Continuous Production of Biodiesel from Waste Cooking Oil in Corning® Advanced-Flow™ Reactor

S. Srinath, Shekhar M. Gaikwad, and S.H.Sonawane

***Abstract*----** This Study includes the continuous production of biodiesel from used cooking oil in Corning® Advanced-Flow™ reactor. Biodiesel can be used most effectively as a supplement to other energy liquid fuels such as diesel fuel. In a lab scale experiment, used cooking oil was used to produce biodiesel via transesterification reaction. The effects of acid catalyst (H_2SO_4) concentration (0-2%), flow rates (10-50 mL/h) of reactant with the increment of 10 mL/h, temperature (40 ºC - 80 ºC) with the increment of 20 ºC were studied. The maximum conversion was obtained at optimal conditions of 2% catalyst, 30 mL/h flow rate and temperature 80 ºC. The residence time for this reaction was 81s.

Keywords---Biodiesel, acid catalyzed transesterification reaction, FAME, Corning®Advanced-Flow reactor.

I. Introduction

BIODIESEL can be used most effectively as a supplement to other energy liquid fuels such as diesel fuel. It can be produce from renewable biological sources such as vegetable oils and animal fats. It is biodegradable and non-toxic, has low pollutant emission and is therefore environmentally beneficial. Biodiesel's physical properties are similar to those of petroleum diesel, but it is a cleaner-burning alternative. Using biodiesel in place of petroleum diesel, especially in older vehicles, can reduce emissions [2]. Biodiesel is defined as a mixture of mono alkyl esters of long chain fatty acids derived from renewable lipid sources [2]. Biodiesel is typically produced by transesterification of vegetable oil or animal fat with alcohol in the presence of a catalyst to yield glycerine and biodiesel. Biodiesel also has desirable degradation attributes. Pure biodiesel (B100) is 100% biodegradable compared to petroleum diesel [2]. Biodiesel has been produced in different ways such as through direct use and blending, transesterification and more recently developed methods such as reaction with supercritical methanol [1][2][3]. In this study biodiesel produced by the catalytic transesterification method.

A variety of oils (both vegetable oils and animal oils) can be used to produce biodiesel such as sunflower oil, palm oil, and pork lard [3]. Feedstock from vegetable oils and animal oils causes the production of biodiesel to be very expensive. Exploring new methods to produce biodiesel from low-cost raw materials such as non edible crude oils, by-products of the refining vegetable oils and waste cooking oil (WCO) are the main interests in recent biodiesel research. The price of waste cooking oil is cheaper than fresh vegetable oils. Therefore, the total manufacturing cost of biodiesel can be appreciably reduced. Due to increasing food consumption large amount of waste cooking oil is available. The conversion of this amount of this waste oil into fuel also eliminates the environmental impacts caused by the harmful disposal of these waste oils.

The objective of the study was the continuous production of biodiesel in AFR™ and to find out the optimal conditions at which maximum oil conversion can be achieved.

II. Experimental

II.1 Chemicals

Waste cooking oil collected from the hostel messes of NITW,INDIA. Methanol (98-99V/V%, SRL Pvt. Ltd., Mumbai), and sulfuric acid (98V/V%, Molychem, Mumbai) were the other chemicals used in the experiments as reactants. Iso propanol (98-99%, SD-Fine Chemical, Mumbai), sodium hydroxide (99%, SD-Fine Chemical, Mumbai), were used for the analysis of biodiesel samples, All chemicals were used as they were received without any further purification. Millipore water is used for preparing the solutions having conductivity of 3μS.

II.2 Reaction

In Advanced-Flow™ reactors, the following reaction has been carried out at a targeted temperature and results were studied. The chemical reaction is shown in equation (1). The reaction is carried out in the presence of sulfuric acid as a homogeneous catalyst in catalytic amounts.

$$RCOOH + CH_3OH \leftrightarrow RCOOCH + H_2O \quad (1)$$

$$\text{Triglyceride} + ROH \leftrightarrow \text{Diglyceride} + R'COOR \quad (2)$$

$$\text{Diglyceride} + ROH \leftrightarrow \text{Monoglyceride} + R''COOR \quad (3)$$

$$\text{Monoglyceride} + ROH \leftrightarrow \text{Glycerol} + R'''COOR \quad (4)$$

