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PARAMETRIC OPTIMIZATION OF BIODIESEL SYNTHESIS FROM RUBBER SEED OIL USING IRON DOPED CARBON CATALYST BY RESPONSE SURFACE METHODOLOGY

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ABSTRACT

The best way to meet the demand for traditional feedstock as an alternative sustainable energy source is by improvising biodiesel production by employing heterogeneous catalyst and its parametric optimization. The present work used RSM (response surface methodology) in conjunction with the CCD (central composite design) for optimizing the activity of catalysts for the biodiesel production. The optimized parameters are time, temperature and catalyst percentage and considered response factors are catalytic conversion and activity. Indigenously prepared iron doped catalyst (Fe/C) is used for the synthesis of biodiesel from rubber seed oil (RSO). The optimized condition obtained by maximizing the catalytic conversion to 51-97 and activity to 53.2-67.9 are time 45 minutes, temperature 85ºCand catalytic percentage 2.5%. The study reveals that both the amount of catalyst loaded on the support, temperature and the time had significant positive effects on the biodiesel yield. Hence, the investigation reached to a conclusion that the rubber seed oil could be an effective feedstock for the production of biodiesel using novel iron doped catalyst at optimized condition to synthesis energy efficient and cost effective Rubber seed oil methyl ester(RSME) towards the production of sustainable energy.

KEYWORDS: RSM (Response Surface Methodology), Rubber Seed Oil (RSO) & Sustainable Energy

INTRODUCTION

Due to the continuous depletion and growing harmful impacts on the environment by the conventional sources of energy, the works on biodiesel has gained lot of interest in the whole scientific community all over the world [1] because of its biodegradability, exceptional cetane numbers, high flash point, low viscosity, lower combustion emission profiles, ability to be blended with fossil-based diesel at any proportions and ability to be used in conventional diesel engines with no further modifications. Homogeneous acid and base catalysts such as potassium hydroxide, sodium hydroxide and sulphuric acid have been conventionally used for biodiesel production. The process is normally performed under mild conditions and produces high amount of methyl esters in a short reaction time due to the formation of two phases in the reaction medium. However, a purification step is required to remove the catalyst from products and this is difficult because of its solubility in the reaction medium. This process leads to generation of large amount of wastewater containing liquids of high basicity or acidity. So the researchers are paying wide attention on the utilization of vegetable oils or their conversion into esters towards their use as an energy source to cope with the depleting petroleum sources. The cost of the biodiesel is the major hurdle to-wards its use on a large scale, which depends mainly on the feed-stock and catalyst used in the reaction [2]. It is estimated that feedstock's alone contributes one third of the total biodiesel production cost [3]. Hence, for producing economically viable biodiesel selection of a suitable and cheap raw material is an important criterion.

The economic production of biodiesel is mainly dependent on the process and its constituents. Hence, estimation of the significant parameters affecting the biodiesel yield profoundly, and optimized value of each process parameter for yielding the maximum biodiesel, have become the important area of research nowadays. It is worthy to mention that there are many parameters affecting the biodiesel yield during the production process. The significance of each parameter and its These statistical techniques are accepted widely for the chemical processes as it considers many process parameters simultaneously to give the best optimized condition with a better regression mathematical model to predict the final performance characteristic [5]. The optimization and evaluation of the significant parameters of the production process are done by the response surface methodology (RSM) in Design Expert 10.0.

Rubber seeds are the important by-product of the rubber trees and treated as a waste. These seeds contain 40-50% oil and still underutilised in India. Thus, oil extracted from the rubber seeds can be used as a prominent feedstock for the production of biodiesel in developing countries like India, where almost 85% of the crude oil imports from the other countries [6]. Thus, conversion of abundant waste into the energy can lead to improving the economy of the developing countries like India and leads to its sustainable development. Hence, in the present study rubber seed oil is used as a feedstock for the synthesis of biodiesel.

The present study mainly focuses on the exploration of the optimized condition of process parameters for maximum production of RSME and assessment of significant parameters affecting the biodiesel yield using Central Composite Design technique in RSM method. The biodiesel is produced from rubber seed oil using indigenously prepared Fe/C catalyst over the influence of various parameters and the parameters such as temperature, time and catalytic percentage are optimized by analyzing its effect on catalytic conversion and activity by RSM method.

