

A CONTINUOUS PROCESS AND AN APPARATUS FOR THE PREPARATION OF BIODIESEL

FIELD OF THE INVENTION

The invention relates to a process for the preparation of a biodiesel and more particularly, it relates to a continuous process for the preparation of a methyl ester.

BACKGROUND OF THE INVENTION

Biodiesel is made from vegetable oils, fats, greases, or other sources of triglycerides. Most biodiesel is prepared by a process of acid or base catalyzed transesterification.

Biodiesel may be prepared by a single-stage or a two-stage transesterification reaction process. These processes may be a batch process or a continuous process. The compounds that are commonly used in these transesterification reaction processes are triglycerides oil, a lower alkyl alcohol and a catalyst.

Transesterification reaction process of the compounds take place in a reactor by for example, mixing the triglycerides and the lower alkyl alcohol in the presence of the catalyst simultaneously in the reactor and subjecting the reaction mixture to heat to reach a temperature sufficient for the reaction to take place. In some cases, prior to introducing the three compounds into the reactor, the alcohol and the catalyst are pre-mixed to form an alcohol/catalyst solution before the solution is admixed with the triglycerides in the reactor and heated to reach the reaction temperature. In yet some cases, the triglycerides is heated prior to admixing with the alcohol and the catalyst or with the pre-mixed alcohol/catalyst solution. In these processes, mixing of the three compounds is traditionally carried out using devices such as a stirring reactor, a chemical mixing pump or a static mixer, etc.

In the above processes, in order to obtain good dispersion of the three compounds to aid in the reaction process, long mixing time is often required. In some cases, good

dispersion of the compounds cannot be achieved despite prolonging the mixing duration.

These processes are generally not cost effective due to inefficient mixing methods being adopted, resulting in long reaction times. Inefficient mixing results in poor dispersion of the compounds. This reduces process efficiency, leading to an increase in chemicals consumption and waste.

It is the object of the invention to provide a process for the preparation of a biodiesel with improved efficiency over the prior art or to at least provide the public with a useful choice.

It is also an object of the present invention to provide an apparatus for the preparation of a biodiesel with improved efficiency over the prior art or at least provide the public with a useful choice.

SUMMARY OF INVENTION

In a first aspect the present invention consists a process for the preparation of a biodiesel from a feedstock containing a triglyceride by transesterification of the triglyceride with a lower alkyl alcohol. The process comprises mixing the triglyceride with the lower alkyl alcohol in the presence of a catalyst in a high-shear mixer to form a reaction mixture, heating the reaction mixture in a heater to a temperature of 50° to 70°C, transferring the heated reaction mixture into a reactor to allow a transesterification reaction process to take place to form a first crude transesterification product and glycerol, separating the glycerol from the first crude transesterification product and subjecting the first crude transesterification product to further treatments to obtain the biodiesel.

Preferably the further treatments of the first crude transesterification product comprises mixing the first crude transesterification product with an additional lower alkyl alcohol in the presence of an additional catalyst in a second high-shear mixer to form a second

reaction mixture, heating the second reaction mixture in a second heater to a temperature of 50° to 70°C , transferring the heated reaction mixture to a second reactor to allow a second stage transesterification reaction process to take place to form a second crude transesterification product and glycerol with an overall conversion rate of at least 99%, and separating the glycerol from the second crude transesterification product.

Preferably the process further comprises the steps of neutralizing the second crude transesterification product, treating the second crude transesterification product with water to remove impurities, subjecting the second crude transesterification product to phase separation to remove the water phase, and drying the second crude transesterification product to obtain the biodiesel.

Preferably the separation of the glycerol from the crude transesterification product is carried out in the reactor that is configured in a manner that allows the glycerol to be removed continuously from the reactor.

Preferably the crude transesterification product is subject to further separation in a separator for removal of glycerol.

Preferably the triglyceride is selected from the group consisting of naturally occurring vegetable oils or animal fats including palm oil, coconut oil, palm kernel oil, rapeseed oil, canola, Jatropha oil and soybean oil.

Preferably the lower alkyl alcohol is methanol.

Preferably the catalyst is an alkali catalyst.

In a second aspect the present invention consists an apparatus for the preparation of a biodiesel from a feedstock containing triglyceride. The apparatus comprises at least two mixing units for mixing the feedstock with a lower alkyl alcohol and a catalyst to form a mixture product, at least two transesterification units each connected to one mixing

unit to allow introduction of the mixture product from the mixing unit to the transesterification unit, at least one separation unit downstream from each transesterification unit and at least one purification unit downstream from the last separation unit provided in the apparatus.

Preferably the purification unit comprises a high-shear mixer, a phase separation unit, and a vacuum drying device.

