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OPTIMIZATION OF BIODIESEL PRODUCTION FROM USED OIL OF FISH PROCESOR USING SODIUM METHOXIDE AND SULPHONATED EGG-SHELLS AS CATALYST

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ABSTRACT

Finding a long-term solution to the world's growing dependence on conventional energy sources and the depletion of fossil fuels is what worries scientists the most right now. An alternative that appears promise is biodiesel. It was investigated how to increase the generation of biodiesel from leftover fish processors' oil using sodium methoxide and sulphonated eggshells as a catalyst, based on Box-Behnken design. The greatest yield of 96.81% was achieved at 1:12 oil to methanol ratio, 65 °C reaction temperature, 90 minutes, and 0.5 w/w% catalysts loading. The variables constant, methanol to oil ratio, reaction temperature, reaction time, reaction temperature* reaction temperature, and methanol to oil ratio*catalyst load all had a significant effect on the biodiesel production, according to the response surface regression. The model does a good job of accounting for the link between biodiesel and process variables. Thus, residual fish processor oil might be effectively converted into more advantageous and environmentally benign biodiesel fuel.

Keywords: Biodiesel, sulphonated egg-shell, sodium methoxide

INTRODUCTION

Due to the expanding population and rapid industrialization, the use of oils derived from fossil fuels has increased recently. The sustainability of the world's energy supply is continuously threatened by the need for fossil fuels in industries like heating and electricity generation. Additionally, the development of internal combustion engines and the transportation industry are causing a quicker pace of exploitation of petroleum reserves. In addition, using fossil fuels causes environmental pollution. Because of the decline in the usage of fossil fuels and the harm they do to the environment, finding a significant alternative energy source is necessary (Behçet, 2011).

Therefore, the necessity to address the issue of fossil fuel shortages and lower the cost of energy must be raised. The only renewable energy source that effectively addresses the issue of the market's escalating energy fuel prices is biomass. Because the Organization of the Petroleum Exporting Countries' (OPEC) production of petroleum was insufficient, a thorough investigation was made to identify other sources of biodiesel (Kahn et al., 2002). According to Edlund et al. (2002), it is viewed as an alternative fuel that tackles the problems of environmental deterioration and a global energy deficit. Additionally, it will take the place of petro-diesel, reducing the amount of pollutants produced by combustion equipment (Lin and Lin, 2006).

Biodiesel production sources that could be used include vegetable oil, animal oil, waste oil, waste from plants and animals, agricultural wastes, and municipal waste (Balat, 2008). Recent studies have concentrated on the production of biodiesel from vegetable oil, spent cooking oil, and industrial oil. The resulting biodiesel may be recycled more than once, has a higher flash point, degrades more quickly, and produces less pollution. Since biodiesel has similar physical and chemical qualities to diesel, it can be combined with diesel oil and utilised in engines. (Zhao et al., 2012).

For the first generation of biofuel, canola oil, palm oil, jatropha, soya bean, and other plants have been the primary sources of feedstock (Ong *et al.*, 2011) or fats oil (Ma *et al.*, 1998). However, the first-generation feedstock's sustainability and economic viability come under attack. Environmentalists assert that large-scale biodiesel production's development of oil crop plantations has led to deforestation in several nations, including Indonesia, Malaysia, Argentina, and Brazil (Gao *et al.*, 2011). Competition for arable land for food and fiber plantations, excessive water and fertilizer demand, and subpar agricultural techniques all worsen the situation. Consequently, it was argued that the first-generation feedstock was ineffective since it had an impact on global food markets and food security (Noraini *et al.*, 2014). Second-generation biodiesel derived from waste and inedible crops is being studied as a solution to this issue. The availability of farmland to create the by-products for commercial-scale biodiesel production, however, is the greatest obstacle to the development of second-generation biodiesel.Therefore, alternative raw materials for biodiesel production that has less impact on the food industry need to be explored (Cheng and Timilsina, 2011). In the last ten years, a promising raw source for the manufacturing of biodiesel is algae.. Numerous studies have further demonstrated that due to its excellent fuel economy and environmental index, algae is a better feedstock for biodiesel production than first and second-generation biodiesel (Brennan and Owendi, 2010). Microalgae can be grown on non-agricultural land, which reduces the need for more arable area for oil crops (Gumba et al., 2016). One of the raw components for making biodiesel is thought to be fish waste. According to Eslick et al. (2009), 60–70% of the total amount of fish produced is used for human consumption as well as the creation of fish meal and oil. Given that fish oil is thought to offer significant medical advantages (Sharma et al., 2014). In general, not all fish portions are consumed; some are thrown away. The components, which are not edible and are regarded as trash, include the spine, skin, heads, tails, and stomachs. There are more than 60 different fatty acids in fish oil, according to the literature. Of these, almost 80-85% are classified into four categories of fatty acids, such as (a) C14:0 and C16:0, (b) C16:1 and C18:1, (c) C20:1 and C22:1, and (i.e) C20:5, C22:5 and C22:6. Fish oil is fortified with eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA), which makes up more than 90% of all polyunsaturated fatty acids (PUFA).Currently, fish oil is a potential feedstock for the production of biodiesel, but research on the biodiesel produced is limited (Nelson, 2006). According to the study, fish waste has limited uses. Waste is around 50% of all fish processing. The overall oil content ranges from 40% to 65% (Barros *et al.,* 2010). Therefore, there is an opportunity to use this fish waste to convert it into biodiesel. Discarded fish waste has reportedly been found to be able to be turned into useful goods like biodiesel, slurry, and biogas for energy production (Yahyaee *et al.,* 2010)

