

Transesterification Optimization using Calcium Oxide from Karanja Oil and Results Validation by ANN

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Satish A Patil^{*} , Dr. Racyya. R. Arakerimath^{}**

^{*} Research Scholar, D Y Patil Institute of Technology, Pune (MS), India,
(sapcoeh@rediffmail.com).

^{**} HOD Mechanical, G H Rasoni College of Engineering and Management, Wagholi, Pune.
Savitribai Phule Pune University, Pune, India. (rachayya.arakerimath@raisoni.net).

*Email: sapcoeh@rediffmail.com; Corresponding Author.

ABSTRACT

Biodiesel is obtained using the transesterification process from renewable oils obtained from vegetable and animal fats. The transesterification process is used to produce biodiesel from Karanja oil with heterogeneous catalyst Calcium Oxide (CaO). In this research work, the Taguchi method has used for the optimization of the transesterification process using five input parameters and five levels for the development of orthogonal arrays. Experiments have conducted as per the L25 orthogonal array developed by Taguchi and yields obtained have been noted. The results obtained by experimentation have been analyzed by Minitab software. The results from Minitab have compared with the results obtained using ANN script analytically as well as graphically. The maximum value of yield has 88% at optimum parametric value namely molar ratio 20% with the addition of 3% Calcium oxide catalyst at process temperature 65°C for 60 minutes reaction time and agitation speed 600 rpm.

Keywords: Biodiesel, Transesterification, Catalyst, Calcium, Heterogeneous.

INTRODUCTION

Petrodiesel fuel is diminishing every day. One of the feasible ways to control petrodiesel scarcity and environmental complications is biodiesel. Biodiesel is obtained using the transesterification process from renewable oils obtained from vegetable and animal fats using alcohol (Mojtaba Mansourpoor et al. 2012). Generally, resources as non-edible oils have focused on. The properties like highly degradable, nontoxic, less CO emission, unburned hydrocarbons, etc. the biodiesel have focused as a substitute to diesel fuel. The existing compression engine can be used with no alterations with biodiesel. The Transesterification process is one of the best methods for biodiesel synthesis from vegetable oils. There is a need for optimization of this process using a heterogeneous catalyst (Satish A. Patil et al.2019). To find out the best heterogeneous catalyst and also the development of mathematical models and validation of results using the software are the main objectives of this study. So, the study of optimization of Transesterification has done for three different heterogeneous catalysts separately from Karanja oil. This study has helped to find out the heterogeneous catalyst which is the best for the production of biodiesel. The optimization results of transesterification using heterogeneous catalyst Calcium oxide have discussed in this paper (N. Viriya-empikul et al. 2012).

Twenty-five numbers of experiments have done as per L25 orthogonal array developed by Minitab using the given parametric conditions namely the molar ratio (MR) with catalyst concentration (CC) maintaining process temperature (PT) and time required (RT) controlling stirring (agitating) speed (SS) (Mojtaba Mansourpoor et al.2012, Satish A. Patil et al.2019).). The Karanja oil yield values obtained through these experimentations have been noted. Results have

been analyzed by the Taguchi method for input parametric optimization for biodiesel production from Karanja oil with Calcium Oxide (CaO) as a heterogeneous catalyst (N. Viriya-empikul et al. 2012). The graphs namely main effect plots, interaction plots, regression plots, and mathematical models have obtained during the analysis. The ANN script has been written for obtaining results. The yield values obtained by Taguchi array experimentation and values obtained by ANN have compared here. The graphical and analytical comparative analysis has been done. The results obtained by Taguchi method experimentation have been validated by ANN.

Karanja: Karanja tree grows naturally in the arid area of India. These trees have very deep roots to reach water having considerable medium size. It can grow up to the height of 15-25 meters above the ground. This crop is commercialized by India for its poor farmers which are part of a large population of rural areas. These trees are very hard, good drought-resistant, and tolerant to salinity. The tree gives green pods within 10 months having a tan colour (Bobade S N et al 2012). These pods are 5 to 7 cm long flat of elliptic shape and have two kidney-shaped kernels with have a brownish-red colour. The kernel yield obtained is in between 8 to 24 kg per tree. The content of oil in these seeds is in the range of 27%- 39% (Bobade S N et al. 2012).