BRIEF DESCRIPTION OF DRAWINGS

For the purposes of illustrating the invention, there is shown in the drawings a form which is presently preferred. It is being understood however that this invention is not limited to the precise arrangements shown.

Figure 1 is a process flow diagram depicting the process and the apparatus associated with the preparation of biodiesel.

DETAILED DESCRIPTION OF THE INVENTION

The following description utilizes Figure 1 which depicts the process associated with one embodiment of the present invention for the preparation of biodiesel, particularly a methyl ester and an apparatus for such process, in flow chart form.

With reference to Figure 1, a feedstock 10, a lower alkyl alcohol 12 and a catalyst 14 are first introduced into a mechanically operated mixer 16. The feedstock 10 may be of any kind of naturally occurring vegetable oils or animal fats including palm oil, coconut oil, palm kernel oil, rapeseed oil, canola, Jatropha oil, soybean oil or other sources of triglycerides. Preferably, the feedstock 10 is triglycerides.

In the preferred embodiment, the lower alkyl alcohol 12 is methanol and the catalyst 14 is an alkali catalyst. The alkali catalyst may be sodium methylate, potassium methylate or metal hydroxides, such as potassium hydroxide or sodium hydroxide, in liquid form.

Those skill in the art will however appreciate that other alcohols and catalyst which are suitable for use in the preparation of biodiesel may also be adopted. About 70% – 90% of the lower alkyl alcohol and the catalyst 14 are added into the mixer 16.

The triglycerides 10, the methanol 12 and the alkali catalyst 14 are mixed intensively in the mixer 16 under a high-shear condition for a period of about 0.1 to 10 seconds under a pressure of about 1×10^5 Pa to 3×10^5 Pa to form a reaction mixture. The mixing of the triglycerides 10 and the methanol 12 in the presence of the alkali catalyst 14 under a high-shear condition is to ensure that the three compounds are well mixed by being in intimate contact with each other before they are subject to heat and/or before they are introduced into a reactor 20 for reaction to take place. The intensive mixing helps to accelerate the reaction by effectively reduces the size of the liquid phase droplets which comprises the methanol 12 and the alkali catalyst 14. This improves the dispersion of the methanol 12 and the alkali catalyst 14 and hence, increases the contacting surface area of the liquid phase exposed to the triglycerides 10. By increasing the number of droplets and reducing the droplet size, chemical equilibrium state can be reached quickly. This enormously shortens the reaction time, reduces chemical consumption and therefore, improves efficiency in the preparation process.

In the preferred form, the mixer 16 is a high-shear mixer with slotted-hole or square-hole high-shear screen.

The reaction mixture obtained from the intensive mixing is then transferred to a heater 18 where the reaction mixture is heated to a temperature of about 50° to 70°C before it is transferred to a first reactor 20.

In the first reactor 20, a first stage transesterification reaction process takes place. A by-product glycerol 21 is formed. Due to its heavier density as compared to the rest of the compounds in the reaction mixture, the glycerol 21 formed thereto separates and settles at the bottom of the first reactor 20 as the reaction continues. The glycerol 21 that is separated from the reaction mixture can optionally be removed continuously from the first reactor 20 while the reaction continues or be removed after the reaction in the first

reactor 20 is completed. In the preferred form, the former is adopted so as to provide a continuous flow of the reaction mixture and the by-product in and out of the reactor. The continuous process helps to reduce the overall process time required for the preparation of the biodiesel. A conversion rate of about 80 to 90% is usually obtained in the first reactor 20. A crude transesterification product is obtained from the first stage transesterification reaction process which comprises mainly crude methyl ester, unreacted triglycerides, glycerol, lower alkyl alcohol, catalyst and other impurities. The reaction time in the first reactor 20 is approximately 30 to 60 minutes.

The next step in the process is phase separation of the crude transesterification product. The crude transesterification product is passed through a separator 22. The separator 22 can be a settling tank or a highly efficient centrifugal separator, within which different phases of the crude transesterification product is separated based on their differences in densities. Due to the low solubility of the glycerol in the crude methyl ester and the distinct density difference, separation of the glycerol from the crude methyl ester generally occurs quickly by merely standing the crude transesterification product in the separator 22 for about 2 seconds to 2 hours depending on the type of separator 22 that has been adopted. The light phase obtained from the phase separation comprises mainly crude methyl ester the unreacted triglycerides, lower alkyl alcohol and catalyst. The heavy phase comprises mainly the glycerol that settles at the bottom of the separator 22, ready to be removed from the separator 22 either continuously or discontinuously at any desired time.