MATERIALS AND METHODS

The method involved in this study entails sample collection and pretreatment, reagents preparations, procedures for physicochemical analyses and analytical instrumentations.

Pre-treatment of Used Oil

The used oil from the fish processor underwent the following pre-treatment. By using filtration, solid particles, salt, pepper, and spices were eliminated. The oil was treated with sodium thiosulfate (Na_2SO_4), which forms clumps when it absorbs water. Decantation was used to remove the crystal. To get rid of any leftover contaminants, used oil was combined with n-hexane (1:3 oil/n-hexane v/v) (Hossain et al., 2008).

Preparation of Catalysts

Preparation of Sulphonated Egg Shell Catalyst

Egg shell sample was gathered in Sokoto's Mana Area. According to the approach described by Babatope and Racheal (2020), sulphonated CaO catalyst was made from used egg shells by calcination. The egg shell was sun-dried after being cleaned with distilled water. The cleaned egg shell was crushed and heated to 900 ^oC in the furnace 4 hours. The produced calcined sample was repeatedly rinsed in hot distilled water until the pH was 7, and then it was dried for 24 hours at 70 ^oC in the oven.. A sulphonated acid catalyst was prepared from a dried calcined sample by combining it with 98 percent sulphuric acid in a closed autoclave and heating it at 160 ^oC for 6 hours. Washing with hot distilled water at (70 ^oC) removed the sulphuric acid that hadn't yet reacted. The catalyst was thoroughly dried in an oven at 70 ^oC after being washed (Rashid et al., 2019; Muhammad et al., 2017).

Plate 1: Egg Shell

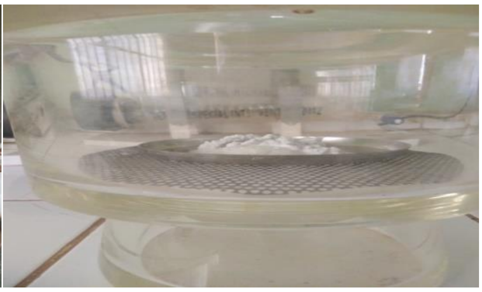

Plate 2: Sulphonated Egg Shell

Transesterification of Fish Waste Oil

500 cm^3 conical flask was filled with a 17 g oil sample, which was then gradually added to with (100 cm^3) sodium methoxide. Heat was applied to the reaction mixture until it reached a temperature of 65^oC for 60 mins with continuous stirring. After 60 minutes The mixture was put into a (500 cm^3) separating funnel and given 24 h to settle. Two distinctive layers was observed, the upper layer is the biodiesel; the bottom layer is the glycerol and is drawn out. The same procedure was reported using 100 cm^3 of methanol and 0.5 w/w sulphonated egg shell in replacement of sodium methoxide. Finally, the same procedure was repeated , but with the use of a combined catalyst (sodium methoxide and sulphonated eggshell).

% yield = $volume of biodiesel after drying$/Initial volume of oil fed into the processX 100

Experimental Design

On MINITAP 17 statistical software, the experiment was designed using the Box-Behnken response surface method. The effect of four quantitative variables; reaction temperature Methanol to oil ratio, reaction time, catalyst amount and one categorical factor; catalyst were investigated. The design generated a total of 54 runs (randomized). The optimum parameters were determined for the highest yield of diesel.