MATERIALS AND METHODS

Materials

The waste biomass flamboyant pods were used as a precursor for the preparation of catalyst support, refined rubber seed oil, analytical grade methanol, ferrous sulphate and sodium hydroxide and deionised water.

Optimization Procedures

Fixing factors, which has to be optimized (those which affect the production of biodiesel from RSO

Factors	Units	Low level	High level
Time –A	Minutes	40	50
Temperature-B	Degree C	80	90
Catalyst –C	Percent	2	30

Fixing the response factors which has to be analysed (catalytic conversion and activity).

Experimental Procedure

The transesterification reaction was carried out in a 1 L three necked glass reactor. The reactor is equipped with the thermometer to note the temperature throughout the span of the reaction and other neck is mounted with vertical helical coil glass condenser to maintain the constant molar ratio of methanol to oil by refluxing of the evaporated methanol during the reaction. The trans-etherification reaction was carried at a different set of parametric conditions following the design matrix developed by Taguchi method, and it is represented in Table 1. Initially, the glass reactor was filled with 20 mL rubber seed oil and an appropriate amount of methanol and heated up to the desired temperature as mentioned in Table 1. As the temperature attained, particular amount of prepared catalyst was added to the reaction mixture and time is noted as the start of the reaction. Then, solid catalyst was separated by filtration from the reaction mixture. The filtrate has then undergone the distillation to recollect the methanol for reuse. After distillation, the remaining mixture was given to separating funnel and kept it for 12 h to separate by decantation. Density difference clearly separated the two layers consists upper organic layer of RSME and a lower layer of glycerol. The yield of RSME produced was calculated on the basis of the amount of RSM

Experimental Design by Central Composite Design (CCD)

Three parameters that have been reported to influence biodiesel production were chosen as independent variables: time, temperature and catalyst percentage and described as X_1, X_2 and X_3, respectively. The minimum and maximum levels of each variable were coded as −1, 0, and +1, respectively. A second-order mathematical equation, including all interaction terms, was used to calculate the predicted response:

3

$$Y =¼\ \alpha_0\ þ \sum_{i}^{3}{}_{¼1\alpha i}\ X_i\ \ þ \sum_{\alpha ii} X_i^2\ þ \sum_{i}^{3}{}_{<j\ \alpha ij}X_iX_j \quad (1)$$

i¼1

Where Y is the predicted response, α_o is the intercept, α_i is the first order model coefficient, α_{ij} is the interactive effect, and α_{ii} denotes the coefficients of Xi^2, and e is the random error.

Statistical Analysis

The observed data were subjected to multiple regression analysis to obtain the coefficients of the quadratic equation. The F-value and the probability p-value were used to appraise the significance of the model. The multiple coefficient of correlation (R) and coefficient of determination (R^2) were calculated to evaluate the performance of the regression equation. The behaviour of the model in the experimental area was investigated graphically. Statistical evaluation of the model was carried out using analysis of variance (ANOVA). The significant effects are visually identified for the Pareto plot of standardized effects. The bars relate to the absolute magnitudes of the estimated effect coefficients. Any effect that goes beyond the vertical line ($p = 0.05$) may be taken to be significant [7].