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II.3Experimental set-up:

The experiments were conducted in Corning Advanced-Flow® reactor lab. The set-up for the synthesis of biodiesel is shown in Fig 1. It consists of AFR^{TM}, syringe pumps, peristaltic pump and a cooling unit. AFR^{TM} contains the heart shaped modules made up of glass with the volume of 0.45 mL each. For this experiment, two modules were used to help maintain suitable residence time. Two syringe pumps were used in order to feed the reactants in the AFR^{TM} system. Flow rates of feed were operated in the range of 10 to 60 mL/h. Teflon tubes were used for connecting syringes with AFR^{TM}. The peristaltic pump was used for maintaining the utility requirements. The cooling flow rate remained a constant at 300 mL/h. Before starting the experiments the system should be calibrated. Once the set-up is calibrated, it is ready for operation.

Fig 1: Experimental set-up of synthesis of biodiesel in two AFR^{TM}reactor modules

In this experiment, the AFR^{TM} consisted of two modules and the plate arrangement shown in the Fig 1. As we can see in Fig 1**,** there are four glass plates are arranged in the manner such that a chemical reaction is takes place inside the reaction layer. Both reactants are fed to the reactor simultaneously, and the outer layer called heat exchange layer is the way for utility. These layers manage to maintain the temperature of chemical reactions by passing the hot or cold utility as per the requirements [12].

II.4 Experimental procedure of synthesis biodiesel

The experiment consisted of two feed streams injected in AFR by syringe pumps as shown in Fig 1. One feed was used cooking oil, and the other was methanol (98-99 W/W %). They were both fed in as 1:1 molar ratio and 1:3 volume ratios [5]. Initially experiments were carried out without using catalyst and then with sulfuric acid as a homogeneous catalyst at same operating conditions. Sulfuric acid was added to methanol in a measured amount and then fed into the Advanced-Flow™ reactor. The use of a catalyst in a reaction accelerates the reaction rate and oil conversion. Experiments were conducted at atmospheric pressure and different temperature conditions were maintained using utility as water from the chiller at respective temperatures. Various experiments were conducted with changes in the system such as different flow rates for feeds, different catalytic concentration, different residence times and different operating temperatures for biodiesel synthesis.

III. Results and Discussion

III.1 Effect of residence time with and without catalyst on the conversion of waste cooking oil

The transtesterification reactions were carried out with and without homogeneous catalyst. Residence time for the reaction varies inversely with the feed rate. Two feeds one was used cooking oil and other was methanol were fed to the AFR^{TM} in the volume ratio of 1:3, and the temperature was 60 °C, Each sample was collected after the fixed time of five minutes. The sample obtained contains the biodiesel (FAME) and glycerol where these two can be separated by gravity settling because of different densities and analyzed by titration. Fig 2 shows that at a flow rate of 30 mL/h and in the presence of 2% homogeneous sulfuric acid catalyst the conversion of oil maximum.

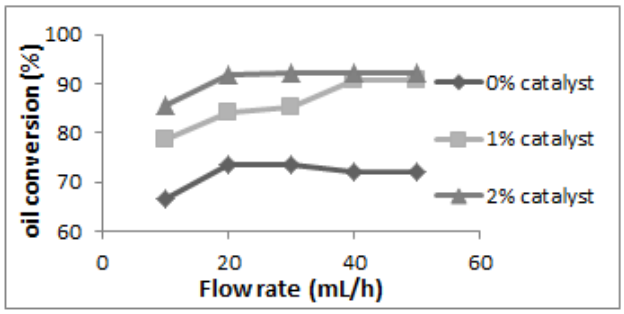

Fig 2: Effect of residence on the conversion of used cooking oil with different flow rates with varying catalyst concentration.

III.2 Effect of different temperatures at different flow rates

For further confirmation of the optimum flow rate and identifying the optimum temperature for synthesis of biodiesel in AFR^{TM}, the reactions were carried out at different temperatures with different flow rates ranging from 10 mL/h to 50 mL/h with the increment of 10 mL/h at catalytic concentration of 2W/W %H_2SO_4. Fig 3 confirms the optimum feed rate as 30 mL/h. Also, it shows that the reaction gives high conversion of used cooing oil as 92.3% at a temperature of 80℃. As the temperature increases from 40℃ to 80℃, the reaction rate and also the conversion increase.