Table 1: Optimization parameters and their levels

Factor	Unit	Low-Level	high level
Methanol to oil ratio	-	6	12
Temperature	-^oC	55	65
Time	Mins	60	120
Catalyst	w/w	0.5	1.5

RESULT AND DISCUSSION

Optimization Process of Biodiesel Yield of Used Oil using Sodium Methoxide and Sulphonated Egg Shell as Catalyst

The results of process parameters optimization namely: methanol to oil ratio, temperature, catalyst load and reaction time for biodiesel yield is shown in Table 4.6.The highest biodiesel yield of 96.81% was obtained at 1:12 methanol to oil ratio, temperature at 65ºC, time at 90minutes and catalyst load of 1.0 %. While the lowest yield of 60.89 % was obtained at 1:12 methanol/oil ratio, temperature at 55 ºC, time at 60 minutes and catalyst load 1 %.

Table 2: Optimization process for biodiesel production and yield obtained.

StdOrder	RunOrder	PtType	Blocks	Methanol/Oil	TMP	Time	Cat	Yield%
1	1	2	1	6	55	90	1	67.33
2	2	2	1	12	55	90	1	76.77
3	3	2	1	6	65	90	1	83.37
4	4	2	1	12	65	90	1	91.16
5	5	2	1	9	60	60	0.5	80.23
6	6	2	1	9	60	120	0.5	85.12
7	7	2	1	9	60	60	1.5	78.90
8	8	2	1	9	60	120	1.5	87.03
9	9	2	1	6	60	90	0.5	91.23
10	10	2	1	12	60	90	0.5	96.10
11	11	2	1	6	60	90	1.5	82.00
12	12	2	1	12	60	90	1.5	69.00
13	13	2	1	9	55	60	1	64.76
14	14	2	1	9	65	60	1	80.00
15	15	2	1	9	55	120	1	79.90
16	16	2	1	9	65	120	1	86.1
17	17	2	1	6	60	60	1	73.77
18	18	2	1	12	60	60	1	91.00
19	19	2	1	6	60	120	1	85.88
20	20	2	1	12	60	120	1	91.50
21	21	2	1	9	55	90	0.5	73.79
22	22	2	1	9	65	90	0.5	94.99
23	23	2	1	9	55	90	1.5	73.00
24	24	2	1	9	65	90	1.5	95.55
25	25	0	1	9	60	90	1	82.76
26	26	0	1	9	60	90	1	88.90
27	27	0	1	9	60	90	1	89.31
28	28	2	1	6	55	90	1	68.10
29	29	2	1	12	55	90	1	77.00
30	30	2	1	6	65	90	1	82.58
31	**31**	**2**	**1**	**12**	**65**	**90**	**1**	**96.81**
32	32	2	1	9	60	60	0.5	73.11
33	33	2	1	9	60	120	0.5	85.00
34	34	2	1	9	60	60	1.5	84.76
35	35	2	1	9	60	120	1.5	86.90
36	36	2	1	6	60	90	0.5	84.25
37	37	2	1	12	60	90	0.5	92.34
38	38	2	1	6	60	90	1.5	83.24
39	39	2	1	12	60	90	1.5	71.00
40	**40**	**2**	**1**	**9**	**55**	**60**	**1**	**60.89**
41	41	2	1	9	65	60	1	81.22
42	42	2	1	9	55	120	1	80.62
43	43	2	1	9	65	120	1	86.56
44	44	2	1	6	60	60	1	82.99
45	45	2	1	12	60	60	1	91.05
46	46	2	1	6	60	120	1	85.84
47	47	2	1	12	60	120	1	91.44

48	48	2	1	9	55	90	0.5	72.77
49	49	2	1	9	65	90	0.5	95.44
50	50	2	1	9	55	90	1.5	74.73
51	51	2	1	9	65	90	1.5	96.50
52	52	0	1	9	60	90	1	88.90
53	53	0	1	9	60	90	1	89.22
54	54	0	1	9	60	90	1	82.70

Design of Experiments

On the statistical program Minitab 17, the Box-Behnken design method was used to create the experiment. Table 3 below shows the impact of four variables, including the Methanol to oil ratio, reaction time, reaction temperature, and catalyst load. On yield, upper and lower levels were examined.