Table 1: Experimental Design Matrix Developed by RSM Method

Select	Std	Group	Run	Space Type	Factor 1 A:TIME minutes	Factor 2 b:TEMPERAT... degree C	Factor 3 C:CATALYST %	Response 1 conversion %	Response 2 activity
	10	1	1	Center	45	85	2.5	74	53.2
	9	1	2	Center	45	85	2.5	51	62.9
	11	1	3	Center	45	85	2.5	88	53.4
	13	2	4	Axial	45	76.3397	2.5	70	62.6
	12	2	5	Axial	45	76.3397	2.5	71	57.3
	19	3	6	Axial	45	85	3.36603	90	67.9
	18	3	7	Axial	45	85	1.63397	66	59.8
	16	3	8	Axial	36.3397	85	2.5	97	67.8
	17	3	9	Axial	53.6603	85	2.5	81	59.2
	20	4	10	Center	45	85	2.5	75	60.4
	22	4	11	Center	45	85	2.5	76	59.1
	21	4	12	Center	45	85	2.5	83	60.1
	14	5	13	Axial	45	93.6603	2.5	76	59.1
	15	5	14	Axial	45	93.6603	2.5	83	53.6
	7	6	15	Factorial	40	90	3	76	65.9
	5	6	16	Factorial	40	90	2	79	60
	8	6	17	Factorial	50	90	3	85	60.7
	6	6	18	Factorial	50	90	2	97	57.4
	3	7	19	Factorial	40	80	3	55	63.2
	2	7	20	Factorial	50	80	2	81	60.8
	1	7	21	Factorial	40	80	2	80	58.9
	4	7	22	Factorial	50	80	3	91	63.9
	24	8	23	Center	45	85	2.5	80	53.6
	23	8	24	Center	45	85	2.5	79	60.7
	25	8	25	Center	45	85	2.5	76	59.2

Analytical Methods

Characterization of the Catalyst

The indigenously prepared activated carbon used as a catalyst support was initially subjected to instrumental analysis to investigate the physical properties like surface area and pore volume. These properties are estimated using a surface area analyser. Surface area is calculated by BET method[8], while pore volume was estimated using DR isotherm equation. From the SEM images using scanning electron microscope, the surface morphology of the catalyst support and Fe/C catalyst was studied. By using X-ray energy dispersive analysis (EDX) the elemental composition of the Fe/C catalyst was also investigated. And to detect the presence of functional groups on the catalyst surface FT-IR analysis of the Fe/C catalyst was also performed.