Heterogeneous Catalyst: The separation of heterogeneous catalysts can do by filtration from the final products of the reaction and they can be reused. Due to the reuse of heterogeneous catalysts, there is less consumption of chemicals and less time of course. Due to having high selectivity, they are separate out easily from the reaction products (N. Viriya-empikul et al. 2012). The contributing role of heterogeneous catalysts is very important. So, they are having a scope in the field of biodiesel production in the future. This heterogeneous catalysis causes less corrosion and it is environmentally suitable, biodegradable, easy recovery of catalyst, and is

having high process integrity (Sani J. et al. 2017) So, heterogeneous catalysts are superior to those of homogeneous catalysis at all levels. Some examples of heterogeneous basic catalysts are oxides of Magnesium, Aluminum, and Calcium, etc. These heterogeneous catalysts are commercially used especially in biodiesel industries. These catalysts are prepared from waste materials at a nominal cost. The interesting area of research is to find such waste materials because of their wide source and low cost. These eco-friendly waste catalysts are having other applications in some organic reactions in addition to the production of biodiesel (N. Viriya-empikul et al. 2012, Sani J. et al.2017).

LITERATURE SURVEY

Some of the previous studies related to this investigation are discussed in the following sections. The different research works done by the authors and has published in their papers are given below in short.

S. Hawash et al. have studied the different parameters namely molar ratio i.e. methanol to oil ratio, the time required for the reaction, and mass percentage of catalyst to oil which are affecting the transesterification process on small scale basis using base catalysts as CaO and acidic catalyst. It has been proved that by the supercritical process of transesterification the obtained yield has more than 95%.

N. Viriya-empikul et al. have produced the biodiesel using a Ca-based solid catalyst which has obtained from industrial wastage. In the research work, the solid catalyst has been obtained from three different raw materials. The physical and catalytic properties of the catalyst have investigated. The heterogeneous CaO catalyst has achieved from eggshells, golden apple snail shells. The biodiesel production using this heterogeneous catalyst by transesterification process has been achieved.

Sani J et al. have used a heterogeneous catalyst obtained from the waste shell of a snail and determined its efficiency for biodiesel production by a transesterification reaction. The research has been proved the successful use of waste snail shells to obtain heterogeneous catalysts for prospective economics of heterogeneous catalysts.

Mojtaba Mansourpoor and Dr. Ahmad Shariati have given the Response Surface Methodology with five levels and with three variables. The developed design has been used for finding the parameters affecting the interaction effects on transesterification reaction. These parameters are the temperature of the reaction, catalyst percentage, and methanol to oil molar ratio on yield of biodiesel.

Nimesh G. Gandhi, Mr. Hardik M. Patel have studied the effect on 4-stroke 4-cylinder diesel engine injection pressure for fewer emissions by the Taguchi method. The optimization of injection pressure was from 100 to 250 bar of diesel engine. The increment of 50 bars has been made by atomizer adjustment to increase the performance of the engine. The parameters such as power, torque, and specific fuel consumption have been measured for the minimization of the engine emissions by the Taguchi method.

Anant Bhaskar Garg et al. have presented the research work in the field of engine development and took an overview of the use of artificial neural networks (ANN) in it. By using ANN in various approaches they have highlighted better results in engine operations modelling. They have proved that the ANN can be used to reduce the time required for engine development. They have also discussed the ANN approach, algorithms, and the importance of architecture in this paper. This paper is useful in advancing ANN research.

EXPERIMENTAL SETUP

The experimental setup reactor has a 1000 ml four-necked spherical flask kept in a heating mantle with a thermo-controller. The condenser has equipped with one side neck and thermowell in the other. For temperature measurement, a thermometer has placed in the thermowell containing glycerol (Bobade S N et al 2012). The condenser has been used to prevent any reactant loss. A stirrer with a motor having a speed regulator has passed in the central neck as shown in Figure1. The specifications of the setup are given below. The procedure of conduction of the transesterification process to obtain biodiesel is explained in short.

First, measure one litre of Karanja oil in the measuring flask and put it into the round bottom flask and set temperature 65-70 °C, and continue heating. If free fatty acid (FFA) percentage is more than 5%, esterification of that oil is necessary otherwise use directly for transesterification. In the transesterification process add the catalyst in methanol and then pour it into the round bottom flask having hot vegetable oil. Continue stirring of the mixture by stirrer with stirring speed up to 600 rpm for one hour approximately (Bobade S N et al 2012).