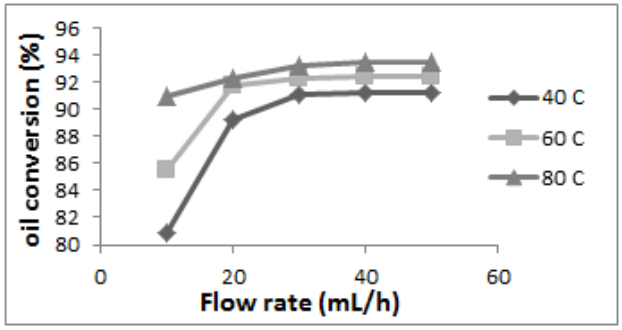

Fig 3: Effect of residence on the conversion of Waste Cooking Oil with different flow rates with different operating temperature.

III.3 Effect of temperature with respect to time on the conversion of waste cooking oil

The effect of temperature on reaction rate and conversion of used oil were studied experimentally. The reactions were

carried out with 2W/W % of H_2SO_4 catalyst and flow rate of 30 mL/h with different operating temperatures as 40^0C to 80^0C with the increment of 20 ^{0}C. It can be seen from the Figure 4 that the oil conversion increases with the temperature. It is constant near the last two points that means it does not change further. Thus 80^0C is most preferable temperature for this reaction to be carried out in AFR^{TM}.

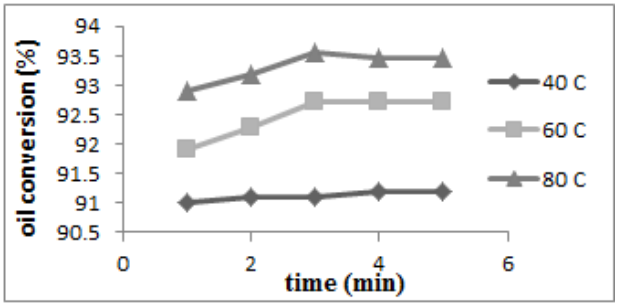

Fig 4: Effect of temperature on the conversion of Waste Cooking Oil with respect to time.

III.5 Effect of catalyst concentration with respect to time on the conversion of Waste Cooking Oil

Reactions were performed using homogeneous catalyst sulfuric acid**.** Fig 5 shows the conversion of used oil with respect to time at different catalyst concentrations. Biodiesel is also formed without catalyst but it has been observed that with the addition of catalyst there is more conversion. Also further addition of H_2SO_4 above 2 W/W % does not show effective changes in the conversion. The conversion of oil remains almost the same after the complete reaction occurs. Further increase in the concentration of H_2SO_4 had minor effects on the rate of production and conversion of used cooking oil.

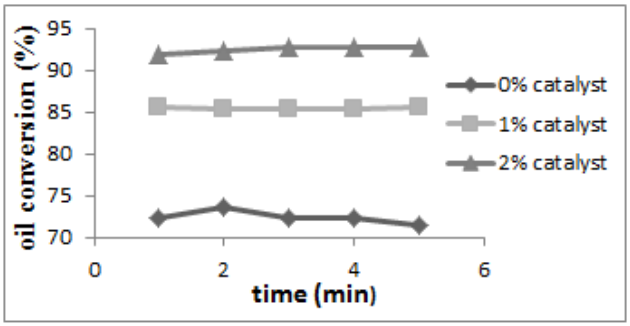

Fig 5: Effect of catalyst concentration on the conversion of Waste Cooking Oil with respect to time.

IV. Conclusion

Corning® Advanced Flow^{TM}reactors are the efficient reactors using advanced technology to convert a batch process into a continuous process, along with fulfilling the measure aspects of process intensification and sustainability. Biodiesel produced efficiently using AFR^{TM} technology. Also, the effect of various parameters like temperature, flow rate, catalyst concentration is studied and the optimal conditions for the synthesis of biodiesel from Waste Cooking Oil were found to be at flow rate of 30 mL/h, temperature 80 ^{0}C and 2 W/W % H_2SO_4 catalyst.

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