Biofuel Research Journal 2 (2014) 70-76

Biofuel
Research Journal
Journal homepage: www.biofueljournal.com

Original Research Paper

Optimization of alkali catalyst for transesterification of jatropha curcus using adaptive neuro-fuzzy modeling

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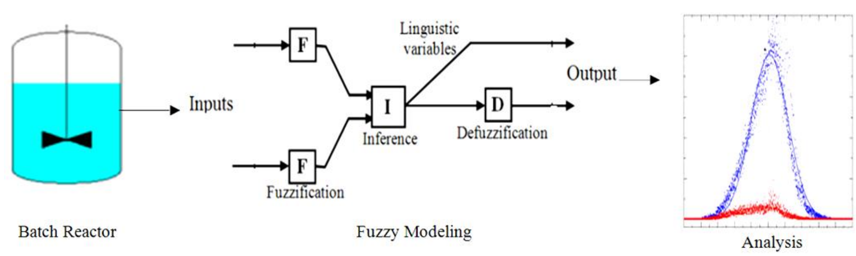
HIGHLIGHTS

- Biodiesel production from non edible oil through transesterification in batch reactor is highly effective technique for kinetic analysis.
- Temperature, molar ratio, mixing intensity and catalyst influenced the biodiesel production and kinetic. Alkaline catalysts are more efficient in nature as compare to acid and base catalyst.
- This paper particularly focuses on the impact of NaOH catalyst on transesterification process and yield of butyl ester production. ANFIS used to modeling and assess the output for large domain. In addition, K-S statistical tests are used for analysis of the ANFIS modeling.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 21 February 2014
Received in revised form 31 March 2014
Accepted 1 April 2014
Available online 13 May 2014

Keywords:
Biodiesel
Jatropha curcus
Transesterification
Batch reactor
NaOH catalyst
Adaptive neuro fuzzy inference

ABSTRACT

Transesterification of Jatropha curcus for biodiesel production is a kinetic control process, which is complex in nature and controlled by temperature, the molar ratio, mixing intensity and catalyst process parameters. A precise choice of catalyst is required to improve the rate of transesterification and to simulate the kinetic study in a batch reactor. The present paper uses an Adaptive Neuro-Fuzzy Inference System (ANFIS) approach to model and simulate the butyl ester production using alkaline catalyst (NaOH). The amounts of catalyst and time for reaction have been used as the model's input parameters. The model is a combination of fuzzy inference and artificial neural network, including a set of fuzzy rules which have been developed directly from experimental data. The proposed modeling approach has been verified by comparing the expected results with the practical results which were observed and obtained through a batch reactor operation. The application of the ANFIS test shows which amount of catalyst predicted by the proposed model is suitable and in compliance with the experimental values at 0.5% level of significance.

1. Introduction

Biodiesel production from non-edible oils involves different chemical reactions, but transesterification with alcohol has long been a preferred method for producing biodiesel. The process of transesterification is controlled by various process parameters such as catalyst type & concentration, molar ratio of alcohol to oil, type of alcohol, reaction time & temperature and mixing intensity. But the most challenging part of the biodiesel industry is the high cost of raw materials due to the low availability of non-edible oils (Jatropha Curcas). The price per unit for building up the catalyst alone makes up about 50% of the total production cost. So the catalysts used for the transesterification process are a rate determining parameter. The transesterification rate can be controlled using three different categories of catalysts. Sodium and potassium hydroxide are more effective

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among alkali catalysts, while acid catalyzed transesterification functions in the presence of HC l and H_2SO_4. Enzymatic catalysts especially lipases are able to effectively catalyze the transesterification of triglycerides in both aqueous or non-aqueous systems.