Table 3: Factorial regression analysis of biodiesel yield (%) versus transesterification variables showing the estimate coefficient of the model

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-	86.97	2.26	38.48	0.000	
Methanol to Oil Ratio	5.38	2.69	1.13	2.38	0.022	1.00
Temperature (^oC)	16.72	8.36	1.13	7.40	0.000	1.00
Time (mins)	7.43	3.72	1.13	3.29	0.002	1.00
Catalyst (w/v)	-3.48	-1.74	1.13	-1.54	0.132	1.00
Methanol to Oil Ratio*Methanol to Oil Ratio	-1.40	-0.70	1.69	-0.41	0.682	1.25
Temperature (^oC)*Temperature (^oC)	-9.62	-4.81	1.69	-2.84	0.007	1.25
Time (mins)*Time (mins)	-5.29	-2.65	1.69	-1.56	0.126	1.25
Catalyst (w/v)*Catalyst (w/v)	-1.24	-0.62	1.69	-0.37	0.716	1.25
Methanol to Oil Ratio*Temperature (^oC)	0.92	0.46	1.96	0.24	0.815	1.00
Methanol to Oil Ratio*Time (mins)	-3.52	-1.76	1.96	-0.90	0.374	1.00
Methanol to Oil Ratio*Catalyst (w/v)	-9.55	-4.78	1.96	-2.44	0.019	1.00
Temperature (^oC)*Time (mins)	-5.86	-2.93	1.96	-1.50	0.143	1.00
Temperature (^oC)*Catalyst (w/v)	0.11	0.06	1.96	0.03	0.977	1.00
Time (mins)*Catalyst (w/v)	-1.63	-0.81	1.96	-0.42	0.680	1.00

Regression Equation in Uncoded Units

Yield% = -891 + 5.40 Methanol to Oil Ratio + 26.23 Temperature (0 ^oC) + 2.055 Time (mins)
+ 33.7 Catalyst (w/v) - 0.078 Methanol to Oil Ratio*Methanol to Oil Ratio
- 0.1925 Temperature (0 ^oC)*Temperature (0 ^oC) - 0.00294 Time (mins)*Time (mins)
- 2.49 Catalyst (w/v)*Catalyst (w/v) + 0.031 Methanol to Oil Ratio*Temperature (0 ^oC)
- 0.0195 Methanol to Oil Ratio*Time (mins)
- 3.18 Methanol to Oil Ratio*Catalyst (w/v) - 0.0195 Temperature (0 ^oC)*Time (mins)
+ 0.023 Temperature (0 ^oC)*Catalyst (w/v) - 0.054 Time (mins)*Catalyst (w/v)

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
5.53523	70.30 %	59.64 %	41.56 %

To examine the impact of transesterification factors, response surface regression was utilized. To determine if a variable or its interaction are statistically significant or not, the p-value was utilized. The variable is considered statistically insignificant if the P-value is greater than 0.05, and vice versa. The models correlation coefficient R2 is 70.30%, which shows that the variable fit the scenario. The variables constant, Methanol to oil ratio, Reaction Temperature, Reaction time, Reaction temperature* Reaction temperature and Methanol to oil ratio*Catalyst Load were statistically significant on Biodiesel yield. The variables catalyst load, Methanol to Oil Ratio*Methanol to Oil Ratio, Methanol to Oil Ratio*Reaction Temperature, Reaction Time(mins)*Catalyst Load and reaction temperature*Catalyst Load have P-Value greater than α-value which shows that they are statistically insignificant on biodiesel yield.

