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OPTIMIZATION OF BIO-LUBE PRODUCTION USING THE AUTOMATED FUZZY LOGIC CONTROLLED TEMPERATURE BIOREACTOR AND D-OPTIMAL DESIGN

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Abstract: Optimization of process parameters of many existing biolubricant were carried out based on the factor interactions and yield without putting the physicochemical properties into consideration. The aim of the work is to optimize the reaction temperature and time for the production of high-quality biolubricant using set objectives in relation to the physicochemical properties of the bio-lube produced. The reactor used consists of a reactor tank (2500 cm³), temperature, time, stirrer (agitation), speed ranged between 0 – 300 °C, 0 – 120 minutes and 80 – 500 rpm respectively. The optimum temperature and time design matrix using Response Surface Methodology (D-optimal design) in Design Expert 11.0 software package. The results of the optimized yield of the castor seed oil biolubricant produced were compared with international standards for lubricant. The result shows better yield in the operational process of the fuzzy logic-controlled reactor. The yield of biolubricant for the experimental design matrix produced using the fuzzy logic reactor ranged between 79 – 96 %. Higher values were obtained from the study, except for the pour point values that were lower. The higher yield and the physical and thermal properties in castor oil biolubricant from the fuzzy logic temperature-controlled reactor could be as result of the uniform temperature and reaction time during the production process. The optimum condition for the bio-lube produced considering the optimum set goals for best quality biolubricant produced considering the set goal objective to comprise all the physico-thermal properties obtained in the study.

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The optimum condition was obtained at ninety-nine (99.5) minutes reaction time and 250 °C reaction temperature with desirability value of 0.76. with the physico-thermal properties of 96.48 %, 33.7 mPa.s (33.7 cP), 15.5 mPa.s (15.5 cP), 179.9 °C, 291 °C, 15.9 °C, for viscosity at 40 °C, viscosity at 100 °C, viscosity Index, Flash point, pour point. The physicochemical properties of biolubricant produced at optimum condition are within the recommended international standard. It can be concluded that the fuzzy logic-controlled reactor product is better biolubricant considering the number of parameters set goal to determine the optimum condition for production.

Key words: Bio-lube, D-optimal, Numerical, Physico-thermal, Point-prediction

INTRODUCTION

Engine oil is prominent amongst the most widely recognized lubricants and well utilized in engineering concepts. Conventional lubricants produce a non-clean, highly toxic working environment due to the burning of lubricant as fuel to generate power. Basic disposal strategies which incorporate recycling, copying, landfill and release into water, is one of the major constraints in developing countries [1], [2].

Research has been conducted to have environmentally friendly lubricants that can be used in place of the conventional lubricants which are called biolubricant [3]. The best application for biolubricant is in machinery that loses oil directly into the environment during use, Total Loss Lubricants (TLLs), and in machinery used in any sensitive areas, such as in or near water. Applications for TLLs include two-stroke engines, chain saw bars and chains, railroad flanges, cables, dust suppressants, and marine lubricants [4], [5].

MATERIAL AND METHODS

The experimental design used for the synthesis of castor oil bio lubricant was optimized with the aid of response surface methodology (RSM) using D-optimal design approach. This methodology is used to scrutinize the relationship between the response variable and a set of experimental factors. A polynomial equation was used to predict the response as a function of independent variables and their interactions. In this work, the number of independent variables was two and therefore the response for the quadratic, cubic polynomials become $Y = \beta_0 + \sum \beta_i x_i + \sum \beta_{ii} x_i^2 + \sum \sum \beta_{ij} x_i x_j$ (1). Where β_0 ; β_i ; β_{ii} and β_{ij} are constant, linear, square and interaction regression coefficient terms, respectively, and x_i and x_j are independent variables Table 1. The design expert software version 11 was used for multiple regression analysis, analysis of variance (ANOVA), optimization analysis set goals data and the response surface regression procedure. The lack of fit of the model was evaluated by the coefficient of determination R^2 , adjustable R^2 and its statistical significance that was checked by the P-value and F-test. Automated Fuzzy Logic Controlled Temperature Bioreactor was used for all the reactions [6].

Table 1. Independent Variables and Their Levels for Response Surface Methodology, D-Optimal Design Response Surface methodology for the Transesterification Reaction of castor oil ester

Independent variables	Variable Levels		
		-1	+1
Time	X_1	30	120
Temperature	X_2	100	250

The experimental factors for this experiment are temperature and time. The two (2) experimental factors were used to establish the number of experimental runs which was given as 13 runs. The details of the factor – levels and values obtain using RSM are as presented in the design matrix Table 2.