Alkali catalysts like sodium hydroxide, sodium methoxide, potassium hydroxide and potassium methoxide are more effective (Zhang et al, 2003) when lower levels of free fatty acids and water are present. Conversely acid catalyzed transesterification is suitable, if the base oil has high free fatty acid content and more water. Sulfuric acid, phosphoric acid, hydrochloric acid or organic sulfonic acids are the various types of acidic catalysts. A methanolysis of beef tallow was studied with NaOH and NaOMe used as catalysts. The study found that NaOH was significantly better than NaOMe (Ma et al, 1998). In addition, it was found that high quality oil is required when NaOMe catalyst is used, which itself is a limiting factor (Ahn et al, 1995). 1% NaOH and 0.5% NaOMe with a higher molar ratio, exceeding the 6:1 ratio, perform almost the same conversion after 60 minutes run (Freedman et al, 1986). Although methanolysis of soybean oil with 1% potassium hydroxide as catalyst has given the best yields but higher viscosities of the esters causes a problem in the separation process (Tomasevic and Marinkovic, 2003). A study of the catalytic activity of magnesium oxide, calcium hydroxide, calcium oxide, calcium methoxide, barium hydroxide, compared to sodium hydroxide during the transesterification of rapeseed oil was carried out (Gryglewicz, 1999) and (Noureddini and D. Zhu, 1997). The study concluded that sodium hydroxide exhibited the highest catalytic activity in this process. The acid catalyzed transesterification wa also studied with waste vegetable oil (Canakci and Van Gerpen, 1999) and showed that the same concentration of HCl and H_2SO_4 in the presence of 100% excess alcohol decreases the viscosity. H_2SO_4 has superior catalytic activity in the range of 1.5-2.25 M concentration. In additional to conventional catalysts, enzymatic catalysts like lipases are suitable substitutes for catalyzing the transesterification of triglycerides (Dorado et al, 2002). However the cost of a lipase catalyst is significantly greater than that of an alkaline one. The transesterification reaction of Jatropha curcas oil using a sodium hydroxide catalyst at 105°C temperature, 250 rpm and molar ratio of Butanol to Jatropha curcas oil (11:1) in batch reactor was also studied (Sohpal et al 2011). Fuzzy models were developed using adaptive neurofuzzy inference systems to evaluate and compare the results (Sohpal et al 2011). The response surface methodology (RSM) can also be used to determine the optimum condition for the transesterification reaction (Pinzi et al 2010). FWM and WDM was analyzed using ANFIS and comparative analysis, showing that the ANFIS approach was close to the real situation (Amarpal et al, 2009).This demonstrates the possible use of a relatively new soft computing technique called adaptive neuro-fuzzy inference system (ANFIS) for predicting uniaxial compressive Strength (UCS) of granites (Yesiloglu-Gultekin et al, 2012).

The present paper aims to optimize the amount of catalyst (NaOH) used in commercial scale, for techno-economy viability of biodiesel production by introducing the fuzzy model. In this study other process parameters (temperature, rpm and molar ratio of Butanol to Jatropha curcas oil) were fixed while catalyst wt and reaction time were two independent variables.

2. Fuzzy modeling of batch reactor based on ANFIS

The modeling of batch reactor for transesterification has been ANFIS, considering input parameters such as weight of the catalyst, time for reaction, and output as esters of butyl (%w/w). In ANFIS, the parameters associated with the membership functions change through the learning process. The computation of these parameters is facilitated by the gradient vector, which provides a measure of how well the fuzzy inference system (FIS) is modeling the input and output data for a given set of parameters. Once the gradient vector is obtained, any of the various optimization routines can be applied in order to accommodate the parameters so as to minimize some measure of error. Some data points are located and have been used as an input for the training of the FIS.

2.1. Architecture of the ANFIS

The fuzzy logic approach has the potential to produce a simplified control for various chemical engineering applications. The rule-based features of fuzzy models allow for a model interpretation in a way that is similar to the one humans use for describing reality. Conventional methods for statistical validation based on numerical data can be complemented by human knowledge which usually involves heuristic knowledge and intuition. A multi-input single output (MISO) fuzzy model of batch reactor for transesterification has been developed using ANFIS by considering two input parameters and one output variable in order to predict the product concentration. This technique provides a method for the fuzzy modeling method to understand information about a data set, in order to evaluate the membership function parameters that best provide the corresponding FIS to track the given input/output data. This learning process works similarly to that of neural networks. The parameters associated with the membership functions will change through the learning process. This method is based on the Sugeno-type fuzzy interface system and can simulate and analyze the mapping relation between the input and output data through hybrid learning to determine the optimal allocation of membership functions. The ANFIS architecture of the type from Takagi and Sugeno has been shown in Figure 1.