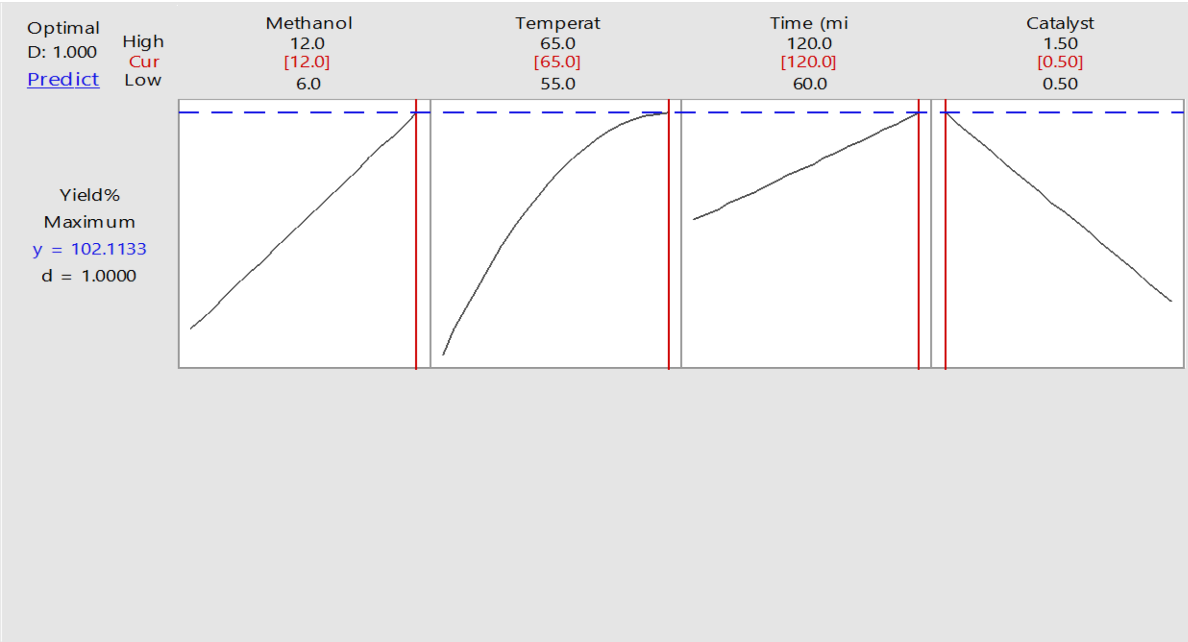

Figure.1: Optimization Plot, Effect of Transesterification Variables on Biodiesel Yield.

The maximum yield 102.11% that may be attained when the target is set to 100 is predicted by the optimization plot in Fig. 3.1. The methanol to oil ratio needed to be 1:12, the temperature needed to be 65^{o}C, the reaction duration needed to be 120 minutes, and the catalyst load needed to be 0.55w/w in order to get the maximum yield as predicted by the statistical model (optimization Plot). The experimental yield was 96.81%, however the optimization plot projected a yield of 102.11%, which is practically identical to it. It demonstrates how regression analysis utilising known catalyst and other reaction factors could optimise biodiesel prediction.

Effect of Operating Variables on Biodiesel

The effects of Methanol to oil ratio, reaction time, reaction temperature, and catalyst load were studied using an optimization plot while the interactions of variables were studied using Contour plot.

Effect of Methanol to oil ratio on Biodiesel Yield.

The esterification and transesterification steps in the manufacture of biodiesel both require the right amount of methanol to alter the equilibrium and increase the creation of fatty acid methyl ester. Figure 1 demonstrates an increase in the methanol to oil ratio from 1:6 to 1:12. The amount of main biodiesel rises, which can be a result of more methanols being available to react with the oil and make biodiesel. The methanol-to-oil ratio of 1:12 produced the highest yield.

Effect of Reaction Temperature on Biodiesel Yield

A picture of how reaction temperature affects biodiesel is shown in Figure 1. The biodiesel yield increases dramatically as the temperature rises from 55 to 65^{o}C. Transesterification required some initial thermal energy because it was an endothermic reaction (Samart *et al.*, 2009). High temperatures are not recommended, though, because when the temperature raises to the point at which methanol reaches its boiling point, the substance vapourizes and produces many bubbles that stifle the reaction and reduce biodiesel yield (Long *et al.*, 2010).

Effect of Reaction Time on Biodiesel Yield

According to Samart et al., (2009), Figure 4.3 shows that the biodiesel output rapidly increases from 60 to 120 minutes of reaction time until biodiesel equilibrium is attained. After equilibrium is reached, the reaction is no longer reversible.

Effect of Catalyst Load on Biodiesel Yield

Figure 4.3 shows that as catalyst load increases, biodiesel yield decreases. With a yield of 96.81%, 0.50 was found to be the ideal catalyst load. According to Yang *et al.* (2009), the extra catalyst load raises the reactant's viscosity, which also lowers biodiesel production.