RESULTS AND DISCUSSIONS

Model Equation

$Y1 = 75.78+2.59A+3.06B+0.83C-1.25AB+3.25AC+0.000BC +4.37A^2-0.28B^2+0.71C^2$

$Y2=243.12118-14.22196A+4.74403B+70.96031C-0.050000AB+130000AC+4.08492E-014BC+0.17489A^2-0.011072B^2$

$+2.82261C^2$

Where Y1-Response 1

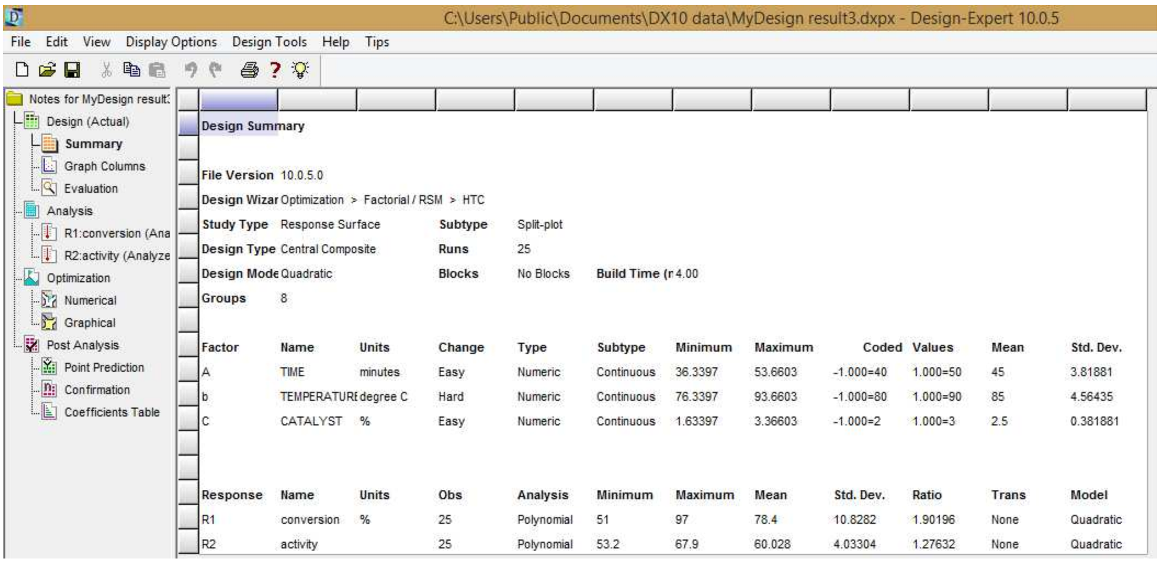
Y2-Response 2

Optimized Parameters

Time: 45 minutes

Temperature: 85ºC

Catalytic percentage: 25%



Design Summary

File Version	10.0.5.0				
Design Wizar	Optimization > Factorial / RSM > HTC				
Study Type	Response Surface	Subtype	Split-plot		
Design Type	Central Composite	Runs	25		
Design Mode	Quadratic	Blocks	No Blocks	Build Time (r	4.00
Groups	8				

Factor	Name	Units	Change	Type	Subtype	Minimum	Maximum	Coded	Values	Mean	Std. Dev.
A	TIME	minutes	Easy	Numeric	Continuous	36.3397	53.6603	-1.000=40	1.000=50	45	3.81881
b	TEMPERATURE	degree C	Hard	Numeric	Continuous	76.3397	93.6603	-1.000=80	1.000=90	85	4.56435
C	CATALYST	%	Easy	Numeric	Continuous	1.63397	3.36603	-1.000=2	1.000=3	2.5	0.381881

Response	Name	Units	Obs	Analysis	Minimum	Maximum	Mean	Std. Dev.	Ratio	Trans	Model
R1	conversion	%	25	Polynomial	51	97	78.4	10.8282	1.90196	None	Quadratic
R2	activity		25	Polynomial	53.2	67.9	60.028	4.03304	1.27632	None	Quadratic