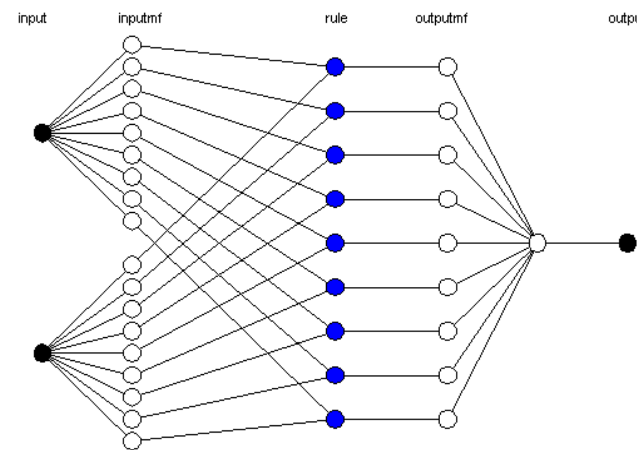

Fig.1. ANFIS Model with sub clustering

This interference system is composed of five layers. Each layer involves several nodes, which are described by the node function. The output signals from the nodes in the previous layers will be accepted as the input signals in the present layer. After manipulation by the node function in the present layer, the output will serve as the input signal for the next layer. To simply explain the mechanism of the ANFIS, we consider two inputs, x and y, and one output f in the FIS. Hence, the base rules will be fuzzy "if-then" rules as follows:

Rule 1: If x is A1 and y is B1, then f = p1x + q1y + r1

Rule 2: If x is A2 and y is B2, then f = p2x + q2y + r2

2.2. Fuzzy Interference System (FIS)

The core of a fuzzy logic controller/modeling is the inference engine, which contains information of the control strategy in the form of "if-then" rules. Since the fuzzy logic, rules require linguistic variables. Inputs and outputs of a process are generally continuous crisp values, therefore the conversion of crisp values into fuzzy values and vice versa are required. The initial step of the fuzzy modeling approach is to determine the input and output variables of the fuzzy logic controller. Sugeno type FIS is used for this purpose. A typical direct fuzzy logic control system is shown in Fig. 2. The ANFIS editor is used to create, train, and assess the Sugeno fuzzy logic. This FIS system is designed for the MISO system. The MISO system includes two inputs and one output.

2.3. Identification of input and output variables

The fuzzy logic is based on the identification of the fuzzy set that represents the possible values of the variables. In Figure 2, a block diagram of the fuzzy control process along with the physical system of the batch reactor for transesterification has been shown. In particular, Figure 3 shows real inputs and real outputs. The fuzzy model described in this article is a MISO

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system with two input parameters of catalyst weight, and chemical reaction time as well as the output parameter of butyl ester production.

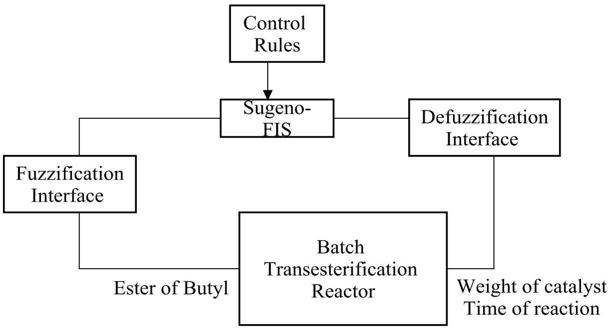

Fig.2. Block diagram for fuzzy control system of batch transesterification reactor.

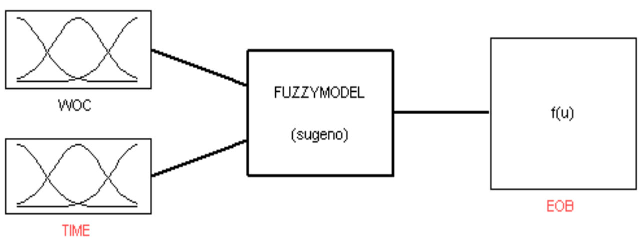

Fig.3. Fuzzy model of batch transesterification reactor showing inputs and output.

The possible universe of discourse for the input parameters has been given below:

Input parameters:

Weight of Catalyst (NaOH) = 20 gm to 40 gm
Time for chemical reaction (min) = 20 min to 60 min
Output parameter:
Ester of butyl (Product) = Predicted as %w/w
(Depending upon input parameters)

Membership Functions for the Input and Output Variables for ANFIS Modeling with sub-clustering and without sub-clustering.

In this process linguistic values were assigned to the variables using fuzzy subsets and their associated membership functions. A membership function assigns numbers between 0 and 1. Zero membership value means a non-member of the fuzzy set, while one represents full membership. A membership function can have any shape but the standard shapes for the membership function include trapezoids, triangles and bell shapes. Modeling without sub-clustering involves three membership functions that are produced for each input variable of catalyst weight (WOC), and reaction time based on the ANFIS. The in1mf1, in1mf2, in1mf3 are three linguistic levels for the amount of catalyst and in2mf1, in2mf2, in2mf3 are for reaction time which have been illustrated in Figures 4 (a), 4(b) and 4(c).