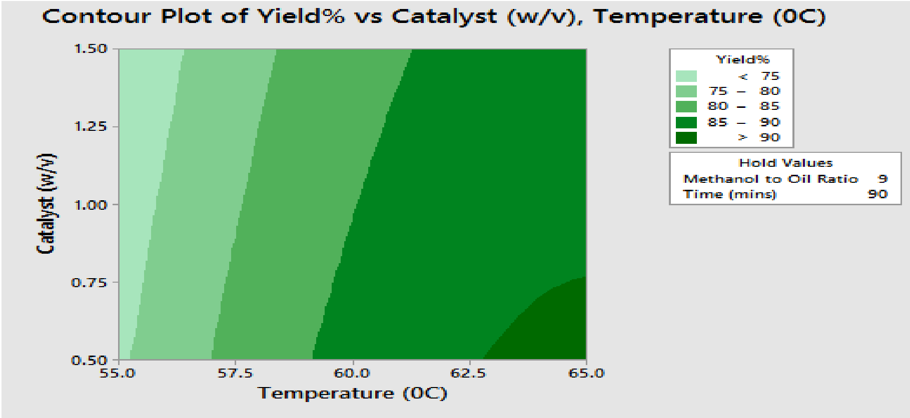

Figure 2: Contour plot of the interaction of catalyst load and reaction temperature on biodiesel yield

Figure 2 above shows that at a catalyst between 0.5-0.75 w/v, the dissolution yield increases with temperature such that yields greater than 90 % can only be attained when the temperature is between 62.7-65 ºC. Similarly, the lowest yield was attained when the dissolution temperature is between 55-55.2 ºC and a catalyst between 0.5-1.50 w/v, hence, regardless of the amount of catalyst utilized, a higher yield is obtainable provided the temperature increases.

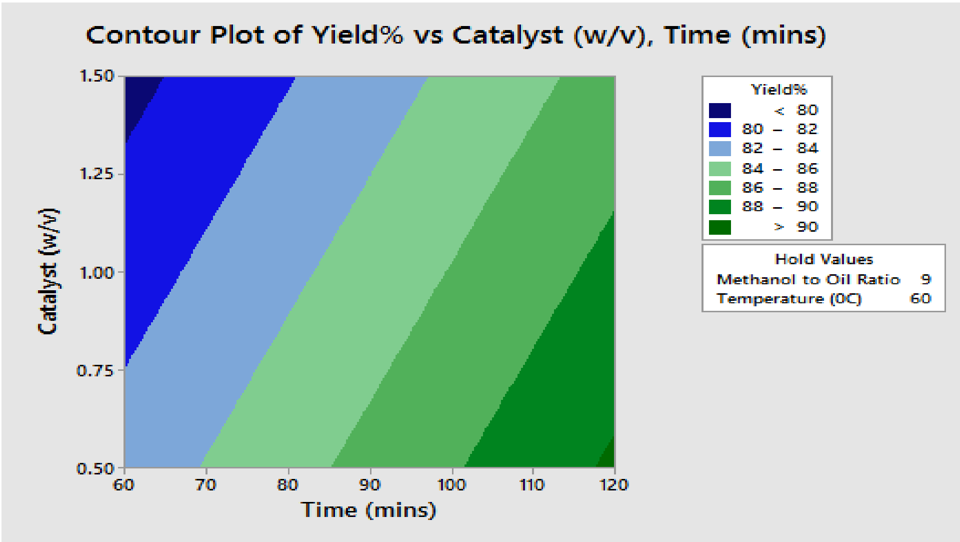

Figure 3: Contour plot of interaction of catalyst load and reaction time on biodiesel yield

Figure 3 shows that at a constant methanol to oil ratio 9, the yield increases with reaction time such that the highest yield > 90 % can be attained when the reaction time is between 102-120 min provided the reaction catalyst is between 0.50-1.15 (w/v). Similarly, the lowest yield of the reaction <80 is attainable only when the reaction time is > 62 min and the catalyst >1.30, in essence, regardless of the amount of catalyst utilized, a higher yield is attainable as the reaction time increases.

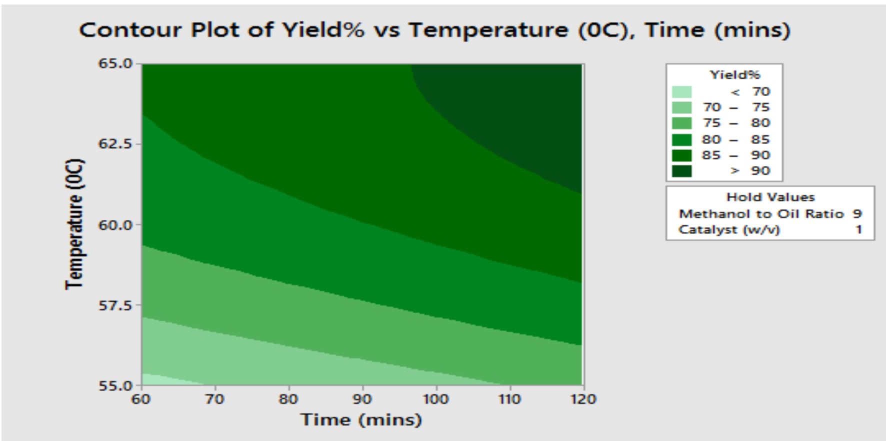

Figure 4: Contour plot of interaction reaction temperature and reaction time on biodiesel yield.