Figure 1: Design Summary

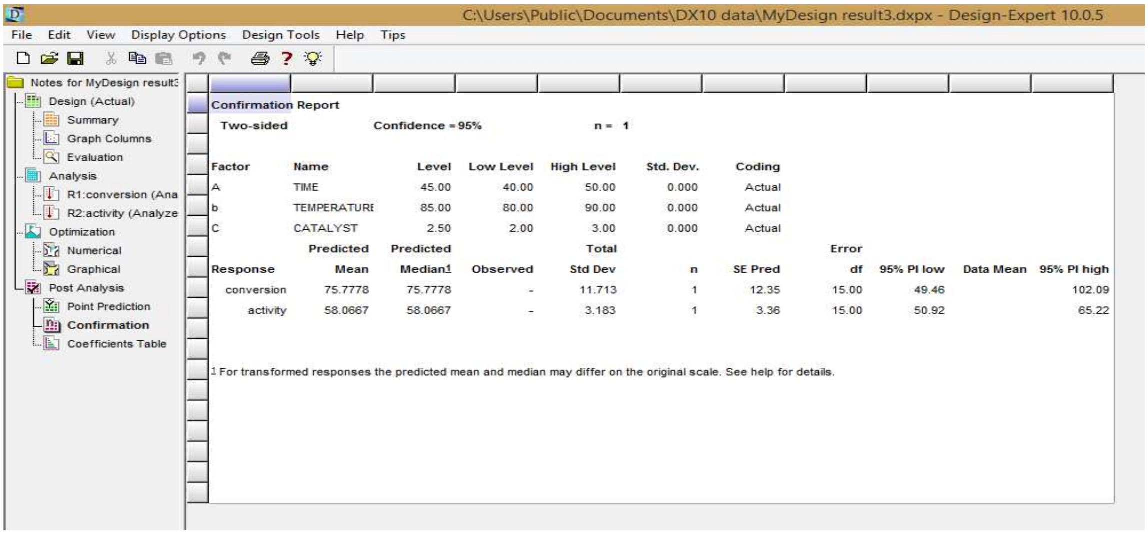


Figure 2: Confirmation Report

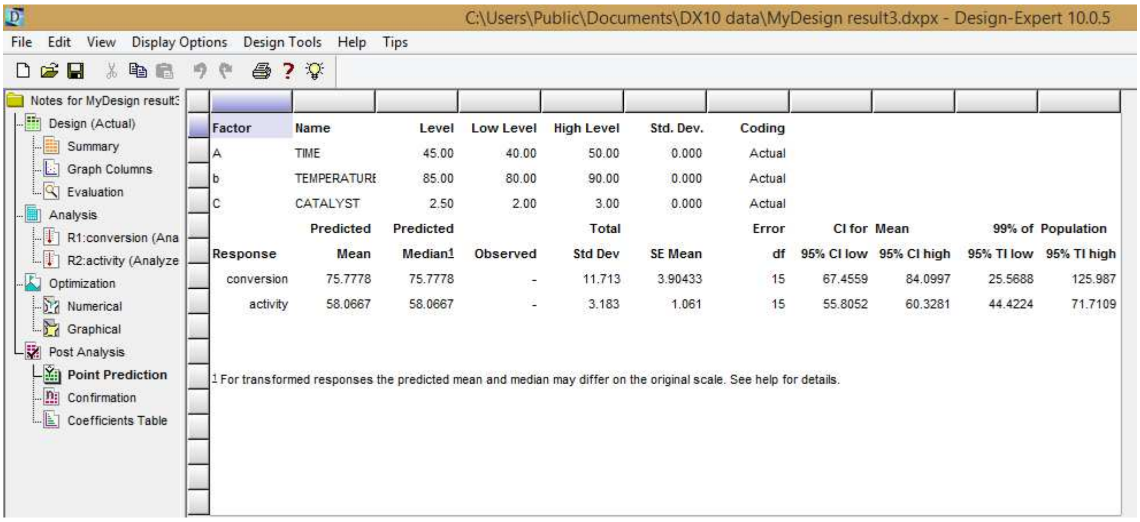
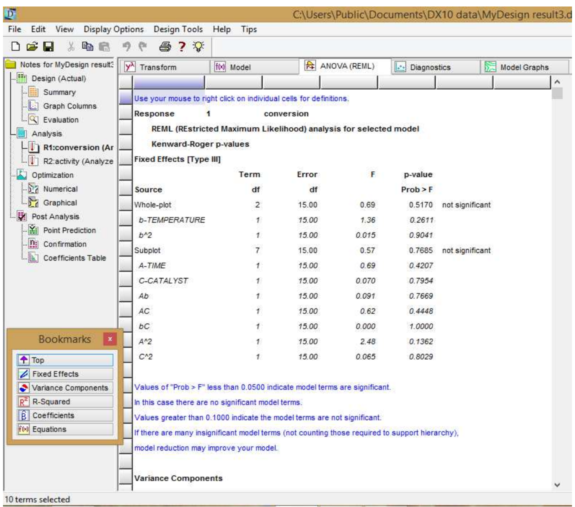


Figure 3: Optimized Parameters



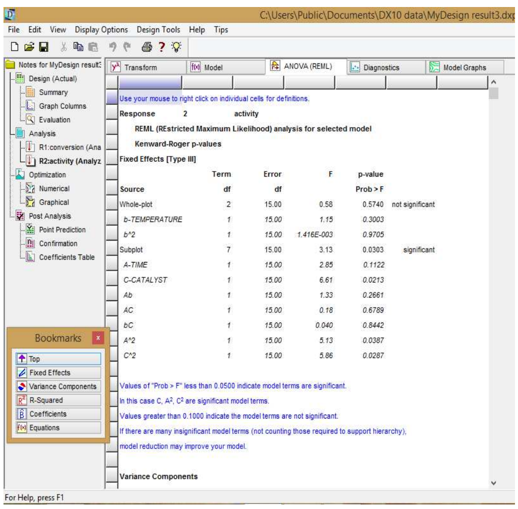


Figure 4: Anova Table (Response 1) **Figure 5: Anova Table (Response 2)**

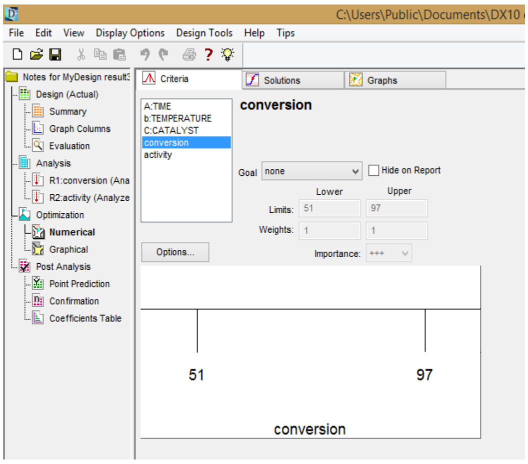


Figure 6: Result (Response 1)