The light phase obtained from the phase separation is then subject to a second stage transesterification. Prior to that, the light phase is admixed with a remaining 10% to 30% of the lower alkyl alcohol 12 and the catalyst 14 in a second mixer 24 to form a second reaction mixture. The second mixer 24 is preferably of the same kind as that of the first mixer 16 so that the same effective mixing results can be achieved. The lower alkyl alcohol 12 and the catalyst 14 are preferably the same as those used in the first stage transesterification reaction process. The second reaction mixture is intensively mixed in the second mixer 24 under a high-shear condition for a period of about 0.1 to 10 seconds, under a pressure of about 1×10^5 Pa to 3×10^5 Pa before it is transferred to a

second heater 26. In the second heater 26, the second reaction mixture is heated to a temperature of about 50° to 70°C. The heated second reaction mixture is then transferred to a second reactor 28 for the second stage transesterification reaction process to take place to form a second crude transesterification product.

The glycerol 21 formed during the second stage transesterification is separated from the second crude transesterification product in the same manner as it is formed and separated in the first reactor 20. The glycerol 21 can optionally be removed continuously or discontinuously at any desired time as in the first stage transesterification. In the preferred form, the glycerol 21 is removed continuously to allow a continuous flow of the reaction mixture and the by-product in and out of the reactor. An overall conversion rate of more than 99% is obtained in the second stage transesterification reaction process. The reaction time in the second reactor is approximately 30 to 60 minutes.

The second crude transesterification product which comprises mainly the crude methyl ester, the glycerol and impurities is subsequently passed through a second separator 30 for phase separation to separate and remove any glycerol from the second crude transesterification product. This is to prevent or reduce any form of back reaction. The second separator 30 can be a settling tank or a highly efficient centrifugal separator as described above. The phase separation in the second separator 30 occurs in the same manner as that described above for the first separator 22. The light phase obtained from the phase separation comprises mainly crude methyl ester and some impurities and the heavy phase comprises the glycerol 21. The glycerol 21 recovers from the bottom of the separator 30, ready to be removed from the separator 30 either continuously or discontinuously at any desired time.

The light phase obtained from the second stage transesterification is optionally passed through a alcohol stripping device 32 for removal of remaining excess alcohol 33 when required. Preferably, the alcohol stripping device 32 operates under a vacuum condition of approximately 4×10^3 to 7×10^3 Pa. Alternatively, a falling film evaporator can be adopted.

The light phase is then subject to neutralization by admixing with a strong acid 35 or an acid complex 35 in a third mixer 34. The third mixer 34 is preferably also a high-shear mixer as described above, although it is envisaged that other type of mixer can also be adopted. The strong acid may be phosphoric acid, hydrochloric acid or sulfuric acid and preferably phosphoric acid is used. If an acid complex is used, the acid complex 35 is preferably citric acid. An acid is added at this stage to neutralize any residual catalyst and to split soap that may have been formed during the transesterification reaction processes into water-soluble salts and free fatty acids.

A water washing step 37 is then carried out to remove any remaining by-products and/or impurities from the light phase, which at this stage, comprises mainly the crude methyl ester. The by-products and/or impurities can be water-soluble salts formed during the neutralization, soap, catalyst or any free glycerol. In the preferred embodiment, the neutralization step takes place before the water washing step 37. This is so as to reduce the amount of water required for water washing and to minimize the potential of emulsions forming when the wash water is added to the crude methyl ester.

The crude methyl ester and the water from the water washing then undergo a phase separation in a centrifugal separator 36 to prevent excessive contacting time between the crude methyl ester phase and the water phase 39 so as to reduce the possibility of hydrolysis from taking place. Additional washing steps may be adopted as desired.

The crude methyl ester phase obtained thereto is then dried under a vacuum drying device 38 to remove residual traces of water. The vacuum drying device 38 operates at a pressure of about 4×10^3 to 1×10^4 Pa. The dried methyl ester is suitable for use as a biodiesel.

The continuous removal of the glycerol described above is made possible by using the reactors 20, 28 having a controlled flow unit with baffles that allows the glycerol to readily separates from the reaction mixture as the transesterification reaction process in the reactors continues. The continuous separation of the glycerol in the reactors allows

the glycerol to be removed continuously from the reactors as it separates and settles at the bottom of the reactors. This continuous removal of the glycerol reduces the time required to carry out a separate separation process and in turn, it reduces the overall process time required to prepare the biodiesel.

