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Production of biodiesel by transesterification of Jatropha oil with microwave heating

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ABSTRACT

Biodiesel has attracted attention due to energy crisis and CO_2 issue. Using an efficient processing technology to prepare biodiesel from Jatropha seed oil is a competitive way to produce biodiesel. Biodiesel was obtained by transesterification reaction using KOH catalyst under microwave radiation in this study. Since the side reactions occur when water and free fatty acids (FFA) exist, a pretreatment is needed. The pre-esterification was used to reduce the amount of FFA. The optimum pre-esterification reaction conditions were 70 °C, 12 wt% methanol, 1 wt% sulfuric acid, and 2 h reaction time. To accelerate the reaction rate of transesterification, microwave radiation was used. The optimum reaction conditions in microwave batch reactor were the molar ratio of oil to methanol of 1: 6, 1 wt% KOH, 200 rpm, and 65 °C. The conversion of the oil was 90% after 10 s reaction time. The reaction rate in microwave reactor was much faster than that by the conventional heating method. The penetration length of microwave in Jatropha seed oil was calculated to be 27 cm. A large scale process was developed by using a continuous microwave reactor. This process has high conversion in a very short reaction time under mild reaction conditions.

1. Introduction

Biodiesel is a reliable, efficient and clean energy supply for sustainable economic development. How to produce biodiesel at a low cost is the main issue in industry. The interest in using Jatropha curcas as a feedstock for the production of biodiesel is growing in the last decade [1–4]. The oil produced by this crop can be easily converted to liquid biofuel that meets the American and European standards. It can be produced in an environmentally and socially sustainable manner in tropical countries. Many researchers used solid base catalysts for this reaction [5]. However, the reaction rate was lower than that of liquid base. In addition, most of solid base catalysts were not stable.

Although transesterification of Jatropha oils to produce biodiesel is a well-established method, the low conversion and energy utilization inefficiencies in the process result in the high cost of biodiesel. The heating method employed in the transesterification is one of the key issues. Conventional heating method requires longer reaction times with higher energy inputs and losses to the ambient. Super and sub-critical methanol process operates in expensive reactors at high temperatures and pressures resulting in higher energy inputs and higher production costs. The enzymatic method, though operates at much lower temperatures, requires much longer reaction times. Microwave-assisted transesterification, on the other hand, is an energy-efficient and fast process to produce biodiesel from various feedstocks.

Microwave irradiation has been used for a variety of applications including organic synthesis reaction. The chemical reactions are accelerated because of selective absorption of microwave energy by polar molecules. Because the mixture of vegetable oil, methanol, and potassium hydroxide contains both polar and ionic components, rapid heating occurs upon microwave irradiation. Because the energy interacts with the reactant on a molecular level, very efficient heating is obtained. Instead, the conventional heating is relatively slow and inefficient because transferring energy into a reactant depends upon convection currents and the thermal conductivity of the reaction mixture.

Although microwave heating has been used for transesterification, the optimum reaction condition and the size of flow reactor has not been extensively studied. The aim of this research was to develop a method to make biodiesel from Jatropha seed oil by using an eco-friendly way. Microwave heating was used in this study, instead of conventional heating method.

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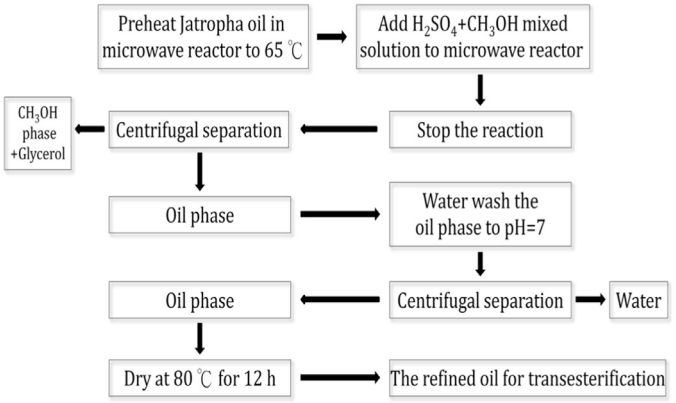

Fig. 1. The process for producing refined Jatropha seeds oil.

2. Experimental

2.1. Chemicals

Jatropha seed oil used in this study was provided by Bioptik Technology Inc., Taiwan. Methanol (Mallinckrodt Chemicals), potassium hydroxide (Nihon Shiyaku Industries), sulfuric acid (Showa), ethanol (J. T. Baker), ether (Tedia) and benzoic acid (Janssen Chimica) were used without purification.