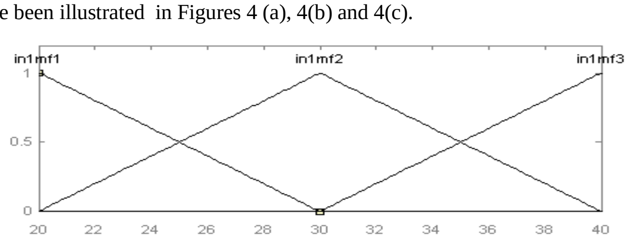

Fig. 4. (a) Membership function plots of input variable weight of catalyst.

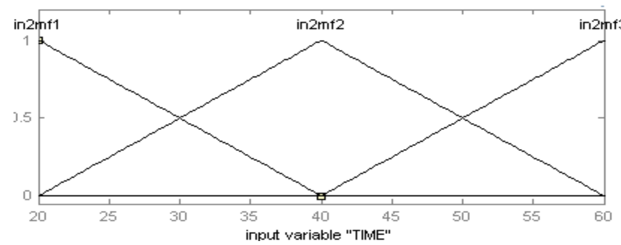

Fig. 4.(b) Membership function plots of input variable time of reaction.

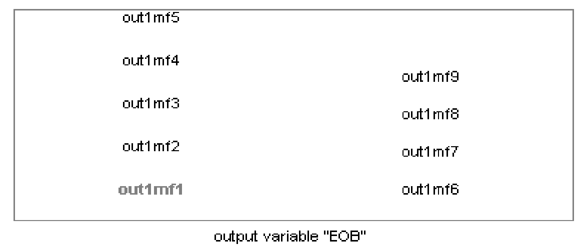

Fig.4. (c) Membership function plots of output variable ester of butyl.

On the other hand, in ANFIS modeling with sub-clustering, nine membership functions were generated for input variables of catalyst weight (WOC), and nine membership functions for reaction time based on the ANFIS. The in1cluster1, in1cluster2, in1cluster3, in1cluster4, in1cluster5, in1cluster6, in1cluster7, in1cluster8, in1cluster9 are nine linguistic levels for the catalyst weight variable and in2cluster1, in2cluster2, in2cluster3, in2cluster4, in2cluster5, in2cluster6, in2cluster7, in2cluster8, in2cluster9 in2mf1, in2mf2, in2mf3, in2mf4 are reaction time variables over a given universe of discourse as shown in Figures 5 (a) and 5(b). The output variable of butyl ester production (EOB) also has four membership functions as shown in Figure 5(c). The span of each function has been tuned within the specified range. Tests were conducted to evaluate the response parameters, and the span was varied accordingly for improvement. After a few iterations, the final membership functions for the system were determined as shown in the respective figures.

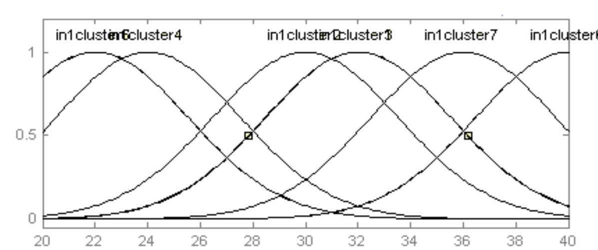

Fig.5. (a) Membership function plots of input variable weight of catalyst.

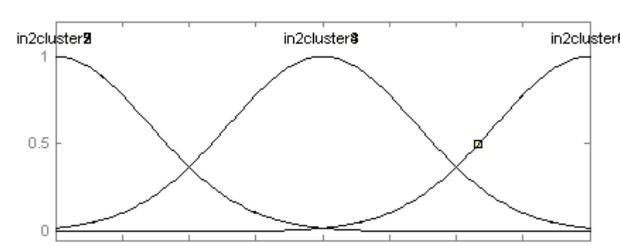

Fig.5. (b) Membership function plots of input variable time of reaction.

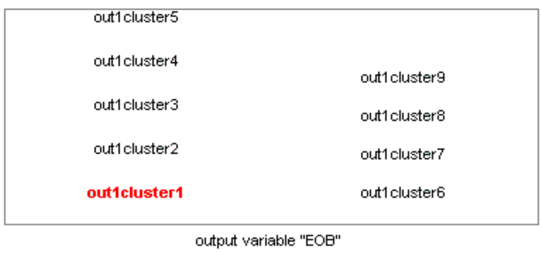

Fig.5. (c) Membership function plots of output variable ester of butyl.