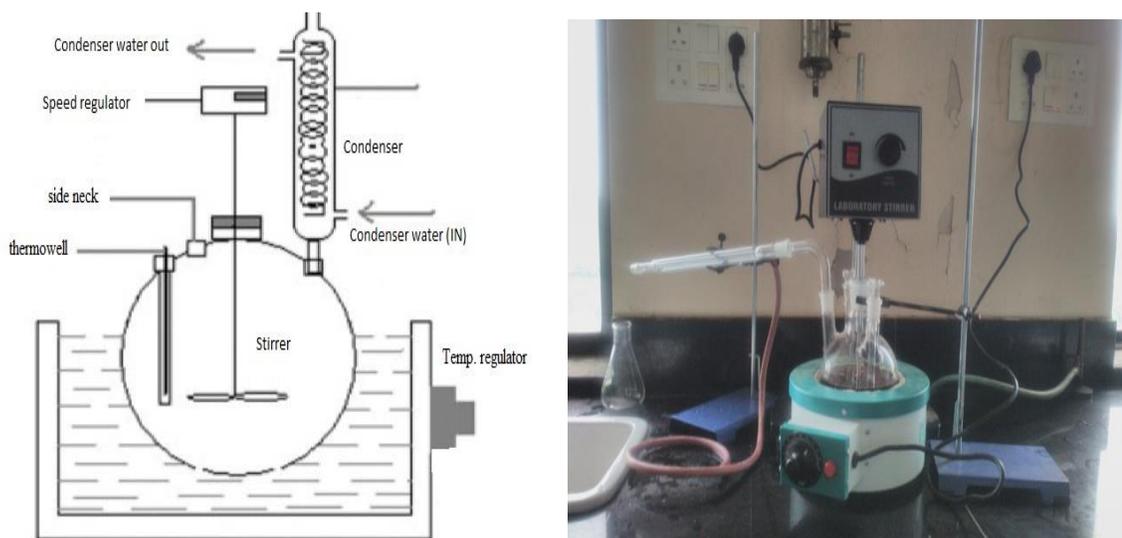


Figure 1: Transesterification process setup sketch and actual Photograph

The specifications of the setup are as follows:

1. Heating Mantles 1 lit (Rating watts-300)
2. Mechanical stirrer with regulator (0 to 100 rpm)
3. Burette stands complete set - 2Nos.
4. Four necks round bottom flask Borosilicate 1 lit capacity.
5. Condenser 300mm, Socket size-24mm.
6. Thermometers 0-100 °C

After completion of the transesterification reaction, the reaction mixture has taken in a separating funnel and has allowed settling for 8 hours. After settling for 8 hours glycerol and methyl esters have been separated manually. The products of the transesterification process namely Karanja biodiesel have been settled at the upper layer and denser glycerol at the lower layer. The unused methanol and catalyst have been collected below the glycerol layer (Bobade S N et al 2012). The results have proved that biodiesel obtained using heterogeneous Calcium oxide (CaO) catalyst has countable potential for biodiesel production (N. Viriya et al.2012).

OPERATING CONDITIONS

The input parameters responsible for the production (transesterification) process for biodiesel with heterogeneous catalysts using feedstock Karanja have as follows (Satish A. Patil et al.2019).

- 1) Mole Ratio %. (MR)
- 2) Amount of catalyst %. (CC)
- 3) Reaction or Process Temperature. (PT)
- 4) Stirring speed. (SS)
- 5) Reaction time. (RT)

The range values of all parameters and levels have given in the Table1 as below:

Table 1. Ranges of values of the parameters

A: MR %	B: CC %	C: PT °C	D: RT min	E: SS rpm
A1 = 5	B1 = 1.0	C1 = 55°C	D1 = 60 min.	E1 = 300rpm
A2 = 10	B2 = 1.5	C2 = 60 °C	D2 = 90 min.	E2 = 400 rpm
A3 = 15	B3 = 2.0	C3 = 65°C	D3 = 120 min.	E3 = 500 rpm
A4 = 20	B4 = 2.5	C4 = 67°C	D4 = 150 min.	E4 = 600 rpm
A5 = 25	B5 = 3.0	C5 = 70 °C	D5 = 180 min.	E5 = 700 rpm

TAGUCHI DESIGN

There were five input parameters and five levels have taken for the development of orthogonal arrays (Nimesh G. Gandhi et al.2014, Avinash et al. 2015). In the Minitab software, the Taguchi method has used for analysis. The designed summary and obtained array have as follows.

Designed Summary

Taguchi Array L25 (5⁵)

No. of Factors: - Five

No. of Runs: - Twenty five

Columns of L25 (5⁶) array:- 1 2 3 4 5

Twenty-five transesterification process experiments as per L25 orthogonal array have carried out to produce biodiesel from Karanja oil using methyl alcohol for different reaction combinations of input parameters and yields obtained have been noted (Nimesh G. Gandhi et al.2014, S. Hawash et al. 2011) and given in Table 2 as below.

