium hydroxide and potassium hydroxide.
16. The method according to claim 1, wherein the alcohol is selected from the group comprising methanol, ethanol, propanol, and other monoalkyl alcohols.