2.4. FIS Rules Employed in the Model

The fuzzy model of the batch transesterification has been used to design the MISO fuzzy model with two inputs, i.e., weight of catalyst and time for reaction , each of which were determined for 3 linguistic variables using the ANFIS modeling method without sub-clustering. These variables generated 9 numbers of conditional statements as “if-and-then” rules of the model. The formulated set of rules of the model has been outlined below:

1. If (WOC is in1mf1) and (Time is in2mf1) then (EOB is out1mf1) (1)
2. If (WOC is in1mf1) and (Time is in2mf2) then (EOB is out1mf2) (1)
3. If (WOC is in1mf1) and (Time is in2mf3) then (EOB is out1mf3) (1)
4. If (WOC is in1mf2) and (Time is in2mf1) then (EOB is out1mf4) (1)
5.1f (WOC is in1mf2) and (Time is in2mf2) then (EOB is out1mf5) (1)
6. If (WOC is in1mf2) and (Time is in2mf3) then (EOB is out1mf6) (1)
7. If (WOC is in1mf3) and (Time is in2mf1) then (EOB is out1mf7) (1)
8. If (WOC is in1mf3) and (Time is in2mf2) then (EOB is out1mf8) (1)
9. If (WOC is in1mf3) and (Time is in2mf3) then (EOB is out1mf9) (1)

Similarly the weight of the catalyst and the time for the reactions, were both variables that were also used for linguistic variables using ANFIS modeling with sub-clustering. These variables generated 9 numbers of conditional statements as “if-and-then” rules of the model. The formulated set of rules of the model has been outlined below:

1. If (WOC is in1cluster1) and (Time is in2cluster1) then (EOB is out1cluster1) (1)
2. If (WOC is in1cluster2) and (Time is in2cluster2) then (EOB is out1cluster2) (1)
3. If (WOC is in1cluster3) and (Time is in2cluster3) then (EOB is out1cluster3) (1)
4. If (WOC is in1cluster4) and (Time is in2cluster4) then (EOB is out1cluster4) (1)
5.1f (WOC is in1cluster5) and (Time is in2cluster5) then (EOB is out1cluster5) (1)
6. If (WOC is in1cluster6) and (Time is in2cluster6) then (EOB is out1cluster6) (1)
7. If (WOC is in1cluster7) and (Time is in2cluster7) then (EOB is out1cluster7) (1)
8. If (WOC is in1cluster8) and (Time is in2cluster8) then (EOB is out1cluster8) (1)
9. If (WOC is in1cluster9) and (Time is in2cluster9) then (EOB is out1cluster9) (1)

Figure 6 (a) and 6 (b) indicates rule viewer that shows the values of the various input to the model and computed outputs.

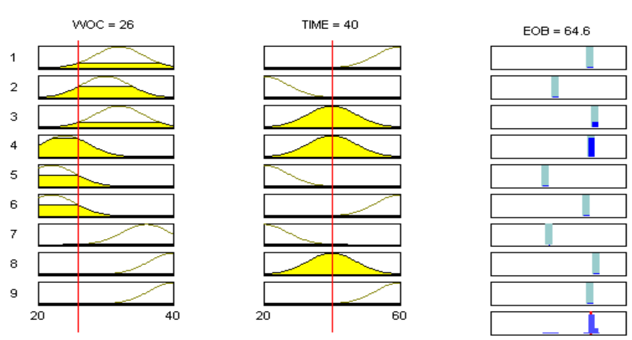

Fig.6. (a) Ruler view of input variable (weight of catalyst and time of reaction) with output variable ester of butyl using without sub clustering.

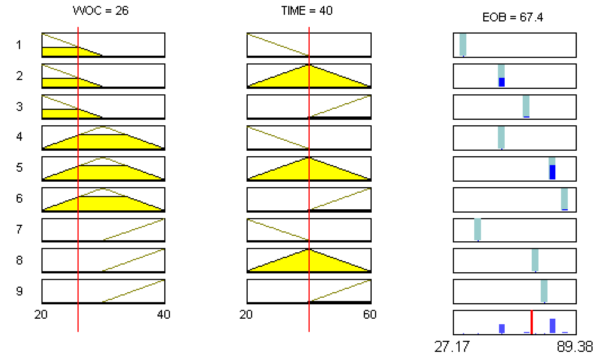

Fig.6. (b) Ruler view of input variable (weight of catalyst and time of reaction) with output variable ester of butyl using with sub clustering.

Here the ester of butyl (output) can be predicted by varying the input parameters weight of catalyst and time for chemical reaction. Figure 6 shows a particular instance with sub clustering having input values given to the system 26 gm for weight of catalyst, and 40 min for time for chemical reaction. The output generated by the system for ester of butyl production is shown as 64.6 %w/w.