2.2. Two-steps method of preparing biodiesel

A two-step transesterification process was used to convert Jatropha seeds oil to methyl esters. The pre-esterification process was used because of the high free fatty acid content of Jatropha seeds oil which caused fatty acid salts (soap) formation during base-catalyzed transesterification. The soap would prevent separation of the methyl ester layer from the glycerin fraction. The pre-esterification was used to reduce the amount of free fatty acids (FFA), then base-catalyzed transesterification was carried out to obtain desired product.

2.2.1. Pre-esterification reaction

The acid-catalyzed esterification was used to remove the free fatty acids in the oil. In this step, free fatty acids were converted to esters in a pretreatment process with methanol and an acid catalyst, H_2SO_4. 1 L reactor was filled with 700 g Jatropha seeds oil. The catalyst, sulfuric acid, was dissolved in methanol and then added into the reactor at a reaction temperature of 70 °C. Agitation was set at a constant speed (400 rpm) throughout the experiment. The reaction was carried out in the best operation condition. The aim of this pretreatment was to obtain refined Jatropha seeds oil in which the acid value was lower than 1 mg KOH/g oil and the amount of water in the oil was less than 1 wt%. The detailed procedure is described in Fig. 1.

2.2.2. Transesterification reaction

In the second step, the product of the pretreatment step was poured into the microwave reactor and preheated to 65 °C. The catalyst, KOH, was dissolved in methanol and then fed into the reactor through the condenser. The reaction was carried out under various operating conditions in order to find the optimum operating condition. At the end of the reaction, the mixture was cooled to room temperature and separated by centrifuge. The methyl ester phase was washed with hot distilled water (70–80 °C). In order to avoid emulsion during the washing step, the condition should be mild. The product of transesterification was measured by hydrometer to obtain the density, and the conversion of Jatropha seeds oil was calculated. The procedure of reaction is shown in Fig. 2.

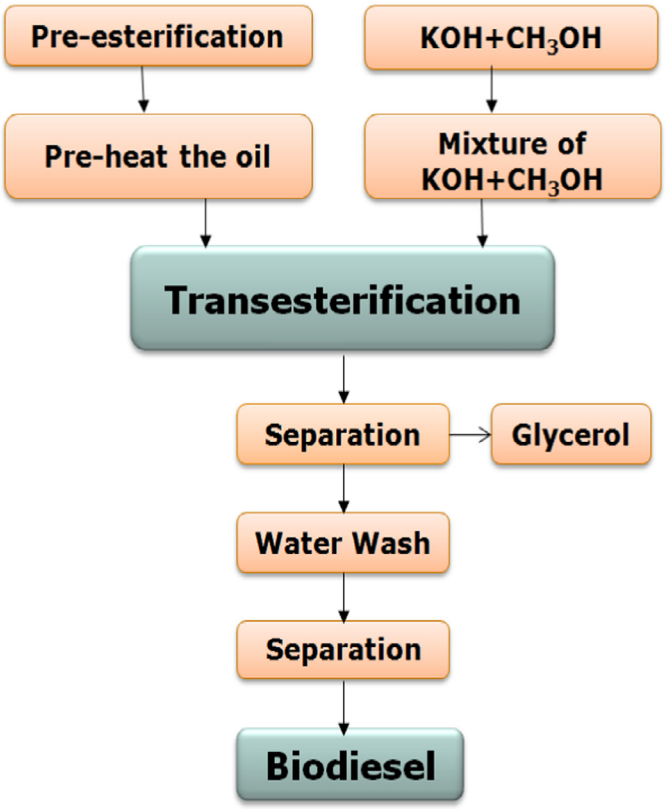

Fig. 2. The process for producing biodiesel.

The microwave reactor used in this research was equipped with a thermometer, a temperature controller, a mechanical agitator fitted with a simple straight-blade turbine provided the mixing requirement and a water-cooled condenser (see Fig. 3). The output power of this microwave reactor was adjustable up to 950 W, controlled by a microprocessor.

2.3. Determination of acid value

Acid value is the mass of potassium hydroxide (KOH) in milligrams that is required to neutralize one gram of chemical substance. The acid number is a measure of the amount of carboxylic acid groups, such as a fatty acid, in a chemical compound or in a mixture of compounds [6]. The acid value was determined by ASTM D1980. In the typical procedure, a known amount of sample dissolved in isopropanol was titrated with a solution of potassium hydroxide with known concentration and phenolphthalein was used as a color indicator.

The concentration, N, of KOH/EtOH standard solution was calculated by the following equation:

$$N = \frac{\text{Benzoic acid(g)} \times 1000}{22 \times \text{the volume of standard solution used in the titration(ml)}} \quad (1)$$

The acid value was calculated by the following equation:

$$\text{Acid Value} = \frac{V \times 56.11 \times N}{W} \quad (2)$$

V = the volume of standard solution used in the titration (ml)

N = the accurate concentration of KOH/EtOH standard solution

W = weight of sample (g)

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