Figure 4 above shows that at a constant methanol to oil ratio (r) of 9, the yield of the reaction increases with reaction time such that the highest yield > 90 % can be attained when the reaction time is between 100min to 120min provided the reaction temperature is between 61 ºC to 65 ºC. similarly, the lowest yield of the reaction is attainable only when the reaction time is < 70 min and the reaction temperature <56 ºC, in essence,as both temperature and time increases, biodiesel yield also increases.

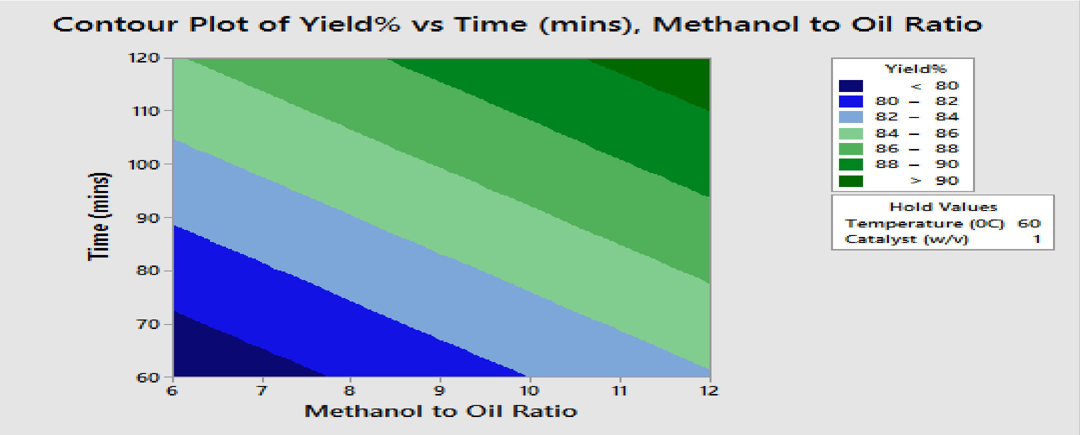

Figure 5: Contour plot of interaction of reaction time and methanol to oil ratio on biodiesel yield.

Figure 5 above shows that the yield of the reaction increases with the volume of methanol to oil ratio and the reaction time. Yields > 90 % are obtainable only when the reaction time is > 110 min with a methanol to oil ratio of >10 w/v. Similarly, at a time of 72 min the lowest yield of the reaction is attainable at a methanol to oil ratio > 7 w/v. Hence, as both the time and the volume of methanol to oil ratio increases, the yield of the reaction also increases.

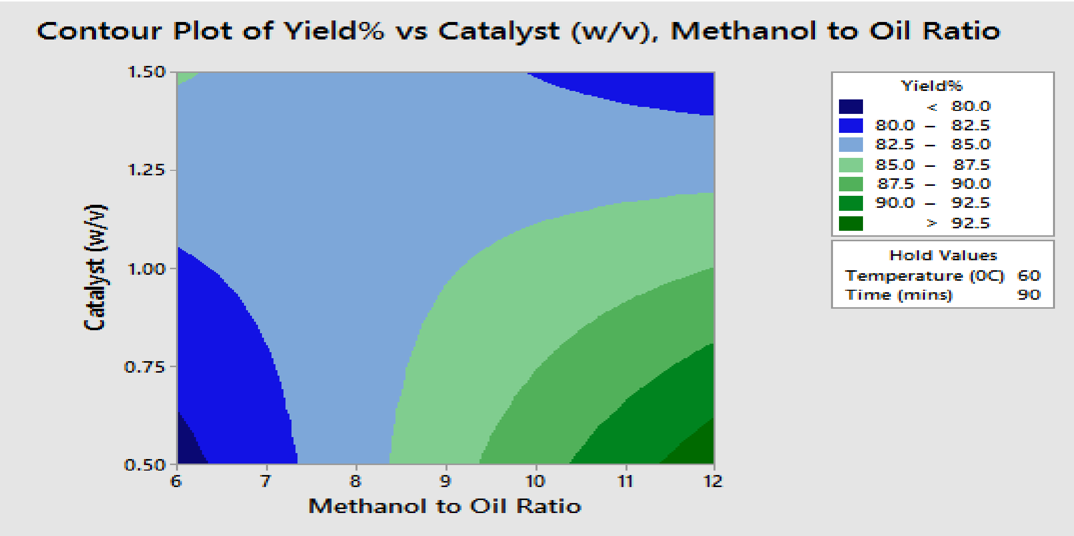

Figure 6: Contour plot of interaction of catalyst load and methanol to oil ratio on biodiesel yield.