A similar output was generated with sub clustering having the ester of butyl concentration 67.4 %w/w. Likewise; this fuzzy model has generated other values of output variable for different sets of data points in the specified range of input variables. Figure 7(a) and (b) show two different views of control surfaces, which indicate the results predicted by the fuzzy model for different sets of data points.

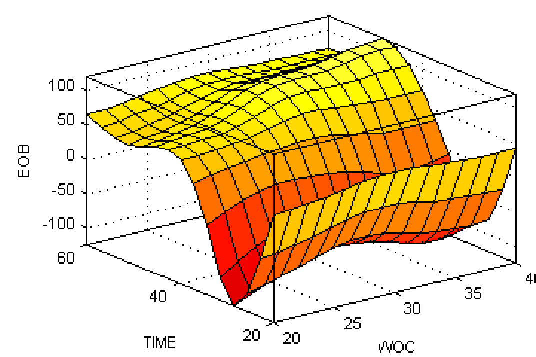

Fig.7. (a) and 7(b) having two different views of control surface of the fuzzy model. (a) Control surface view of the fuzzy model with sub clustering.

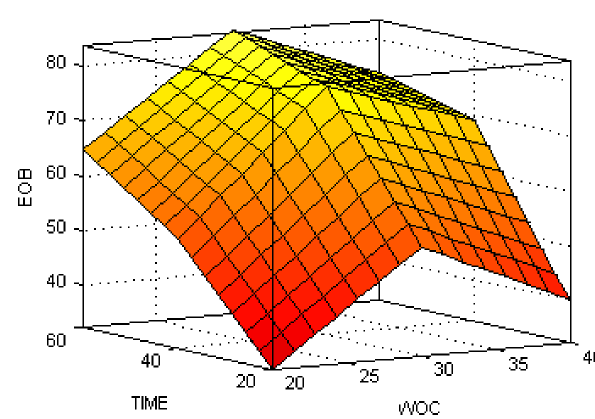

Fig.7. (b) Control surface view of the fuzzy model without sub clustering.

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These control surfaces have been shown given the interdependency of input and output parameters guided by the various rules in the given universe of discourse. It has already been finalized that there are nine rules predicting the ester of butyl production depending upon the input parameters, weight of catalyst, and time of reaction, for MISO fuzzy model. These rules have been implemented in the MATLAB environment using the Sugeno type of FIS in fuzzy logic toolbox. The results predicted from this fuzzy model of batch transesterification of Jatropha curcas have been compared with the experimental results for its validation in the latter part of the article.

3. Experimental Setup & Methodology

Figure 8 shows the batch transesterification reaction system used in the experiments to validate the results of the designed fuzzy model.

A 1500 ml glass reactor equipped with control driven mechanical stirrer, thermocouple, condensing coil and sample port have been used in all kinetic experiments. The thermocouple was immersed in constant volume batch reactor, which was capable of controlling the temperature to within deviation of ±0.50C .A mechanical stirrer fitted with stainless steel propeller provided the mixing requirement. Thirty reactions were carried out over the entire duration of experimental work. The conditions of temperature, molar ratio and mixing intensity were fixed, where catalyst weight and time of reaction were considered as independent variable.

Fig.8. Batch transesterification reactor.

(i) The reactor was initially charged with Jatropha oil depending upon the required molar ratio of oil to Butanol. The reactor was then placed under constant heat to reach the desired temperature. (ii) A measured amount of Butanol and sodium hydroxide stock solution, were separately heated to the reaction temperature, and then added to the reactor. A mechanical stirrer was used as per required temperature. The reaction was timed as soon as the mechanical stirrer was turned on. (iii) During the experiment the samples were taken at 20 minute intervals. Approximately 20 to 25 samples were collected during the course of each reaction (60minutes). (iv) Samples were collected in 10ml test tubes filled with 4ml of distilled water. The test tubes were kept in an ice bath at about 50C prior to use. (v) Samples of (2ml) were quenched in the test tubes by being immediately placed in ice baths following their removal from the glass reactor. The test tubes were then shaken to stop the reactions. (vi) After measuring their residual weight, the upper and the lower portions were analyzed for the composition by using gas liquid chromatography (GLC).

4. Results and Discussions

A study of literature reveals that alkali metal alkoxides are found to be more effective transesterification catalysts compared to acidic catalysts. So the base catalyst (NaOH) was used for transesterification of Jatropha oil in the present work. The reaction was carried out at different catalyst weights, ranging from 20-40 gm in the presence of excess alcohol, results of which have been shown in Figure 9. It can be seen that the ester of butyl production through transesterification (EOB) of Jatropha oil with Butanol is approximately a logarithmic function of catalyst up to 30gm.