Table 2. Experimental matrix design for the optimization of the Transesterification reaction process for biolubricant production

Coded factor			Actual factor	
Run	X_1 : Time	X_2 : Temperature	X_1 : Time	X_2 : Temperature
1	-1.00	-1.00	30	100
2	-0.11	-0.15	70	164
3	1.00	0.33	120	200
4	0.00	-1.00	75	100
5	0.47	1.00	96	250
6	0.62	-0.25	103	156
7	-0.25	0.57	64	218
8	-1.00	0.00	30	175
9	-0.61	-0.50	48	138
10	-1.00	-1.00	30	100
11	-1.00	1.00	30	250
12	1.00	-1.00	120	100
13	-1.00	1.00	30	250

Characterization of castor oil biolubricant

Physicochemical properties of castor oil biolubricant were carried out using the American Standard Testing Methods [8]. The main properties of lubricant oil required for good performance were carried out.

These properties are:

1. Determination of Acid Value and Free Fatty Acid

One gram (1g) of oil was placed inside 100ml beaker, 25ml of petroleum ether with 25ml of alcohol (methanol) was added, 4 drops of phenolphthalein was added as an

indicator and mixed thoroughly, after that, the solution was titrated against 0.1M KOH until the end point was reached that is there was colour change which turn to pink. [6].

The Acid Value and Free Fatty Acid (mgKOH/g) was calculated as:

$$\text{Acid Value} = \frac{\text{Titre (ml)} \times 0.1 \times 56.10}{\text{Weight of Sample (1g)}}$$

$$\text{Free Fatty Acid} = \frac{\text{Acid Value}}{2}$$

2. Determination of viscosity and Viscosity Index (VI)

Viscosity of the oleo-chemicals was determined using a viscometer. Two hundred millilitres (200ml) of the sample was poured into a beaker and heated to 40 °C and 100 °C respectively. A desired rotor number of the viscometer was attached to the upper coupling between the thumb and forefinger while cautiously rotating the rotor in anticlockwise direction. The rotor was immersed in the middle of the sample up to the indentation level on the shaft.

The viscometer was then switched and let to run at 60 rpm. The result was obtained and recorded as the viscosity of the sample in mPa s (cP) [9].

The result at 40 °C and 100 °C was used to calculate the Viscosity Index

$$\text{Viscosity Index (VI)} = \frac{L - U}{L - H} \times 100$$

3. Determination of Pour Point

This was measured according to ASTM D97. A medium sized test tube was filled with the oil sample and (the test tube with its content) then be placed in a test tube holder. The set up was then placed in a refrigerator and allowed to solidify. After it solidified, the test tube was removed and a thermometer was used to read the temperature of the solidified sample.

4. Flash Point Determination

heat was supplied to a 10ml of the sample in crucible to raise the temperature of the sample. A thermometer probe was suspended in the sample with a retort stand. The flash point obtained was the minimum temperature at which the vapour of the sample ignited when a flame was brought near [10].

RESULTS AND DISCUSSION

Castor oil methyl ester (biodiesel) feed stock was used in production biolubricant using response surface methodology, D-optimal experimental design.

The response of the experiment as presented in Table 3 shows the different actual factor-levels and their corresponding actual yield in percentage and the physico-thermal properties. [8].

Table 3. Response analysis of biolubricant Yield and physicothermal properties

	Factor 1	Factor 2	Response 1	Response 2	Response 3	Response 4	Response 5	Response 6
Run	A: Time	B: Temperature	Biolubricant Yield	Viscosity @40	Viscosity @100	Viscosity index	Flash Point	Pour point
1	30	100	79	31	7.5	166.282	275	-18
2	70	164	84.5	35	10	173.839	269	-15
3	120	200	91	37	12	175.612	281	-16
4	75	100	84	32	9	174.904	269	-19
5	96	250	96	34	18	176.365	294	-15
6	103	156	86.5	30	11	182.823	288	-15
7	64	218	88	32.5	14	180.643	290	-16
8	30	175	83	35	10	173.839	285	-14
9	48	138	81	37	12	175.612	277	-17
10	30	100	79	32	8.5	172.152	263	-19
11	30	250	94	37.5	15	176.43	285	-14
12	120	100	83	34	9	171.073	260	-18
13	30	250	93	33	13	180.163	291	-15

A numerical, graphical and privative optimization method was used to analyze the data collected from two process parameters (Time and Temperature) to generate the best operational conditions that will convene all goals set (Table 4). To obtain the desirability index for biolubricant produce with relation to the investigated physicochemical properties qualities. Goals were set for each condition and properties at maximum, minimum and in-range for castor oil biolubricant produced.