Figure above shows that the yield of the reaction increases with the volume of methanol to oil ratio. Yields > 92.5 % are obtainable only when the catalyst is >0.50 w/v with a methanol to oil ratio of >11. Similarly, at a catalyst of >0.60 w/v lowest yield of the reaction is attainable at a methanol to oil ratio > 6. Hence, regardless of catalyst as the volume of methanol to oil ratio increases, higher yield of the reaction also increases.

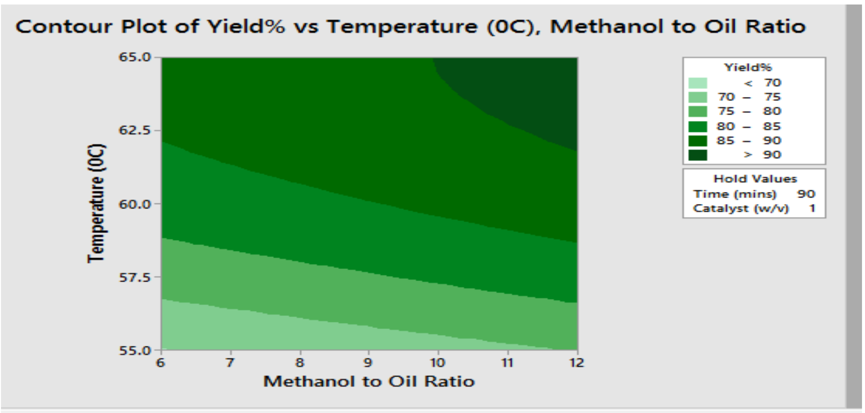

Figure 7: Contour plot of interaction of reaction temperature and methanol to oil ratio on biodiesel yield.

Figure 7 above shows that a higher yield >90 % is attainable when the methanol to oil ratio is >10 such that the volume of methanol to oil ratio is >11 at a temperature >61 ºC. Also, the lowest yield <70 % is obtainable when the temperature is >56 ºC and methanol to oil ratio <12 w/v. Hence, as both temperature and methanol to oil ratio increases, biodiesel yield also increases.

Response Optimization and Validation of Used Oil

Based on the model obtained to confirm the optimization results, the Box Behnken design in the minitab statistical tool was able to work as an optimal design for the intended response. With the ideal process conditions of Methanol to oil ratio 12 and 12, temperature 65 and 64.5250, reaction time (min) 120 and 120, and catalyst (w/v) 0.5 and 0.901872 in the validation process, the expected outcomes for optimal solutions were achieved through optimization as shown in Table 3.3 However, it predicted a maximum yield of 102.113 and 96.810 with desirability of 1 and 1 respectively. From a validation experiment performed at the values of the process variables predicted in dissolutions 1 and 2, dissolution yields of 102.02% and 97.40% were obtained. The optimized predicted and experimentally validated results for the two solutions, 102.113% and 96.810%, were found to be 102.02% and 97.40%, respectively. Since the predicted yields of 102.113% and 96.810% are similar to the experimental yields of 102.02% and 97.40%, it indicates a relative deviation of 0.093% and 0.59%, respectively, which makes the optimisation model more desirable.

Table 4: Response Optimization and Validation of Used Oil

Solution	Methanol to oil ratio	Temperature (ºC)	Time (min)	Catalyst (w/v)	Yield		Desirability
					Predicted	experimental	
1	12	65	120	0.5	102.113	102.02	1
2	12	64.5250	120	0.90187	96.810	97.40	1

CONCLUSION

The Box-Behnken method proved successful in optimizing the transesterification reaction of used fish processors' oil to biodiesel, yielding an optimal yield of 96.81% at the optimum transesterification conditions of 65 ºC, 90 min, 1:12 methanol to oil ratio and 0.5 w/w%. According to the response surface regression, the biodiesel yield was statistically significantly affected by the variables constant, methanol to oil ratio, reaction temperature, reaction time, reaction temperature* reaction temperature, and methanol to oil ratio*catalyst load.

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