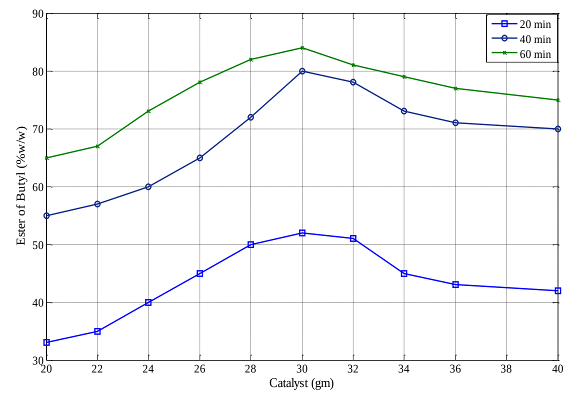

Fig.9. Output of product (EOB) versus catalyst under different reaction time in batch transesterification reactor.

The formation of 65 (%w/w) butyl ester was found to take 60 minutes. On increasing the weight of the catalyst a maximum of up to 30 gm of catalyst and 85% of butyl ester formation was observed. However on further increasing the amount of catalyst, soap was formed, thus reducing the transesterification rate. Moreover in all reaction times studied, 30 gm of catalyst was found to give the best and optimum result.

Figure 10 (a), 10(b) and 10 (c) compares the predicted production of ester of butyl using the fuzzy model and the data reported for batch transesterification reactor experiments. Figure 10 (a) shows the plot between productions of ester of butyl and time of reaction (20 minutes) and Fig. 10(b) shows the plot between the production of ester of butyl and time of reaction (40 minutes), and Fig. 10 (c) shows the plot between production of ester of butyl and time of reaction (60 minutes).

An increase in the production rate of the butyl ester, in the initial stages of the curve, along with the increase in time is due to more residence time. After that, the decrease in the ester production rate caused by an increase in time and weight of the catalyst, is due to soap formation and hinders the reaction. This trend seen in rate of butyl ester production is closely followed by the outcome of the designed fuzzy model. Out of the various outputs generated by the fuzzy model, only 2% of the data cross the experimental results, when the FIS system is used without sub clustering. FIS with sub clustering has only 0.1% point deviate from the experimental analysis. With the average error being 0.055%, the mean accuracy of the model comes out to be 99.95%. In the present study, the total number of data points involved was 90. Thus, it can be concluded that there is a close relationship between the simulated results and the practical results obtained at similar reactor conditions for predicting butyl ester production as shown in Figures 10 (a), 10(b) and 10 (c).

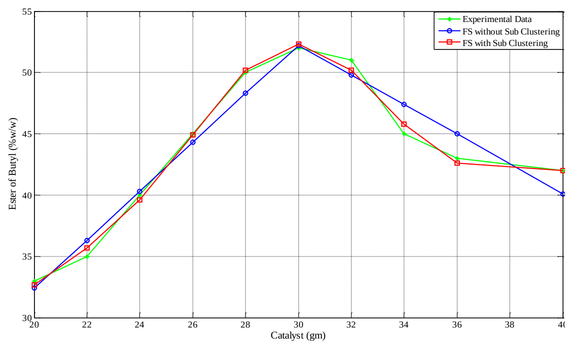

Fig.10. (a) Output of product (EOB) versus catalyst at the reaction time of 20 min in batch transesterification reactor.

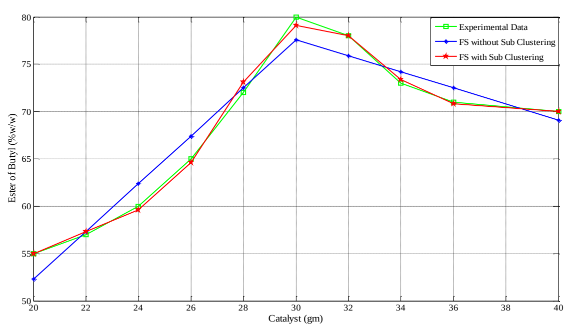

Fig.10. (b) Output of product (EOB) versus catalyst at the reaction time of 40 min in batch transesterification reactor.