Table 4. Numerical Optimization Constraints for Biolubricant Produced

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:Time	is in range	30	120	1	1	3
B:Temperature	is in range	100	250	1	1	3
Biolubricant Yield	maximize	79	96	1	1	3
Viscosity @40	minimize	30	37.5	1	1	3
Viscosity @100	maximize	7.5	18	1	1	3
Viscosity index	maximize	166.282	182.823	1	1	3
Flash Point	maximize	260	294	1	1	3
Pour point	minimize	-19	-14	1	1	3

The predicted result shows that operational condition that gave fourteen desirability solution the best confidence interval in terms of physico-thermal properties and biolubricant yield are 96.48 %, 33.7 mPa.s (33.7 cP), 15.5 mPa.s (15.5 cP), 179.9 °C, 291 °C, -15.9 °C, respectively for biolubricant yield, viscosity at 40 °C, Viscosity at 100 °C, viscosity index, flash point and pour point (Table 5). The desirability with functional object which vary between 0 and 1 with relation to all parameter goals set on the numerical optimization, the selected desirability condition value of the two available solution for the castor oil biolubricant yield and physicochemical properties was 0.76 was the highest desirability value with time and temperature condition of 99.5 minutes and 250 °C respectively.

Table 5. Numerical Optimization desirability Solutions of the produced Biolubricant

Num	Time	Temp.	Biolub. Yield	Viscosity @40	Viscosity @100*	Viscosity index	Flash Point	Pour point	Desirability
1	99.54	250.00	96.48	33.70	15.50	179.90	291.00	-15.8	0.76
2	99.86	250.00	96.49	33.71	15.56	179.91	291.00	-15.8	0.76
3	99.31	250.00	96.47	33.70	15.49	179.89	291.01	-15.8	0.76
4	100.79	250.00	96.53	33.73	15.51	179.93	290.99	-15.8	0.76
5	98.32	250.00	96.43	33.67	15.48	179.87	291.02	-15.8	0.76
6	97.78	250.00	96.41	33.66	15.47	179.85	291.03	-15.8	0.76
7	102.01	250.00	96.58	33.76	15.53	179.97	290.97	-15.8	0.76
8	104.36	250.00	96.67	33.83	15.57	180.03	290.94	-15.9	0.75
9	93.12	250.00	96.22	33.58	15.41	179.72	291.08	-15.8	0.75
10	91.41	250.00	96.15	33.55	15.38	179.68	291.10	-15.8	0.74
11	108.74	250.00	96.83	33.98	15.63	180.15	290.89	-15.9	0.73
12	88.76	250.00	96.04	33.52	15.35	179.60	291.14	-15.7	0.72
13	110.70	250.00	96.90	34.05	15.69	180.20	290.87	-15.9	0.71
14	85.93	250.00	95.92	33.50	15.31	179.53	291.17	-15.7	0.69

*Intervals adjusted for variation in the factors (POE).

The solutions within one and eight can be considered usable all depending on the properties rely on biolubricant grade. The responses optimisation is comparable to research work by [11], that the *Jatropha* bio-lubricant yield increased with increase in temperature over time variation.

Graphical optimisation Overlay plot in Figure 1 shows the coefficient magnitudes and significance for all analysed responses in the numerical optimisation solution of the experimental factors that makes it easy to see what terms are common to all the response models, displays the area of feasible response values in the factor space.

Figure 1. Optimisation Overlay plot of biolubricant in relation to Temperature and Time

On the plot, the bright yellow points range between temperature of 100 – 148 °C with time of 30 to 120 minutes shows where the entire range of all intervals of the produced biolubricant meet the specified standards that best conciliation can be picked (the sweet spot). The gray colour corresponds to where the point estimate meets the criteria requirements, but part of an interval estimate does not. The standard best conciliation shows that the reaction of the biolubricant will be best done at the sweet spot zone considering only the experimental factors, while the gray shot in consideration of all the tested parameters of the produced castor oil biolubricant. The point prediction of produced biolubricant (Table 6) shows the two-sided points (low and high) of the experimental responses to assist in the uses of prediction and interval estimates at a confidence interval of ninety five percent. The predicted mean, median, standard deviation of all the responses was within standard biolubricant.