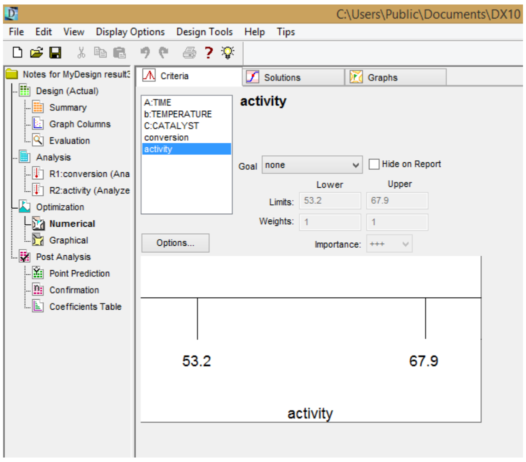


Figure 7: Result (Response 2)

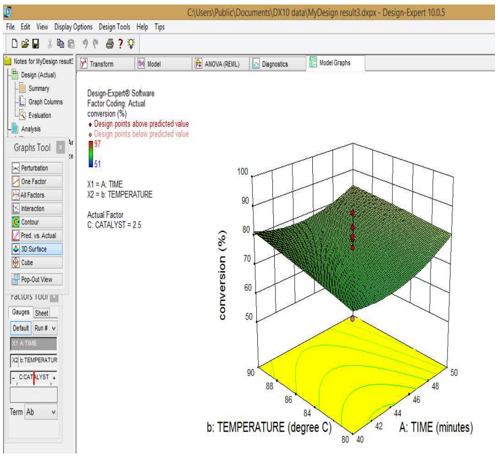


Figure 8: 3D Plot (Response 1)

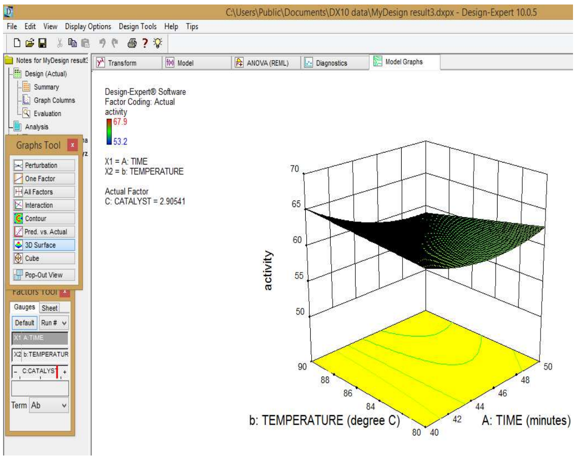


Figure 9: 3D Plot (Response 2)

CONCLUSIONS

The indigenously prepared iron doped catalyst (Fe/C catalyst) in the present study showed betterperformance for catalysing the rubber seed oil into rubber seed methyl ester. The optimization of biodiesel synthesis from the rubber seed oil has been done in the present study over the influence of 3 parameters viz. Catalyst percentage, time and temperature using central composite design by response surface methodology. The experimental results were statistically analysed by ANOVA study and contribution factor. The conclusions drawn from the experiment is that the catalyst can achieve a conversion level of 51-97 and activity level of 53.2-67.9 at an optimized condition of time(45 minutes), temperature(85ºC) and catalyst percentage(2.5%).

Thus, the study can be concluded by saying that the indigenously prepared heterogeneous iron doped catalyst from flamboyant waste pods are effective for the transesterification of the rubber seed oil into the corresponding esters (rubber seed methyl ester) successfully and its properties suggest that it could be used as energy efficient and eco-friendly fuel in diesel engines.

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