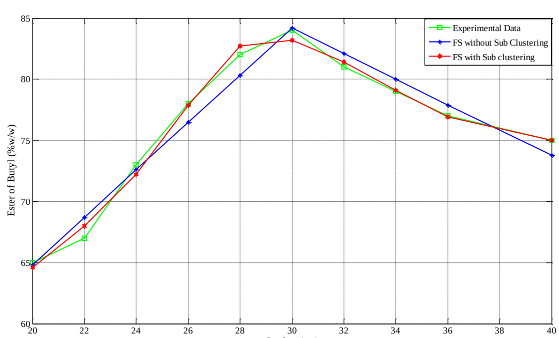

Fig.10. (c) Output of product (EOB) versus catalyst at the reaction time of 60 min in batch transesterification reactor.

4.1 Kolmogorv-Smirnov (K-S) test for significance of the ANFIS model

The difference between the experimental and theoretical values of the fuzzy model can be evaluated using the K-S test of statistical methods. This test is one of the most valuable non parametric statical methods, for comparing two samples, as it is sensitive to differences in both location and shape of the empirical cumulative distribution functions of the two samples. The calculated value of maximum difference between the cumulative distributions (D), probabilities (P) and significance confidence level limits above 95% have been listed in Table 1. Since the calculated values of D and its corresponding P values are around 0.985 to 0.996, there is no significant difference between the ester of butyl production values generated by the fuzzy model and experimental values under null hypothesis.

Table 1
Kolmorgov-Smirnov (K-S) test output for experimental data/ FIS models.

K-S test	Parameters	20 minutes	40 minutes	60 minutes
Experiment Data/ FIS without sub clustering	D	0.1818	0.1818	0.1591
	P	0.985	0.985	0.996
	>95 Confidence (EOB % w/w)	42.34 through 52.45	71.54 through 73.65	78.10 through 84.40
Experiment Data/ FIS with sub clustering	D	0.1818	0.1818	0.1591
	P	0.985	0.985	0.996
	>95 Confidence (EOB % w/w)	50.24 through 52.45	75.54 through 79.75	79.32 through 83.69

Moreover the difference between the ANFIS models (with and without sub clustering) have also evaluated using K-S test. It has been found that most of the data spread into a large fraction of the plot. This is a sign of a normal distribution of the data. The KS test has also found the data to be consistent with a log normal distribution P= 0.90 and normal distribution: P= 0.95. Thus making the maximum difference in the cumulative fraction is D=.0909 as shown in Figure 11. Hence the probability of generating accurate values for the rate ester of butyl production by using the fuzzy model is 99.90%.

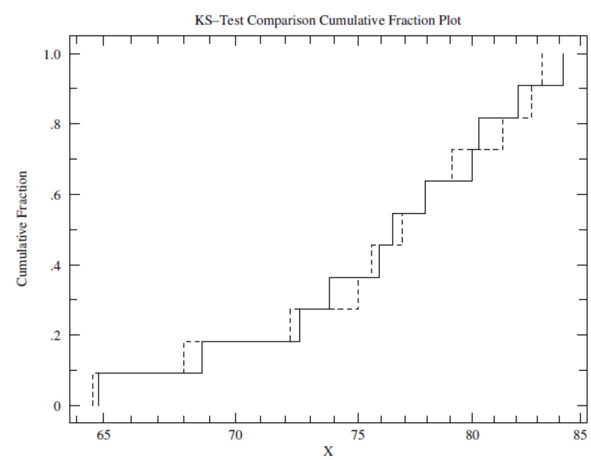

Fig.11. Cumulative fraction plot of FIS model with sub-clustering and without sub-clustering for 60 minutes operation.

5. Conclusions

This paper deals with the application of the fuzzy logic for predicting the production rate of the ester of butyl in the batch transesterification reactor. In this article, the MISO fuzzy model is developed using ANFIS and validated through experimental results for given conditions. With more than 99% average accuracy, it has been found that the results generated by the designed fuzzy models are close to the experimental results. From statistical analysis, it has been concluded that the maximum differences between the cumulative distributions (D) is in the range of 0.1591 to 0.1818 in comparison to the experimental data obtained using FIS. With this low deviation, the accuracy of the model can be used by the process engineer who would like to get quick answers for online intelligent control and/or optimization. The optimum amount of catalysts required in this particular reaction is 28.5-30 gm, and in its current state, the model is limited to the amount of catalyst and reaction time. This study supports the idea that the fuzzy logic technique can be used as a viable alternative for carrying out analysis. Moreover, the Fuzzy logic allows for the modeling and control problem to be treated simultaneously.

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Please cite this article as: Sohpal, V.K., Singh, A . Optimization of alkali catalyst for transesterification of jatropha curcus using adaptive neuro-fuzzy modeling. Biofuel Research Journal 2 (2014) 70-76.