In the present invention, a more efficient mixing of the compounds and a more favorable reaction kinetics can be achieved to provide for shorter residence times and a more economical process for the preparation of the biodiesel. The 2-stage transesterification reaction process of the present invention allows a high degree of reaction conversion and enables a continuous removal of the by-product, i.e. glycerol, from almost every stage of the reaction process. The efficient mixing methods used in combination with the continuous transesterification process results in an efficient process for the preparation of the biodiesel. This combination also helps reduce the amount of chemicals, particularly alcohol, required for the process. This reduces chemicals consumption.

The apparatus used in the present invention comprises the components of at least two reaction units and a purification unit. Each reaction unit comprises a mixer, a reactor and a separation unit. In the assembled form, the mixer of each reaction unit is provided downstream of the separation unit of another reaction unit so as to provide for a continuous flow of the preparation process. The mixer used in the present invention is a high-shear mixer as described above. The separation unit preferably is a settling tank or a centrifugal separator.

The purification unit of the apparatus preferably comprises an alcohol stripping device, a mixer, a phase separation unit and a drying device. The mixer used in the purification process is preferably a high-shear mixer, although it is envisaged that other types of mixer can be adopted.

The foregoing describes the invention including preferred forms thereof. Alterations and modifications as will be obvious to those skilled in the art are intended to be incorporated within the scope hereof as defined by the accompanying claims.

Claims

1. A process for the preparation of a biodiesel from a feedstock containing a triglyceride by transesterification of the triglyceride with a lower alkyl alcohol, comprising the steps of:-

mixing the triglyceride with the lower alkyl alcohol in the presence of a catalyst in a mixer under a high-shear condition to form a reaction mixture;

heating the reaction mixture in a heater to a temperature of 50° to 70°C;

transferring the heated reaction mixture into a reactor to allow transesterification reaction process to take place to form a first crude transesterification product and glycerol;

separating the glycerol from the first crude transesterification product; and

subjecting the first crude transesterification product to further treatments to obtain the biodiesel.

2. The process according to claim 1, wherein the further treatments of the first crude transesterification product comprising the steps of:

mixing the first crude transesterification product with an additional lower alkyl alcohol in the presence of an additional catalyst in a second mixer under a high-shear condition to form a second reaction mixture;

heating the second reaction mixture in a second heater to a temperature of 50° to 70°C;

transferring the heated reaction mixture into in a second reactor to allow a second stage transesterification reaction process to take place to form a second crude transesterification product and glycerol with an overall conversion rate of at least 99%; and

separating the glycerol from the second crude transesterification product.

3. The process according to claim 2, further comprising:-

neutralizing the second crude transesterification product;

treating the second crude transesterification product with water to remove

impurities;

subjecting the second crude transesterification product to phase separation; and
drying the second crude transesterification product to obtain the biodiesel.

4. The process according to claim 3, wherein the neutralization of the second crude transesterification product is carried out in a third mixer under a high-shear condition by mixing the second crude transesterification product intensively with a strong acid or a strong acid complex selected from phosphoric acid, hydrochloric acid, sulfuric acid, citric acid.
5. The process according to claims 1 or 2, wherein the separation of the glycerol from the crude transesterification product is carried out in the reactor that is configured in a manner that allows the glycerol to be removed continuously from the reactor.
6. The process according to claim 5, wherein the crude transesterification product is subject to further separation in a separator for removal of glycerol.
7. The process according to claim 6, wherein the separator is a settling tank or a centrifugal separator.
8. The process according to any one of the preceding claims, wherein the mixer is a high-shear mixer.
9. The process according to any one of the preceding claims, wherein the triglyceride is selected from the group consisting of naturally occurring vegetable oils or animal fats including palm oil, coconut oil, palm kernel oil, rapeseed oil, canola, Jatropha oil and soybean oil.
10. The process according to any one of the preceding claims, wherein the lower alkyl alcohol is methanol.
11. The process according to any one of the preceding claims, wherein the catalyst

is an alkali catalyst.

12. The process according to claim 11, wherein the catalyst is sodium methyllate, potassium methyllate, potassium hydroxide or sodium hydroxide.

13. The process according to any one of the preceding claims, wherein the biodiesel is methyl ester.

14. An apparatus for the preparation of a biodiesel from a feedstock containing triglyceride, the apparatus comprising:

at least two mixing units for mixing the feedstock with a lower alkyl alcohol and a catalyst to form a mixture product;

at least two transesterification units each connected to one mixing unit to allow introduction of the mixture product from the mixing unit to the transesterification unit;

at least one separation unit downstream from each transesterification unit; and

at least one purification unit downstream from the last separation unit provided in the apparatus.

15. An apparatus according to claim 14, wherein the purification unit comprising:

a mixer;

a phase separation unit; and

a vacuum drying device.

16. An apparatus according to claims 14 and 15, wherein the mixer is a high-shear mixer.