Table 6. Point Prediction of produced castors oil biolubricant properties

Response	Predicted Mean	Predicted Median	Std. Dev	SE Mean	95% CI low for Mean	95% CI high for Mean	95% TI low for 99% Pop	95% TI high for 99% Pop
Biolubricant Yield	97.2138	97.2138	1.325	1.59	93.43	100.98	87.75	106.67
Viscosity @40	34.4622	34.4622	2.686	3.23	26.81	42.11	15.28	53.64
Viscosity @100	15.7904	15.7904	1.430	0.95	13.65	17.92	8.183	23.39
Viscosity index	180.462	180.462	3.618	2.42	175.0	185.86	161.22	199.70
Flash Point	290.756	290.756	6.699	4.48	280.7	300.74	255.13	326.37
Pour point	-15.946	-15.946	0.907	1.09	-18.5	-13.36	-22.42	-9.470

Two-sided Confidence = 95% Population = 99%

CONCLUSIONS

The research statistical data analysis clearly shows that the responses parameters as significant effect on the optimization process of castor oil biolubricant as the overlay point shows the sweet spots range of 100-148 °C temperature with time of 30 to 120 minutes with prediction mean biolubricant yield of 97.21 % and the optimization process desirability function of both the factors and the responses have biolubricant yield of 96.4 % at desirability of 0.76 at 250 °C temperature with time of 99.5 minutes. But the desirability optimisation gives the optimisation in terms of best quality of biolubricant.

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**OPTIMIZACIJA PROIZVODNJE BIOMAZIVA KORIŠĆENJEM
AUTOMATIZOVANE FUZZY LOGIČKE KONTROLE TEMPERATURNOG
BIOREAKTORA SA D-OPTIMALNIM DIZAJNOM**

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Sažetak: Optimizacija procesnih parametara mnogih postojećih biomaziva izvršena je na osnovu interakcije faktora i prinosa bez uzimanja u obzir fizičko-hemijskih osobina biomaziva. Cilj rada je optimizacija temperature, reakcije i vremena za proizvodnju visokokvalitetnog biomaziva pomoću postavljenih ciljeva u odnosu na fizičko-hemijske osobine proizvedenog biomaziva.

Reaktor ima parametre: rezervoar od 2500 cm^3 , temperature od 0 do 300°C , vreme reakcije od 0 do 120 minuta, i brzinu mešanja koja se kretala 80 - 500 rpm.

Matrica optimalnog dizajna temperature i vremena, kontroliše se primenom metodologije optimalne površine (D-optimalni dizajn) sa softverskim paketom Design Expert 11.0.

Rezultati optimizovanog prinosa proizvedenog biomaziva od ricinusovog ulja upoređivani su sa međunarodnim standardima za maziva.

Rezultat pokazuje bolji prinos u operativnom procesu logički kontrolisanog reaktora.

Prinos biomaziva dobijen sa dizajnom eksperimentalne matrice, proizvedenog pomoću fuzzy reaktora, kretao se između 79 - 96%. Dobijene su veće vrednosti iz studije, osim vrednosti tačke prelivanja, koje su bile niže. Veći prinos, fizička i termička svojstva u biomazivu od ricinusovog ulja iz reaktora sa fuzzy (nepreciznom) kontrolom, mogu biti rezultat ujednačene temperature i vremena reakcije tokom proizvodnog procesa. Iz studije su dobijene veće vrednosti, osim vrednosti za tačke prelivanja koje su bile niže.

Veći prinos i fizičko- termičke osobine za biomazivo ricinusovog ulja iz reaktora sa fuzzy kontrolom temperature mogu biti rezultat ujednačene temperature i vremena reakcije tokom proizvodnog procesa.

Optimalni uslov za proizvedeno biomazivo uzimajući u obzir optimalno postavljene ciljeve za najkvalitetnije proizvedeno biomazivo, uzimajući u obzir postavljeni cilj koji treba da obuhvati sve fizičko-termičke osobine koje su dobijene u studiji.

Optimalni uslovi su dobijeni u toku 99,5 minuta vremena reakcije i temperature u reaktoru od 250°C , sa poželjnim vrednostima od 0,76., i fizičko-termičkim osobinama od: 96,48%, 33,7 mPa.s (33.7 cP), 15,5 mPa.s (15.5 cP), $179,9^\circ\text{C}$, 291°C , $-15,9^\circ\text{C}$, za viskozitet biomaziva na 40°C , za viskozitet na 100°C , indeks viskoznosti, tačku paljenja, tačku tečenja biomaziva.

Fizičko-hemijske osobine biomaziva proizvedenog u optimalnim uslovima su u okviru preporučenih međunarodnih standarda.

Može se zaključiti da je produkt sa fuzzy logikom (neprecizna logika) kontrolisane proizvodnje reaktora, bolje biomazivo, obzirom na broj postavljenih kontrolisanih parametara za određivanje optimalnih uslova proizvodnje.

Ključne reči: Biomaziva, D-optimalni dizajn, Numeričko, Fizičko-termičke osobine, tačkasto predviđanje

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