Table 2. Sample readings from the Experimentation array by Taguchi

MR %	CC%	PT.	RT (min)	SS (rpm)	Yield %	SNR	SNRES
5	1	55	60	300	54	34.64788	0.133598
15	1.5	67	60	500	69	36.77698	3.219887
20	2.5	60	180	500	84	38.48559	1.131218
20	3	65	60	600	88	38.88965	1.068839
25	1	70	150	500	69	36.77698	-0.01354

25	1.5	55	180	600	77	37.72981	0.413598
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In this Table 2, the yellow-coloured row has given the highest value of SN ratio, which has given optimum input values of parameters for maximized yield (Satish A. Patil et al.2019). Here for yield SNR- Higher is better. This ratio indicates how the extent of the response varies relative to the target value with other noise conditions. The experimental output yield has a maximum value of 88% for optimal conditions of input parameters molar ratio 20%, catalyst 3%, reaction temperature 65°C, the time required 60 minutes and speed 600 rpm.

MODEL SUMMARY

The model summary obtained has shown below in Table 3. The R square value obtained is 91.26% which gives how extensively a model is fitted with data (Satish A. Patil et al.2019).

Table 3. Model Summary

S	R square	R sque.(adjusted)	R sque(predict)
3.62218	91.26%	88.97%	84.86%

REGRESSION EQUATION

The regression equation (1) obtained has given below (Satish A. Patil et al.2019).

$$\text{Yield \%} = 47.84 + 0.904 \text{ MR \%} + 11.16 \text{ CC \%} - 0.156 \text{ PT } ^\circ\text{C} + 0.0027 \text{ RT (min)} - 0.00420 \text{ SS (rpm)} \dots\dots\dots (1)$$

VALIDATION OF EXPERIMENTAL RESULTS BY ANN

Artificial neural networks are among the most powerful learning models. They are recently used in various branches of engineering widely. As the mathematical models developed, the optimization of calibration maps can be done by using ANN techniques. The complex functions can be performed by neural networks in various applications in different fields. Now neural

networks can be used to solve problems that are difficult for computers or human beings (Anant Bhaskar Garg et al.2012). An ANN is an information processing paradigm. This is inspired by the way biological nervous systems like the brain process information. It is important in accessing the algorithm which is suitable for a particular situation (Anant Bhaskar Garg et al.).

The use of ANN script is given in short. Start the Neural Network by start command and click on pattern reorganization tool. Click next in welcome screen and go to select data for input and for targets. Click next and go to validation and test data. See the actual output from the network. This output shows the probability for possible label. See the most probable label and compare it to the actual.

ANN Script for the Analysis performed

```
clc; close all; clear all;

x = xlsread('Input1');

y = xlsread('Output2');

net = new ff(minmax(x), [20,1], {'logsig','purelin','trainlm'});

net.trainparam.epochs = 1000;

net.trainparam.goal = 1e-15;

net.trainparam.lr = 0.01;

net = train(net, x, y);

y_net = net(x);

plot(y);hold on; plot(y_net, 'r');

error = (y - y_net);
```

The yield values obtained experimentally, by ANN and % error are as given in Table 4.

Table 4. Sample results for validation.

MR %	CC %	RT °C	PT (min)	SS (rpm)	Experimental Yield %	Yield % by ANN	Error	Error%
5	1	55	60	300	54	55.77	-1.77	-3%
5	1.5	60	90	400	55	54.25	0.75	1%
5	3	70	180	700	70	73.30	-3.30	-5%
10	1	60	120	600	56	58.74	-2.74	-5%
10	1.5	65	150	700	63	58.66	4.34	7%

COMPARATIVE STUDY OF YIELD BY EXPERIMENTATION AND ANN

The graphical comparison of yield obtained by experimentation and ANN has appeared in Figure 4. The values of yield obtained by experimentation, by ANN, the difference in these values, and also the % errors have given in Table 4. There is a very small difference between these values and % errors are also very small. So, results have been validated.

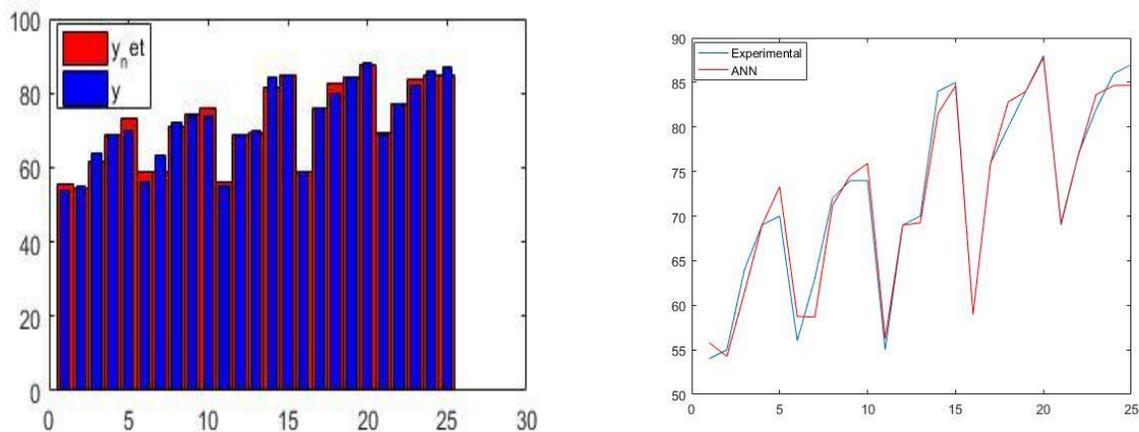


Figure 4. Comparative study of yield obtained by Experimentation and ANN

REGRESSION PLOTS

The plots obtained by Minitab and ANN are as below.

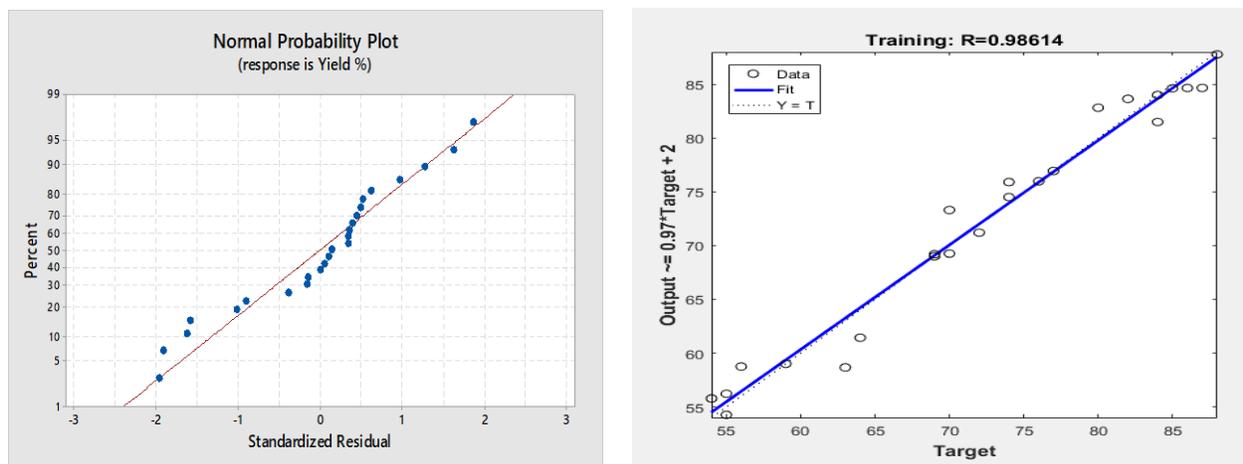


Figure 5. Comparison of plots by Minitab and by ANN

The regression plot obtained by ANN has compared with the Minitab regression plot as shown in Figure 5. The residual values obtained are along or on the line. The R-square value for this regression is 91.26%. There is a similarity in these plots, hence results have been validated.

CONCLUSIONS

Here the experimentation work has done for parametric optimization of production of Karanja biodiesel using heterogeneous (CaO) catalyst. The results by experimentation through Minitab have been validated by ANN (Artificial Neural Network), the conclusions are;

- 1) The experimental yield obtained has 88% with optimal input parametric conditions namely molar ratio 20% with the addition of 3% CaO catalyst at process temperature 65°C for 60 minutes of reaction time and required agitation speed 600 rpm.
- 2) Interaction plots have shown the higher SNR value at 3% catalyst and 20% molar ratio.
- 3) The R square value obtained has 91.26% which gives how extensively a model is fitted with the actual data
- 4) There are very small errors in between the values of yield obtained by experimentation and by ANN.

- 5) The regression plot obtained by Minitab is similar to the ANN regression plot.
- 6) In this way, various results obtained by Minitab have been validated by ANN.

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